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Birefringence characterization of injection molded microplates

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ABSTRACT

Birefringence affects the quality of image analysis in injection molded micro-plates. Depending upon their manufacturing / production processes and the type of material, different plates exhibit varying amounts of birefringence. This birefringence is attributed to residual stress generated during the molding process. Polarimeter is the standard tool for birefringence distribution visualization and quantification. Broad chemical resistance and high mechanical stability of the plates are the desirable properties that can be characterized by birefringence measurement. Birefringence, expressed in nm/cm is light retardance (nm) after passing through a sample with certain thickness (cm). Low or uniform birefringence plates provide high-resolution demonstrating higher performance, hence suitable for bio-chemical analysis.

Keywords: Birefringence, photoelasticity, residual stress, micro plates, polarimeter, retardance

1. INTRODUCTION

Injection Molding techniques is one of the popular techniques for manufacturing numerous plastic parts which has been clearly explained by^{1,2}. Advantage of injection molded parts is related to its high production rate and low cost manufacturing. Molded micro plates are desired to be precise in size, shape and uniform throughout the production. Although the advancement in manufacturing of injection molded micro plates is in progress, there are still challenges behind perfect and precise manufacturing as per specifications. Due to free mold shrinkage and stress induced distortion, depending on the materials used; there are errors in the radii of curvature and thickness of the plates³. A small distortion may result in formation of residual stress and retardation which are further errors in injection molded micro plates. For injection molded materials, there is always a residual stress because of distortion present in the material which has been studied previously by⁴. As polymer melt is forced to flow in a narrow region in high pressures, which result anisotropic polymer structure of the molded parts resulting two effects, birefringence and residual stress in the molded plates thus degrading the quality of the manufactured micro plates. Stress is dependent on the birefringence of the material, hence residual stress⁵ and birefringence of the micro plates can be calculated at the same time. The most commonly used material for micro plates are polypropylene, polystyrene, flexible vinyl and cyclo-olefin polymers. The residual stress due to birefringence present in the specimen results in deformation of the micro plates. The objective of this work is to assess the quality of the injection molded micro plates by measuring birefringence and retardation present in the micro plates. Birefringence is optically anisotropic characteristic property exhibited by material because of mechanical stresses present during manufacturing which causes part deformation in the material⁶. This characteristic was first observed in calcite explained by Danish scientist Rasmus Bartholin in 1669. During 19th century, birefringence was correctly described by using polarization method present in the optically anisotropic materials, behaving light as a wave with field components i.e., electric and magnetic fields are perpendicular to each other described by Augustin Jean Fresnel. Birefringence of a material is defined as,

$$\Delta n = n_e - n_o \quad (1)$$

where n_e and n_o are the refractive indices of extraordinary and ordinary axis present in the material. The birefringence of the material is influenced by using polarized light which can be obtained by phase shifting polarimetry and thus stress distribution can be calculated⁷. The materials possess optical axis in which component of light perpendicular and parallel to it have different refractive indices. Likewise after measuring birefringence characteristics of the material, residual stress and retardation present in the specimen can be measured both destructively⁸ and non-destructively which is based on the principle of photoelasticity^{9,10} given by,

$$\Delta = 2\pi C / \lambda (\sigma_1 - \sigma_2) \quad (2)$$

Where Δ is the induced retardation, C is the stress-optic coefficient, t is specimen thickness, σ_1 and σ_2 are first and second principal stresses, respectively.

2. WORKING PRINCIPLE

Figure 1: Working Principle

Birefringence measurement system measures retardation of the optical sample along the optical axis. It measures both magnitude and fast axis orientation of the samples possessing optical retardation¹¹. It facilitates users to switch different measurement angles. He-Ne laser beam of wavelength 632.8 nm is polarized and then modulated by the photoelastic modulator transmitted through the sample and divided by a beam splitting mirror. As shown in the figure 1 the beam passes through a combination of analyzer, optical filter and photo detector. Software algorithm process signal level from electronics module into linear birefringence magnitude and axis angle.

Computer selects from two inputs, by sequential measurements from the two signal channels. Data is analyzed; retardation magnitude and fast axis angle are displayed and stored in a file.

