# Processing-structure-property investigation of HDPE precursor films containing row-nucleated lamellar structure

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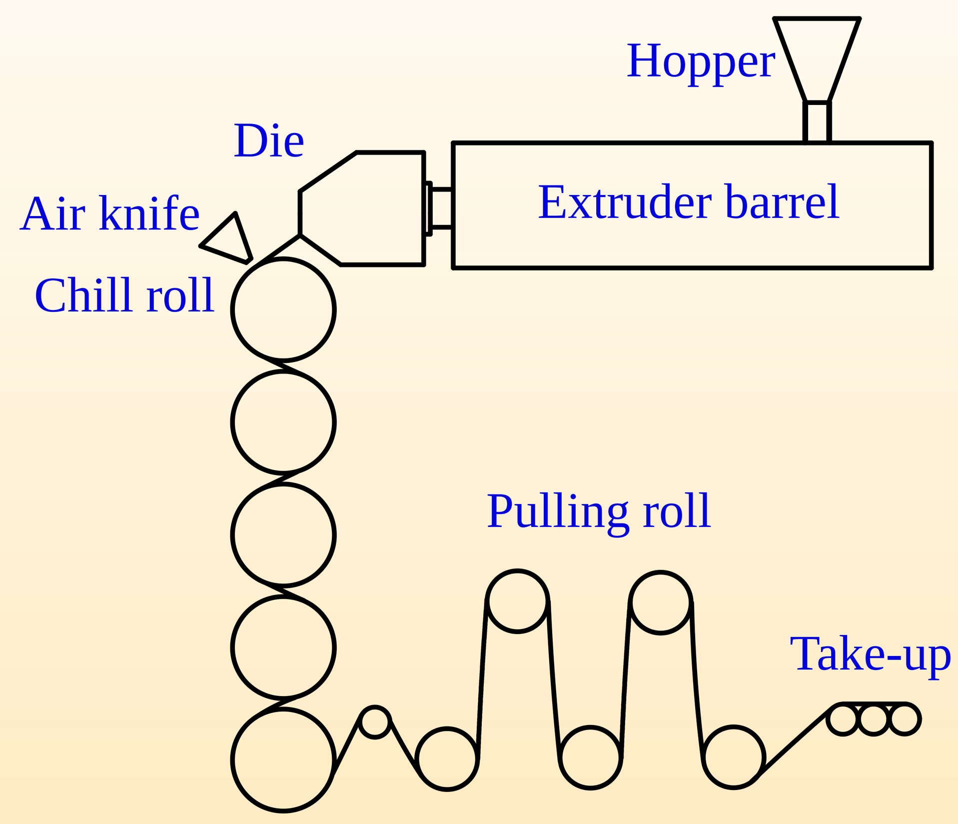
## Introduction

Most of commercially available lithium battery separators are polyolefins microporous such as PP and PE microporous. Three processes are used for making polymeric microporous membranes: solution casting, particle stretching, and dry-stretching. Costly processes and difficulties in dealing with solvent and particle contaminations are main drawbacks of the first two methods. However, the dry-stretching process is cheaper and less pollution which is based on the stretching of a polymer precursor film containing a row-nucleated lamellar structure. The lamellar structure of extruded PE precursor films predetermines their hard elastic properties, and correspondingly, their ability to pore formation during the uniaxial extension of the samples. Therefore, to have a quantitative analysis on the processing–structure–property relationship of hard elastic precursor films is crucial.

While there have been many studies investigating on make microporous membrane using the hard elastic materials PP, there has been little work carried out on PE. The PE separator, which melts down at 120-130℃, is expected to provide greater safety than the PP separator, which has a fuse temperature of 165℃.

In this work, the extrusion and casting parameters to prepare PE precursor membranes were investigated. By utilizing WAXD、SAXS techniques and mechanical analysis explored the relationship among casting processing, microstructure of precursors and mechanical properties.

