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A comparison of vertical scanning interferometry (VSI) and atomic force microscopy (AFM) for characterizing membrane surface topography

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Abstract

In this paper, vertical scanning interferometry (VSI) and atomic force microscopy (AFM) were used to characterize the topography of several nanofiltration and reverse osmosis membrane surfaces. Comparing roughness results from the two different characterization techniques revealed unique results for the various membrane surfaces. Roughness values tended to be higher from the interferometry measurements compared to those from AFM measurements for the same membranes. This was attributed to the inability of the AFM to capture dramatic changes in surface height of several microns or more. Based on interferometric measurements surface roughness was also found to increase with increasing scan-size up to a scan-size of 250,000 μm^2 after which it remained relatively constant. Because such large scan-sizes are too large to be captured through AFM measurements interferometry appears to provide a more comprehensive characterization of membrane surface roughness.

Keywords: Interferometric method; AFM; Roughness; Scan area; Membrane surface topography

1. Introduction

There currently exists a variety of analytical tools for characterizing the morphology of polymeric and ceramic membrane surfaces [1–4]. Some of the more popular surface characterization techniques for membranes include scanning electron microscopy (SEM) and atomic force microscopy (AFM). Each of these tools is capable of providing atomic level quantitative analyses of the morphological characteristics of membrane surfaces. For instance, using these tools it is possible to characterize membrane properties like surface roughness [5–8], porosity and pore size distribution [9–11], and deposit layer thickness [12]. Such characterization is important given findings demonstrating the critical role of these characteristics in determining membrane performance [6,7,13]. Of the surface characterization techniques that are available to membrane scientists, AFM has been used due to the ease of sample preparation and its ability to characterize membrane surfaces in both wet and dry environments [2,5,7,9–11,14–16].

An atomic force microscope operates using a combination of principles from the scanning tunneling microscope and the stylus profilometer [17,18]. Here, a sharp tip, with a radius of around 50–100 nm, is scanned over a surface with feedback mechanisms that enable piezo-electric scanners to maintain the tip at a constant force (to obtain height information), or height (to obtain force information) above the sample surface. Tips are typically made from silicon nitride and extend down from the end of a flexible cantilever. Surface morphology and/or surface interactions are measured based on the vertical deflection of the cantilever. The resolution of the AFM is determined by the sharpness of the tip and typically approaches the atomic scale. Thus, AFM may be used to provide high-resolution information regarding membrane surface morphology in addition to other characteristics. Nevertheless, despite the many advantages of AFM and its success in characterizing membrane surfaces a number of limitations do exist.

One of the principle drawbacks of the AFM is the relatively small area that can be scanned at any given time. For instance, the maximum scan area for most AFMs is approximately 100 μm^2.

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This limitation is principally due to the operational set up of the AFM and the size of the single cantilever used to produce the AFM image. Such a limited scan-size makes it difficult to determine how representative the measured image may be of the surface at large. For instance, Boussu et al. [9] found that membrane surface roughness increased with increasing scan-size, suggesting that roughness statistics derived from relatively small scan areas may be misleading. Another issue pertains to the distortion of surfaces features as a result of tip convolution. Additionally, it is difficult to characterize surfaces where the changes in height are dramatic ($\Delta > 5\ \mu m$) in which contact may be lost between the tips and sample, on the tip may become damaged. Further drawbacks of the AFM include the time required to obtain an image and the operation of the instrument, which are both rather intensive.

Optical interferometry is a rather new technique that may be used to characterize membrane surfaces with regard to surface morphology and structure [19]. Optical interferometry is a type of microscopy where nanometer level characteristics of a sample surface may be characterized through the interpretation of light reflected from a surface. With optical interferometry it is possible to obtain scan-sizes of up to a square millimeter with a vertical resolution of approximately 2 nm. The larger scan-size made possible with optical interferometry, allows for a more comprehensive analysis of surface roughness and is drastic improvement of the small scan-sizes (typically 100 μm^2) that are possible with an AFM. In this respect, the impact of scan-size on calculated roughness statistics may be determined, providing new insight and perspective into the interpretation of values derived from much smaller scan-sizes, as is the case for AFM generated values.

In this paper, roughness statistics generated by AFM measurements are compared to those determined using optical interferometry for several nanofiltration and reverse osmosis membranes. The impact of scan-size on the calculated roughness statistics is used to evaluate the ability of AFM to fully characterize membrane surface roughness as a function of length scale. The role of optical interferometry as a characterization technique for membrane surface is compared with that of AFM.

2. Experimental

2.1. Membranes

This study examined three commercially available nanofiltration (NF) and reverse osmosis (RO) membranes; GE Osmonics HL (Minnetonka, MN), the DOW Film Tec NF70 (Minneapolis, MN) nanofiltration membranes, and the Hydranautics LFC-1 (Oceanside, CA) reverse osmosis membrane. These membranes were selected as they represent a range of surface morphologies as determined in previous investigations [13,20,21]. Each membrane was supplied as dry flat sheets and was stored accordingly until used.

2.2. AFM analysis

All AFM experiments were carried out using a Park Scientific Instruments (Sunnyvale, CA) Autoprobe CP atomic force microscope. Measurements were performed on dry membrane samples under ambient atmospheric conditions. Silicon cantilevers with integrated pyramidal tips (Model #: MPP-11100, VEECO Instruments Inc., Fremont, CA) were used to image membrane surface topography. The membrane surfaces were imaged in tapping mode. At least five separate scans, each covering an area of 100 μm^2, were acquired on each membrane to

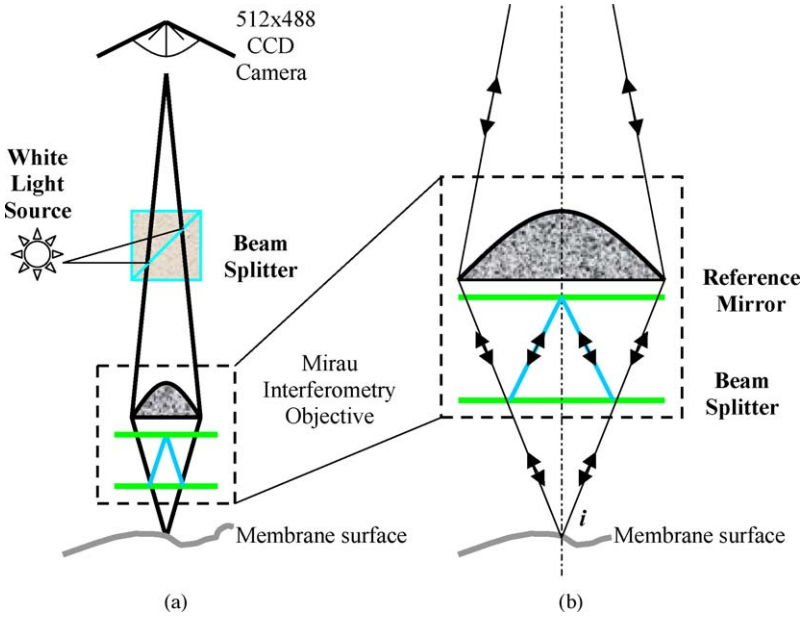

Fig. 1. (a) Sketch of a double-beam Mirau interferometer with CCD camera. (b) Details of (a) showing the light path in the Mirau interferometer and to a sample surface [19].