Figure 2: Photoelastic modulator assembly

During automatic mapping, the XY translation stage will move to next predetermined measurement location. Results are displayed in specified formats. Photoelastic modulators modulate the polarisation of a beam of light, especially used for measurement of birefringence, circular, linear dichroism, optical rotation, ellipsometry, polarimetry, reflection difference spectroscopy and FTIR(Fourier Transform Infrared) double modulation. The principle of operation is based on photoelastic effect in which mechanically stressed sample exhibits birefringence proportional to the resulting strain. It is resonant device producing oscillating birefringence in low frequency ultrasound range. It consist of a rectangular bar (transparent material like silica) attached to a piezoelectric transducer. The frequency of the bar is determined by length of bar and speed of longitudinal sound wave. The transducer is tuned to same frequency as that of rectangular bar which is driven by electronic circuit controlling the amplitude of vibration. Hence, when transparent solid material is compressed or stretched; the material becomes birefringent, different polarisation of light have slightly different speeds of light when passing through it. The vibration is sustained by a quartz piezoelectric transducer attached at the end of the bar.

2.1 Modes of operation

Photoelastic modulator can be used both as a modulator and analyzer. It can be used as a modulator to produce polarisation modulation of light beam while analyzer as determining polarisation state of light beam. The modes are briefly discussed below:

2.2 Modulator

As a modulator, incoming light is linearly polarized in a plane at 45 degree with the axis of modulator. The light oscillates between left and right circularly polarized light with elliptically polarized light between extremes as shown in figure 3. The optical oscillation frequency is at the modulator frequency (1f) used for the studies of circular dichroism.

Figure 3: Experimental setup as polarizer

2.3 Analyzer

It can be used to analyze the state of a polarized beam of light as shown in figure 4.

Figure 4: Experimental setup as analyzer

Net circular polarization component will produce an electrical signal in the detector at modulator frequency ($1f$). Similarly, a net linear polarization component at 45° with respect to modulator axis will produce an electrical signal in the detector at twice the modulator frequency ($2f$). Hence by using reference signal and received signal, it enables us to measure the birefringence of the optical materials.

3. EXPERIMENTAL WORK

3.1 Micro plate material

Various micro plates are assessed for their qualitative and quantitative analysis. Micro plates are made up of polystyrene, polypropylene and cyclo-olefin polymers. For cell culture and calorimetric assays, polystyrene micro plates are used. For high resistance to solvents including DMSO (Di-Methyl Sulf Oxide) and ethanol, especially in compound storage or assays; polypropylene is preferred. Flexible vinyl is known for economical purpose and used as non-sterile general assay plates. Polypropylene and polystyrene are the most widely used materials these days for manufacturing most of the micro plates¹². Polystyrene being clear polymer, possess excellent optical properties useful for precise measurements. Polypropylene is distinguished because of its excellent chemical and thermal stability, so used where temperature and chemical interaction is the altering factor. Minimal auto-fluorescence, remarkable transparency particularly in UV range with substantially high clarity is present in polypropylene as compared to polystyrene.

3.2 Part Geometry

Parts involved in the measurement are of circular and rectangular shape depending on their type which are presented in table 1.

Table 1: Dimension of Specimen Plates

Type	Material	Shape	Diameter (mm)	Thickness (mm)	
Conventional	BD falcon	Circular	7.00	0.19	
	Cop	Rectangular	8.00 × 8.00	0.76	
	Corning	Rectangular	8.00 × 8.00	1.00	
	Grenier	Rectangular	3.50 × 3.50	0.19	
	Nunc 96	Circular	4.50	1.20	
	Nunc 384	Circular	5.00	1.20	
	Perkin Elmer	Circular	6.50	0.76	
Modern	1a	Circular	2.50	1.00	
	1b	Circular	2.50	1.00	
	1c	Circular	2.50	1.00	
	1d	Circular	2.50	1.00	
	1e	Circular	2.50	1.00	
	Advanced Plasma method	Biomatcoc	Circular	3.50	1.25
		COC 24w	Rectangular	11.00 × 11.00	1.25
		CR8	Rectangular	11.00 × 11.00	1.25
		G2CR8	Circular	2.00	1.25
		COC1DOC1	Circular	2.00	1.25
		COC2	Circular	3.50	1.25
		PS1	Circular	2.50	1.25
		PS2	Circular	2.00	1.25

3.3 Injection molding Process

Molded Inserts are used for molding of micro plates specimen. They are usually made up of high precision aluminum mold inserts made by ultra-precision machining known as single point diamond turning machine. Mold inserts are also made up of tool steel, plated by nickel and then turned by diamond to achieve the surface roughness in nanometer (nm) precision level.