## Experiments and data




Material: HDPE HTA001

Temperature in extruder barrel: 150, 195, 220, 230, 220, 230

Processing variables:

(1) Draw ratio

(2) Die temperature

(3) Distance from die to chill roll

(4) Temperature on chill roll

**Figure 1.** A schematic of preparation of HDPE precursors process

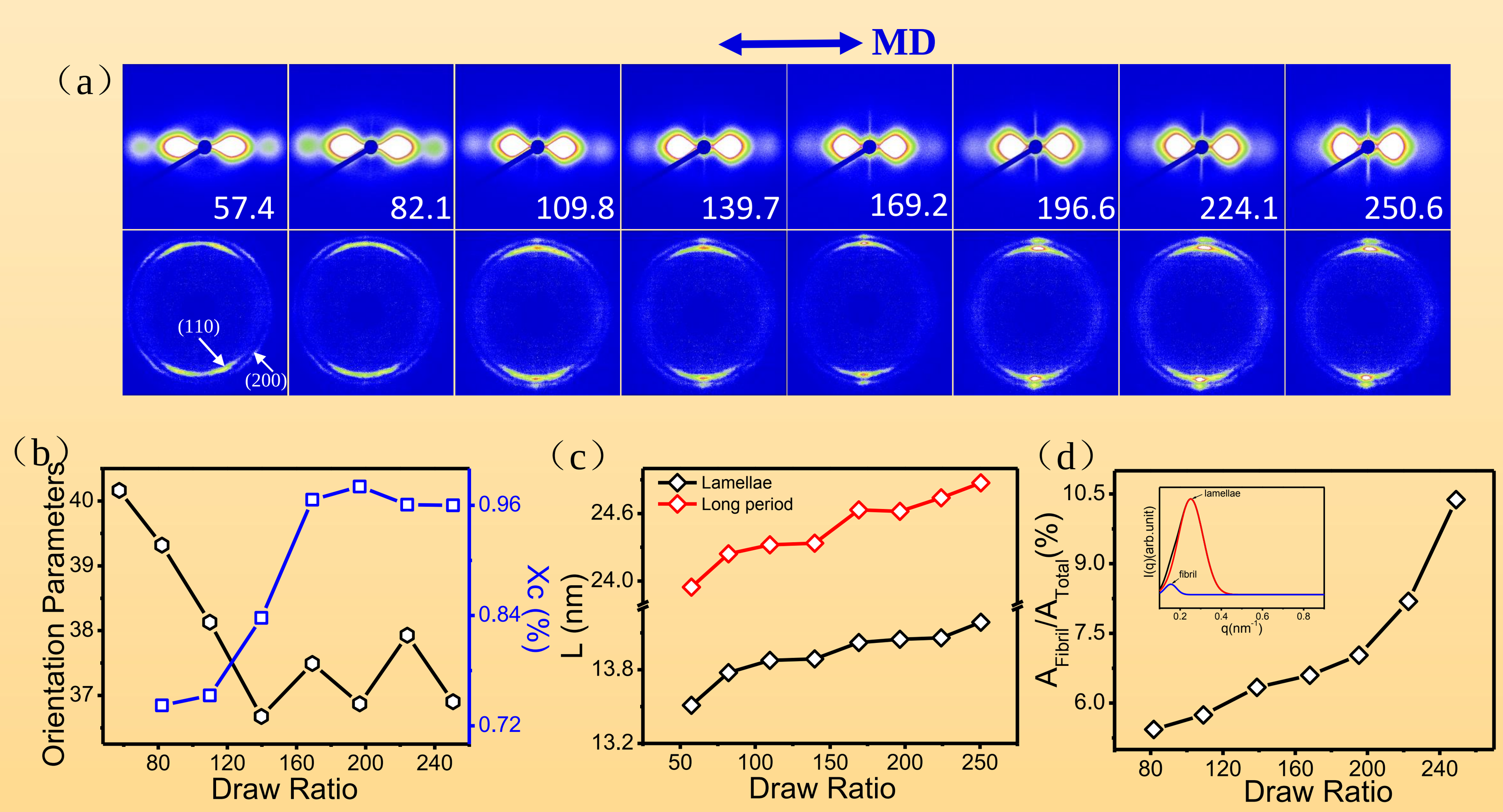



**Figure 2.** Precursor films microstructures vary with draw ratio obtained by SAXS and WAXS (a) SAXS and WAXS diffraction patterns (b) Orientation parameters and degree of crystallinity (c)Long period and the thickness of lamellae (d) The percentage of fibrous crystal (the method to calculate the content of fibrous crystal was insert)

