



Characterization of a polyethylene–polyamide multilayer film using nanoscale infrared spectroscopy and imaging[☆]



Mauritz Kelchtermans^a, Michael Lo^b, Eoghan Dillon^b, Kevin Kjoller^b, Curtis Marcott^{c,*}

^a ExxonMobil Chemical Europe Inc., Hermeslaan 2, B-1831 Machelen, Belgium

^b Anasys Instruments, Inc., 325 Chapala Street, Santa Barbara, CA 93101, USA

^c Light Light Solutions, P. O. Box 81486, Athens, GA 30608-1484, USA

ARTICLE INFO

Article history:

Received 5 August 2015

Received in revised form 30 October 2015

Accepted 19 November 2015

Available online 22 November 2015

Keywords:

Infrared
Atomic force microscopy
Multilayer films
Tie layers
Polyethylene
Polyamide

ABSTRACT

Atomic force microscopy (AFM) and infrared (IR) spectroscopy have been combined in a single instrument (AFM-IR) capable of producing IR spectra and absorption images at sub-micrometer spatial resolution. This new device enables cross sections of multilayer films to be spectroscopically characterized at levels not previously possible. In particular, it was possible to observe nanoscale IR spectroscopic differences, as well as thermal and mechanical property differences, in the tie layers located between the individual polyethylene and polyamide layers of a multilayer cling film of initially unknown structure. It also appears that a two- μm -thick barrier layer between two polyamide layers near the center of the multilayer cling film consists of an ethylene-vinyl alcohol copolymer. Mechanical stiffness and thermal property differences are also observed between the various layers in the film. This powerful capability should prove generally useful for reverse engineering complex unknown multilayer film materials, as well as in aiding the intelligent design and coextrusion of superior multilayer film materials.

© 2015 Elsevier B.V. All rights reserved.

1. Introduction

Multilayer films are of great importance in many products, especially packaging materials. As multilayer films become thinner and the number of individual layers increase, there is an ever increasing need to characterize these structures. Chemical treatment of the polymer film surfaces and/or the addition of tie layers, can improve the adhesion of the main polymer film layers, which are often incompatible, i.e., not naturally adhering to each other. Without proper binding between chemically different polymer film materials, the layers may eventually pull apart, resulting in failure of the packaging film. Techniques that enable the exploration of the thermal, mechanical, and chemical nature of multilayer films at the important boundary region between layers will aid not only in the development of better packaging materials, but also to reverse engineer all kinds of already manufactured multilayer films.

Fourier transform infrared (FT-IR) attenuated total reflection (ATR) [1,2] spectroscopy is a technique which has been used by many researchers to chemically characterize the surface of

polymer film samples. It can be useful for identifying tie layers and surface treatments of the internal layers of multilayer films, assuming they can be delaminated cleanly to expose the internal layers, and assuming those layers are thick enough to distinguish them from the subsurface layers. FT-IR microspectroscopy is also widely used for characterizing cross sections of multilayer films. It provides information about the chemical nature of the individual polymers layers, as long as they are at least a few micrometers thick. The spatial resolution limit of conventional transmission FT-IR microspectroscopy is limited by fundamental diffraction limitations associated with the wavelength of light used to make measurements in the mid-IR spectral range (2.5–14 μm). Studies using ATR objectives coupled with FT-IR microscopes equipped with focal-plane array (FPA) mercury–cadmium–telluride (MCT) detectors have been used to characterize layers as thin as a few microns when a high refractive index internal reflection element (IRE) like Ge is used as the ATR objective, but it is yet not possible to reach sub-micrometer spatial resolution in FT-IR microscopy, even when using synchrotron radiation as the light source [1,2].

The spatial resolution of Raman spectroscopy depends on the laser excitation wavelength, but is generally higher than FT-IR microspectroscopy. However, Raman spectroscopy suffers from the lack of spectral sensitivity for polar substrates which are often used in multilayer films.

[☆] Selected paper from 8th International Conference on Advanced Vibrational Spectroscopy, 12–17 July 2015, Vienna, Austria.

* Corresponding author.

E-mail address: marcott@lightlightsolutions.com (C. Marcott).