3.4 Birefringence Measurement Instrument

Birefringence measurement based on photo elastic modulation¹³ is carried out by Exicor 150AT to measure low level strain birefringence in optically transparent samples up to (150mm × 150mm) at 100-240 Volts AC with a frequency of 50-60 Hz and current of 2 A. The light source contained in the sample scanning module is Helium-Neon laser of low power 5mW and 633nm wavelength belonging to class IIIa CDRH (Center for Devices and Radiological Health) and IEC(International Electro technical Commission) Class 3R which is safe¹⁴. The retardation range is of 0-316 nm of 1mm measurement spot size with maximum sample height of 250 mm with data capture rate of 0.01 seconds per data point to 1.50 seconds per data. The machine is desired to operate on a temperature range of 18-24 degree Celsius, stable to within 1 degree Celsius per hour.

3.5 Design of Experiment

There are 20 specimens; each micro plates readings are taken two times for correlation purpose, pre-classifying the modern and conventional specimen plates. Minimum spacing of 0.5 mm was made on the specimen plates. The number of data points collected varies on the specimen size. Maxima, minima, mean and standard deviations are calculated based on the measured data. Moreover, standard deviations are calculated to confine the data lying within 99.7% of the values recorded.

3.6 Product Characterization

In this study, there are two types of specimen plates, seven micro plates are conventional and rest are modern. The shape of the plates are also different, few are rectangular while rest are circular. The raw materials made from the plates are different like polystyrene, polypropylene and cycloolefin copolymer. Depending upon their chemical composition, their performance characteristics are different.

4. RESULTS AND DISCUSSION

Figure 5: Birefringence of different micro plates

4.1 General Observation

The molded micro plates are made up of different materials exhibiting varying properties. Polystyrene, polypropylene, cyclic olefin copolymers are common materials for manufacturing micro plates. High Birefringence is observed in BD falcon, Grenier and NUNC384 specimens which are made up of crystal clear polystyrene, polyolefin and polypropylene material. The thickness of the

micro plate ranges from (0.19-1.20) mm which is clearly illustrated in Table 1 with their respective diameters. Birefringence and retardation values along with standard deviation were calculated in nanometer range provided in Table 2 which represents the material uniformity throughout the specimen. From the Table 3 BD falcon has higher birefringence while COC24 W and CR8 possess low birefringence. Nunc 384 has higher non uniformity of the material while COC 24 W has good uniformity throughout the material.

4.2 Birefringence and Retardation Analysis

Figure 5 represents the birefringence values of different micro plate specimen carried out in the experiment. BD falcon on the top first shows higher birefringence, NUNC 384 on top second represents high birefringence deviation, COC 24W on top third has low birefringence, rest are normal birefringence images of micro plates. Ideally, optically transparent micro plates are stress free and the refractive index is quite uniform in nature. During manufacture upon applied stress and different factors like temperature gradient and thermal expansion, the material will undergo deformation due to change in material microstructure, hence has different particle densities along axes. This different particle density along axes results different light velocities transmission due to different refractive index known as birefringence of the material. Due to birefringence, the optical characteristics of material are changed which deters the image quality and provides inaccurate reading of the specimen. The birefringence of the plates changes the polarization state of the beam which results different imaging characteristics on the specimen of same material. The light is polarized splitting into two different components with different propagation velocities, i.e. ordinary and extraordinary rays; explained in equation (1). Once they are passed through micro plate, there relative phase is different from its initial predetermined value, hence the polarization state has effectively varied due to birefringence. This phase lag can measured with efficient technique known as photoelasticity. As polarized light travels through the micro plate specimen with a thickness of t , relative retardation is induced which is expressed by the equation (2). The stress optic coefficient is desired to be constant for isotropic specimen but for anisotropic birefringent material stress optic coefficient varies with the orientation of stress. Retardation is the amount of light retarded through the specimen due to birefringence and the stress difference present in the specimen. Retardation is directly proportional to the residual stress present in the specimen. Hence birefringence and retardation in the material are caused due to change in material microstructure represented in the Figure 5. Lower and uniform birefringence enable us to get good luminescence discernment, scintillation calculation and high-resolution microscopy during confocal imaging, while high and non-uniform birefringence makes reading or data values inaccurate due to poor visualization and image detection. For materials with high birefringence, there is more pixelate effect, fewer details can be observed on the image while materials of low and uniform birefringence have comparatively less pixelate effect providing more details which can be clearly proved in Figure. Hence, qualities of specimen are quickly assessed by birefringence measurement system.

