



The information and data contained in this document were prepared by a technical committee of the Association. The committee and the Association assume no liability or responsibility in connection with the use of such information or data, including but not limited to any liability under patent, copyright, or trade secret laws. The user is responsible for determining that this document is the most recent edition published.

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Rheological measurements for characterization of polyolefins: low-density polyethylene (LDPE) for extrusion coating

1. Introduction

1.1 In extrusion coating a thin molten polymer film is coated on some kind of substrate. At high extrusion coating speed, even a minor disturbance on the melt web causes major quality problems, which very rapidly lead to large quantities of waste. Therefore, higher coating speeds require polymers with high and even quality in order to avoid waste due to polymer edge instability and web break.

1.2 The rheology-related phenomena that may cause problems in extrusion coating are neck-in (NI) and draw-down (DD). The neck-in is the reduction of the film width and may cause uncoated areas on the substrate. The neck-in is less if the melt elasticity is high. The draw-down is the ability of the melt to be drawn to thin films without breaking. Draw-down is favored by a melt that is more viscous than elastic.

1.3 Changes in the molecular structure have a great impact on the rheological and processing properties of a polymer. Storage modulus and zero shear viscosity have been found to be useful parameters to predict the extrusion coating performance of LDPE. Storage modulus quantifies a material's ability to store energy elastically and so to deform under stress. As the storage modulus increases, the ability of the material to deform decreases. Zero shear viscosity is related to molecular weight; a small change in molecular weight results in a big change of zero shear viscosity. Knowing this, there is a relation between storage modulus, zero shear viscosity, draw down and neck-in. As storage modulus and zero shear viscosity increase, neck-in decreases and so does the draw down capacity, meaning a lower line speed at which the polymer can be drawn down to a desired thickness.

2. Scope

In optimizing the extrusion coating processing performance it is of utmost importance to balance the rheology of the polymer. This method describes how rheological measurements can be used to characterize LDPE. The

Approved by the Standard Specific Interest Group for this Test Method



Documents
Document Sharing Hub

storage modulus and zero shear viscosity have been found to be useful parameters to predict the extrusion coating performance of LDPE.

3. Significance

LDPE from high-pressure autoclave reactors for extrusion coating with a Melt Flow Index (MFI) range from 4 to 15 g/10 min., and with a density of 915-924 kg/m³ has for a long time been considered as a uniform commodity. However, as evolution goes towards faster coating lines, differences in processing performance between various LDPE grades have been observed. Various LDPE grades behave differently in the coating process due to different rheological properties.

4. Definitions

- 4.1 The storage modulus and zero shear viscosity are determined from linear viscoelastic data.
- 4.2 *Storage modulus*: The storage modulus at a loss modulus of 500 Pa measured at 170°C expressed in Pa.
- 4.3 *Zero shear viscosity*: The constant plateau value of the complex viscosity at low frequency measured at 170°C expressed in Pas. The zero shear viscosity defined in this method is a fitting parameter of the Cross model. This zero shear viscosity could be different from the zero shear viscosity determined at long creep tests.

5. Apparatus

- 5.1 *Laboratory hot press*.
- 5.2 *Controlled-stress rheometer or controlled-strain rheometer* with parallel plates ($\varnothing = 25$ mm) geometry operated in the oscillatory mode. In using the controlled-stress mode a sinusoidal torque of constant amplitude is applied and the resultant sinusoidal displacement function and the phase shift between the torque and displacement functions are determined.
- 5.3 In using the controlled-strain mode a sinusoidal displacement of constant amplitude and the resultant sinusoidal torque function and the phase shift between the torque and displacement functions are determined.
- 5.4 Measurements of the oscillatory rheological properties of specimens in accordance with this standard are restricted to the linear viscoelastic region of behavior. Measurements are done under nitrogen atmosphere to prevent degradation/crosslinking of the sample.

6. Test specimens

- 6.1 Compress mold pellets of sample at 150°C with a preheating for 60 s, a linear increase of pressure up to 50 bar during 50 s. Keep the pressure at 50 bar for 40 s. Cool for 30 s with cooling cassette (cold water circulation).
- 6.2 Stamp discs with a diameter of 25 mm and a thickness of 1.2–1.5 mm from the pressed specimen. Make five test specimens.

7. Conditioning

Condition the sample at 170°C for 4 min in the rheometer before starting measurements. The gap should be preferably 0.8–1.2 mm. After 2 min of the conditioning time, open the oven and carefully trim the edges.

8. Procedure

Perform a frequency sweep between 20–0.01 Hz (125–0.06 rad/s) divided logarithmically into 18 steps at 170°C in the linear viscoelastic region. The linear viscoelastic region is determined by a stress or strain sweep