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TESTING

## Material Properties

## In-plane orthotropic property characterization of a polymeric battery separator

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## ABSTRACT

The in-plane characterization of an orthotropic porous polymer thin film used as separators in lithium-ion batteries was investigated. The mechanical behavior of an orthotropic material is described by the stress-strain relationships in the principal material directions and shear, and the Poisson's ratios. The measurements of the shear properties and the Poisson's ratio for polymer films with a thickness of tens micrometers has not been well established. In this work, these measurements were attempted for a separator of 25  $\mu\text{m}$  thickness. The mechanical testing was performed with a dynamic mechanical analyzer (DMA). Digital image correlation (DIC) was used for strain measurements. The measured major Poisson's ratio was at a constant value whereas the minor Poisson's ratio increased linearly with strain. The symmetry condition for the elastic orthotropic relation was found to be valid up to 1% strain under transverse tension. The shear properties were measured with an off-axis tensile experiment. The shear modulus determined from the DIC strain measurement agreed well with that from the off-axis modulus and elastic constants. Based on the analogy for anisotropy between the elastic and linear viscoelastic domains, the shear creep response was measured with the off-axis tensile creep experiments. The creep compliances in shear and in the principal material directions were determined. This work laid the foundation for the development of orthotropic elastic and viscoelastic models for battery separators.

## 1. Introduction

Battery separators are microporous membranes. Fig. 1 shows the surface microstructure of a typical PP separator for lithium ion batteries (LIBs) [1]. This porous structure was created by a multi-step process including melt-extrusion, annealing, stretching and heat fixation [1]. The resulted material is highly anisotropic. The separator was treated as an orthotropic material with the two in-plane principal material directions referred to as the machine direction (MD) and the transverse direction (TD).

In a battery, the separator provides the electrical insulation between the anode and cathode while allowing ionic transport between the two electrodes for electrochemical reactions. Its mechanical integrity is critical to the safety of the LIBs. To improve the safety design of electrical vehicles, it is desirable to predict the mechanical response of the separators under various conditions, ranging from thermal ramp to vehicle collision and crash.

Polymer based separators exhibit strong time and temperature dependences [2–6]. This characteristic is particularly important in the thermal ramp predictions. So far, few works have explicitly modeled

the separator in finite element analysis (FEA). In these cases, the separators were modeled as an isotropic viscoelastic material which ignored the anisotropy [7] or with an anisotropic honeycomb model which does not consider the time and temperature dependence [8]. In commercial finite element codes, there is no suitable orthotropic thermomechanical models for the separators.

To model the mechanical behavior of an orthotropic material requires knowledge of the stress-strain relationships in the principal material directions and shear, and the Poisson's ratios. A model based on an orthotropic viscoelastic framework will also require the viscoelastic functions in these directions.

For common types of polymer separator, the stress-strain relations in the MD and TD are available. However, the data on Poisson's ratio and shear properties are scarce. The viscoelastic measurement for polymer separators has only been reported for the MD [3,4].

The shear properties of an orthotropic material may be measured by an off-axis tensile experiment. This method has been widely used in the shear property measurement of unidirectional composites [9–11]. It has also been used in measuring the shear modulus of polymer thin films [12,13] but the details of the experiments were not provided.

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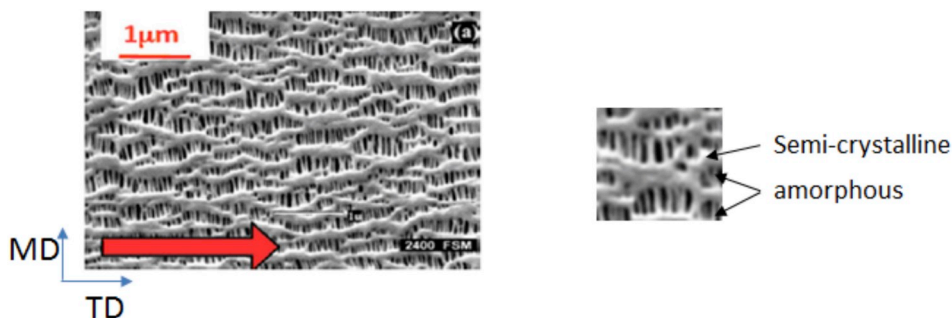


Fig. 1. Surface microstructure of Celgard® 2400 [1]. The two in-plane material directions are referred to as the machine direction (MD) and the transverse direction (TD).