Table 2: Birefringence and retardation measurement

Specimen	Birefringence (nm/cm)	Retardation (nm)	Standard Deviation (σ_b)	Standard Deviation(σ_r)
BD Falcon	16470.7051	312.9434	310.9325	5.9077
COP	257.6164	19.5788	144.4593	10.9789
Corning	1727.2142	172.7214	372.0727	37.2073
Grenier	15224.3604	289.2628	114.4622	2.1748
NUNC 96	2279.7094	273.5651	11.4290	1.3715
NUNC 384	11459.0846	217.7378	1104.0846	20.9776
Perkin Elmer	1902.1509	144.5635	271.6642	20.6465
1a	2389.1670	238.9167	215.4653	21.5465
1b	1912.5812	191.2581	514.0056	51.4006
1c	2298.7438	229.8744	372.3111	37.2311
1d	1750.4025	175.0402	557.8175	55.7818
1e	2222.1133	222.2113	551.3633	55.1363
Biomatcoc	365.8540	45.7318	44.9277	5.6160
COC 24W	82.8833	67.4133	10.3604	8.4267
CR8	82.8833	10.3604	67.4133	8.4267
G2CR8	307.1393	38.3924	14.1715	1.7714
COC1DOC1	299.6995	37.4624	10.3982	1.2998
COC2	324.3212	40.5401	11.4876	1.4359
PS1	926.2357	115.7795	514.3733	64.2967
PS2	668.1868	83.5233	353.6996	44.2125

4.3 Sources of Errors

The errors arise in the micro plate material due to mold insert deviation during manufacturing process on conventional machining and polishing. For the molding of high end micro plates, ultra-precision machining is employed to manufacture high precision molds. The roughness values are less than micrometer or in nanometer range; but during repeated molds the precision and accuracy of the mold degrades, which degrade the specimen plate. Free mold shrinkage is next problem that arise during manufacturing which depends on the molded material chemical properties. Flow induced stress and temperature gradient present in the molds can cause distortion of a micro plate material. Birefringence and retardation measurement of different micro plates are carried out; mean and the standard deviation are carried out to characterize their uniformity properties. The variation in uniformity of the micro plates is highly dependent on their physical properties like refractive index, transmissivity and thermal expansion coefficient of the material which is clearly mentioned in the Table 3 provided by Baumer.¹⁵

Table 3: Properties of micro plate's material

S.N.	Materials	RefractiveIndex(μ)	Transmissivity (%)	Thermal expansion($10^{-5}/^{\circ}\text{C}$)
1	Acrylic	1.497	85-91	6.0-7.0
2	CycloOlefin Polymer(COP)	1.537	87-92	6.0-7.0
3	CycloOlefinCopolymer (COC)	1.540	92	6.0-8.0
4	Polycarbonate	1.593	92	6.6-7.0
5	Polystyrene	1.604	92	6.74
6	Polysulfone	1.634	84	5.40

5. CONCLUSIONS & FUTURE WORK

Standard materials like polypropylene and polystyrene are used to manufacture the majority of micro plates. Polystyrene, being a clear polymer bears excellent properties; ideally well for precise optical measurements. Polypropylene possesses excellent chemical and thermal stability, so used as a micro plate. Moreover these days, cyclic olefins copolymers are also used as a micro plates, exhibiting low level of auto fluorescence, exceptionally high clarity in UV range of low birefringence than that of polypropylene and polystyrene plates. It is highly optimized for flatness and uniformity possessing least birefringence deviation of the materials. Hence, measuring birefringence and retardation of the micro plates easily asses the quality of the micro plates. Low birefringence with least standard deviation is recommended to use as a micro plate. From the table material COC 24 W has low birefringent value, while COC1DOC1 has low non-uniformity throughout the material, both of which are made up of cyclic olefin copolymer. The residual stress can be calculated by finding stress optic coefficient of the material. It can be obtained by proper calibration on strain gauge loading machine which is the future work to be carried on.

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