## Discussion and Conclusion

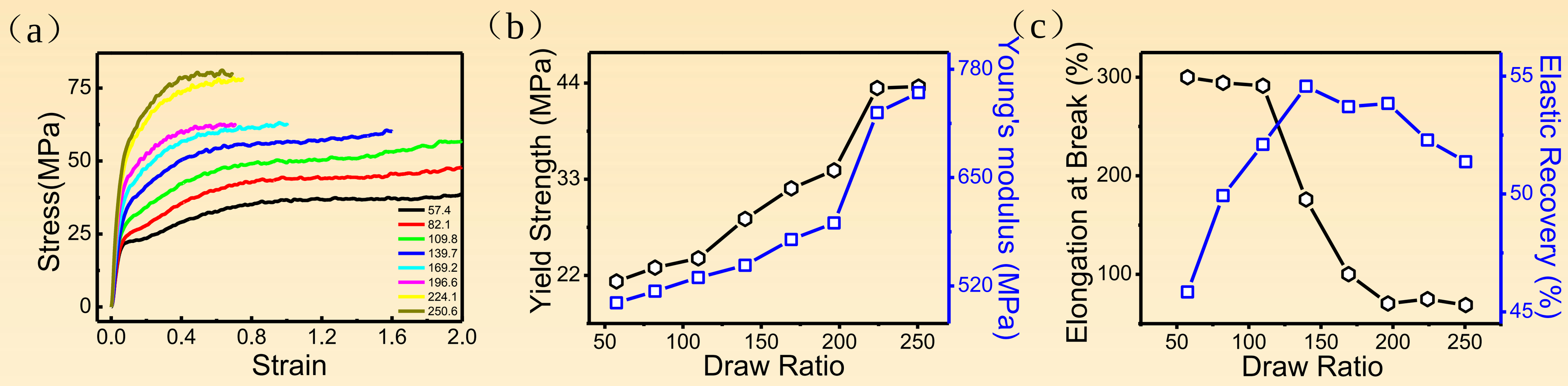



**Figure 3.** The mechanical properties of precursors vary with draw ratio (a) stress-strain curve (b) Yield strength and Young's modulus (c)Elongation at break and elastic recovery

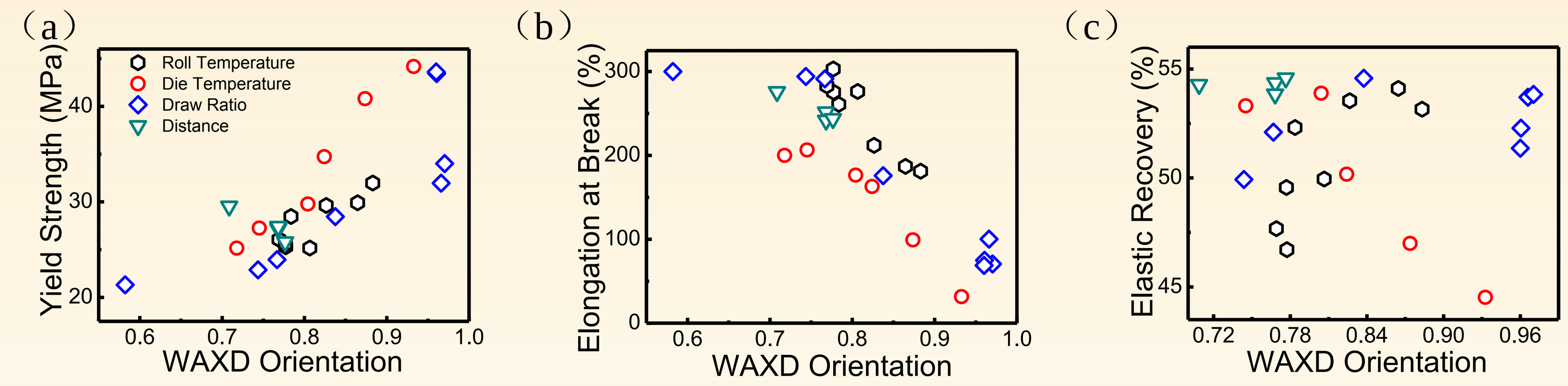



**Figure 4.** The mechanical properties (a)Yield strength (b)Elongation at break (c)elastic recovery of precursors vary with orientation parameters obtained by WAXS