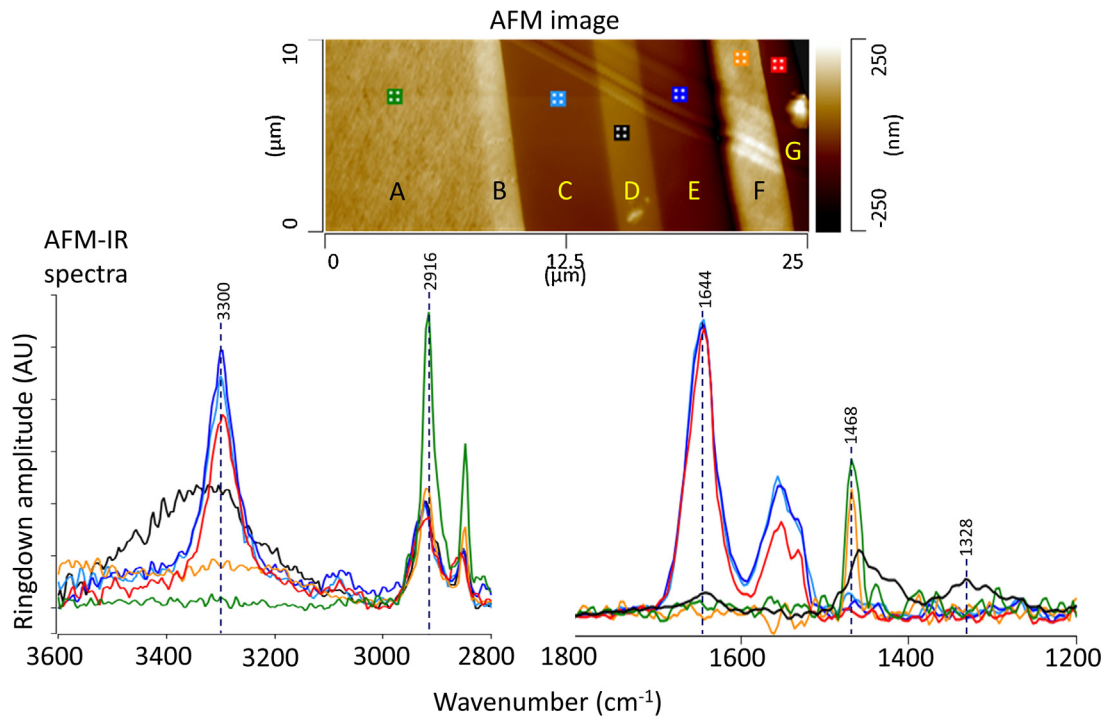


Fig. 1. Tapping mode AFM height image and AFM-IR spectra from seven layers of a multilayer cling film mainly consisting of polyethylene (PE) and polyamide (PA). The colored marker locations on the AFM image are locations where the corresponding AFM-IR spectra of the same color were obtained.

The combination of an atomic force microscope (AFM) with a tunable IR laser source in a single instrument has been shown to be capable of collecting sub-diffraction-limited measurements of IR absorption spectra on sample regions as small as $20\text{ nm} \times 20\text{ nm}$. This technique, known as AFM-IR, is based on the photothermal induced resonance effect (PTIR) [3–8].

The AFM-IR technique used in this work focuses radiation from a tunable IR laser source onto a location on the sample top surface

from above [3–8]. Unlike conventional FT-IR spectrometry, where a high-sensitivity MCT single-element or FPA detector senses the internally reflected IR beam, the AFM-IR approach uses a sharp AFM tip attached to a cantilever to detect the rapid thermal expansion of the sample caused by absorption of short (10 ns) pulses of IR radiation at a given wavenumber. When the monochromatic laser radiation occurs at a wavenumber that excites molecular vibrations in the sample, the light is absorbed

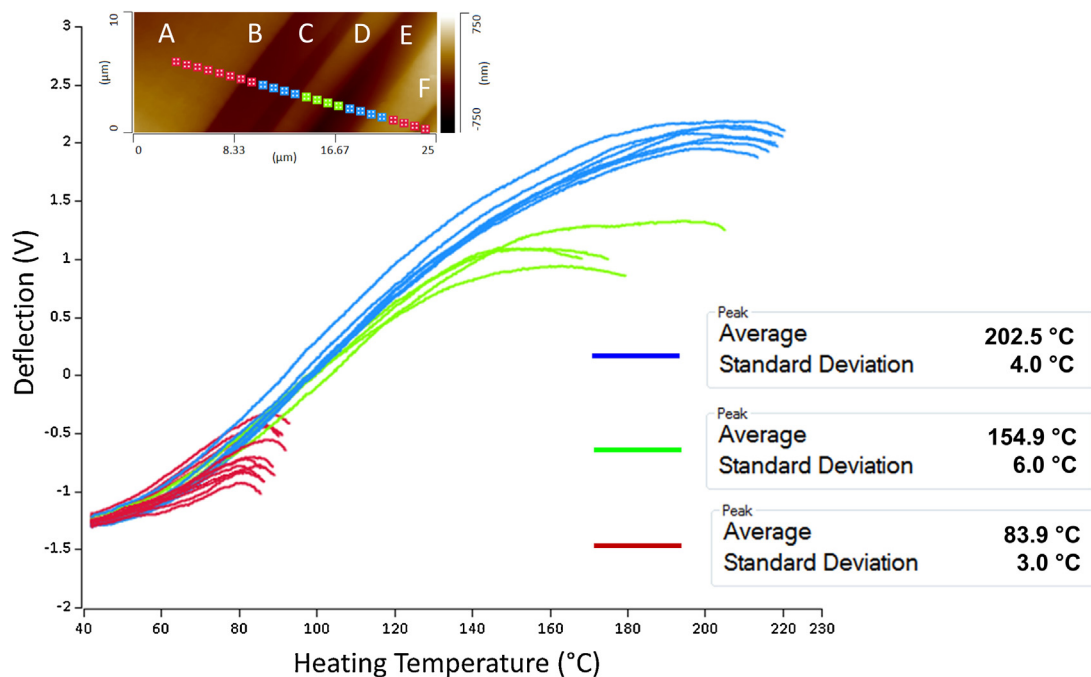


Fig. 2. Contact mode AFM height image and nanoscale thermal analysis of a seven-layer cling film sample mainly consisting of PE and PA. The colored marker locations on the AFM image (spaced $1\text{ }\mu\text{m}$ apart) represent locations where the nanothermal measurements were recorded. The red marker locations with lower transition temperature correspond to the PE layers.

Download English Version:

<https://daneshyari.com/en/article/1250101>

Download Persian Version:

<https://daneshyari.com/article/1250101>

[Daneshyari.com](https://daneshyari.com)