Both the off-axis experiments and the measurement of Poisson's ratios require measuring strains in more than one direction. This poses a challenge for thin, flexible films such as battery separators.

The focus of this paper is on the experimental methods for the characterization of the in-plane orthotropic property of polymer separators, particularly the measurement of the Poisson's ratio and shear properties. The experiments were performed with a dynamic mechanical analyzer (DMA). The use of off-axis tensile experiments for shear property measurement was examined. The strain measurements in multiple directions were performed using the digital image correlation (DIC) technique. The in-plane characterization was carried out for Celgard® 2400, a typical PP separator. The measurements made included the stress-strain curves in the MD, TD and shear at three loading rates, the major and minor Poisson's ratios, and the creep responses in the MD, TD and shear. The creep compliances were determined and fitted with a Prony series. The relationships for the elastic orthotropic solid such as the symmetry condition and the relation between the shear modulus and the off-axis modulus were examined. This work laid the foundation for the development of orthotropic elastic and viscoelastic models for battery separators. The data provide the inputs needed for orthotropic elastic-plastic or orthotropic viscoelastic material models.

## 2. Experimental

### 2.1. Material

A representative separator Celgard® 2400 was investigated in this work. Celgard® 2400 is a monolayer PP porous film with a nominal thickness of 25 μm. Its surface microstructure is shown in Fig. 1. Specimens in the form of long strips were cut from a 93 mm wide separator roll using a razor blade. Fig. 2 shows the orientation of the MD, TD and 45° off-axis specimens. The nominal dimensions of the specimen were 45 mm in length and 5 mm in width.

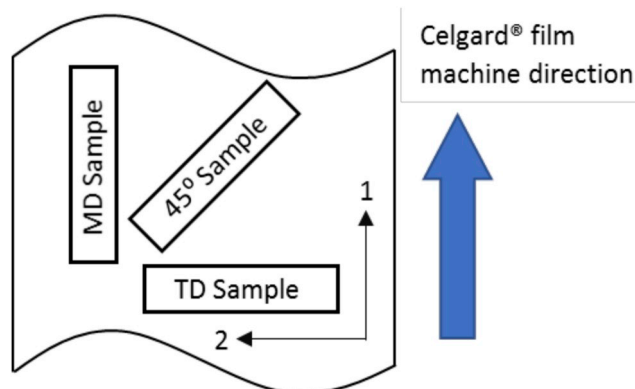


Fig. 2. The MD, TD, and 45° off-axis samples.

For an orthotropic solid, the two in-plane material directions are defined as 1 and 2 directions. In later discussions, the MD and TD are also referred to as 1 and 2 directions, respectively.

### 2.2. Loading

All experiments were performed with a TA 2980 DMA in tensile mode. The DMA measures the mechanical properties of materials as a function of time, temperature, and frequency. It is widely used in viscoelastic characterization of polymers.

Two types of experiment were performed. The tensile stress-strain curves were measured at three loading rates at 0.1, 1, 10 N/min. The viscoelastic properties were measured in tensile creep mode. The experimental set-up was the same for both experiments. Fig. 3 shows a specimen mounted in the DMA tensile clamp. The test length of the specimen was about 15 mm. All measurements were conducted at the ambient temperature.

### 2.3. Strain measurement

The measurement of the Poisson's ratio and shear properties requires measuring strains in other orientations in addition to the longitudinal direction. The DIC technique was used in these measurements. DIC is a noncontact, full field experimental technique. In this method, the surface of the specimen is covered by random speckle patterns.



Fig. 3. A sample in tensile clamp.

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