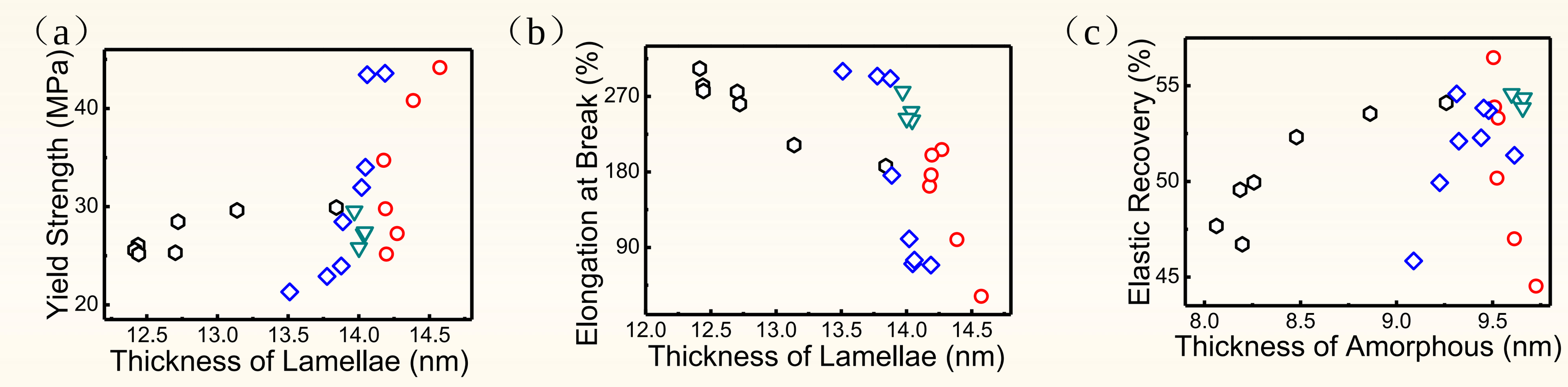



**Figure 5.** The mechanical properties (a)Yield strength (b)Elongation at break (c)elastic recovery of precursors vary with the thickness of lamellae and amorphous (Hexagon: Roll Temperature, Circle: Die Temperature, Diamond: Draw Ratio, Downtriangle: Distance from die to chill roll)

Precursor films with row-nucleated lamellar structure have been prepared successfully. Draw ratio($\lambda$) is the greatest impact factor among the processing variables for regulating the microstructure of precursor films. When $\lambda$ <82.1, twisted lamellae formed when HDPE were melt-extruded and crystallized under low stress, however when $\lambda$ >82.1, unpleasant shish signal appeared(Figure 2a). Orientation parameters, long period and the percentage of fibrous crystal increase with draw ratio(Figure 2b-d), because tensile is a dominant role in the precursor film formation process. Meanwhile the microstructures of extruded precursors determine their mechanical properties. In addition orientation parameters and the thickness of lamellae are the decisive structures to properties (Figure 4-5).

## Outlook

In theory, fibrous crystal is undesirable in the process of separating lamellae during pore-formation, but it can induce row-nucleated lamellar structure better to promote a higher orientation function. This is a rather contradictory relationship, so it is a great challenge to make the precursors with perfect structure.

There are many controversies regarding to mechanism of initiation of cavitation. If we can establish the corresponding relationships between molecular parameters and structure of precursor, crystal morphology and mechanism of micropore formation, there is no doubt that it can guide us to produce high-performance microporous membrane.

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