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The role of wetting on the water flux performance of microsieve membranes

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Abstract

Microsieve membranes are microfabricated devices that possess a high and controlled porosity. Due to the extremely high product throughput, microsieves are very sensitive systems in terms of flux decline during clean water measurements. The goal of this investigation was to study the influence of the surface properties of silicon nitride microsieves and the hydrodynamic parameters on the clean water flux performance. First, we studied the properties of bare silicon nitride dices. Contact angles and XPS measurements clearly demonstrated that silicon nitride experiences aging and variations in its surface properties, making it necessary to hydrophilize and homogenize the substrates. To understand the role of the surface properties on the water permeation, the water flux through unmodified, hydrophilized and hydrophobized silicon nitride microsieves was studied as a function of the wetting procedure and the presence of air in the feed liquid. For most surfaces, a good wetting protocol and the exclusion of contaminating particles and air from the system led to stable fluxes. Hydrophobized membranes presented low performance when they were poorly wetted. In these cases, air was massively deposited on the surface, since the low working pressures made the filtration sensitive to air bubbles. For highly hydrophobized membranes (contact angle, $\theta = 112^{\circ}$) no stable fluxes could be measured due to rapid dewetting.

Keywords: Silicon nitride; Microsieves; Wetting; Surface properties; Flux decline

1. Introduction

Ideally, a microfiltration membrane should combine high porosity and well-defined pore size, together with a thin selective layer. However, conventional microfiltration polymeric membranes do not fulfill these requirements and they suffer from the trade-off between permeability and selectivity. Consequently, there is a great need for an alternative to the traditional membranes used in industry. Specialty high flux membranes such as microsieves can be a new choice for several applications in the membrane field. They present a very well-controlled structure and pore size; their high porosity leads to very high fluxes and they can be operated at extremely low pressures, therefore providing an energy and economically friendly process.

In the past years silicon micromachining technology has provided a promising tool as a membrane fabrication method. In this paper, we study the properties of a special generation of inorganic membranes, microsieves, which can provide an additional technology for bioseparations, for example. Silicon nitride microsieves are produced by techniques well known in the semiconductor industry, including standard mask lithography or laser interference lithography [1,2]. The peculiarity of microsieves lies in the thin resistant selective layer, the high surface porosity and completely regular patterned pore structure, combined with mechanical strength and improved filtration performance. Since the fabrication techniques provide full control on the membrane design, microsieves contain pores with the same size and shape all along their surface. With these high-flux membranes a high product recovery can be achieved under ecological and mild conditions, without degrading or altering the functional properties of the desired component.

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Due to its optimal thermal, electrical, optical and mechanical properties [3], silicon nitride is a common material used in numerous applications such as optical waveguides, insulators, and other micromachined devices. However, it is not completely inert. Silicon nitride surfaces are sensitive to chemical compositional changes such as oxidation [4]. Therefore, the material has to be altered to obtain a well-defined surface with a constant quality for an optimal membrane performance. For this, surface modification strategies, for instance silanization or plasma treatment, offer an attractive approach. Such treatments will deliver tailored surfaces that can be used in aqueous (milk defatting, water purification in cleanrooms, etc.) as well as non-aqueous (solvent purifications, water-in-oil emulsifications, etc.) applications.

Wetting of such microfabricated architectures is a very important aspect in order to obtain maximum performance. The main goal of this research is to study the influence of the surface properties on the microsieve performance in aqueous media, as well as the establishment of the critical parameters that can disturb the flux or induce flux decline in aqueous systems. Surface characterization is performed using techniques such as contact angle measurements, scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). Several parameters such as wetting procedure and feed composition are varied to determine their influence on the membrane performance.

2. Experimental

2.1. Materials

Silicon-rich silicon nitride (Si_xN_y) dices and porous microsieve membranes provided by Onstream BV, 1 cm × 1 cm, were used as substrates for modification. The microsieve structure was fabricated in a 1 μm thin Si_xN_y layer. Below the sieve structure a 500 μm silicon support is present, which incorporates 30 μm high permeate channels. The membranes used have a pore size of 1.2 μm, a calculated porosity of 9.2% and a pitch of 2.12 μm. A scanning electron microscope image of the membrane surface used in this work is presented in Fig. 1. A sieve bed (Fig. 1a), a pore grid

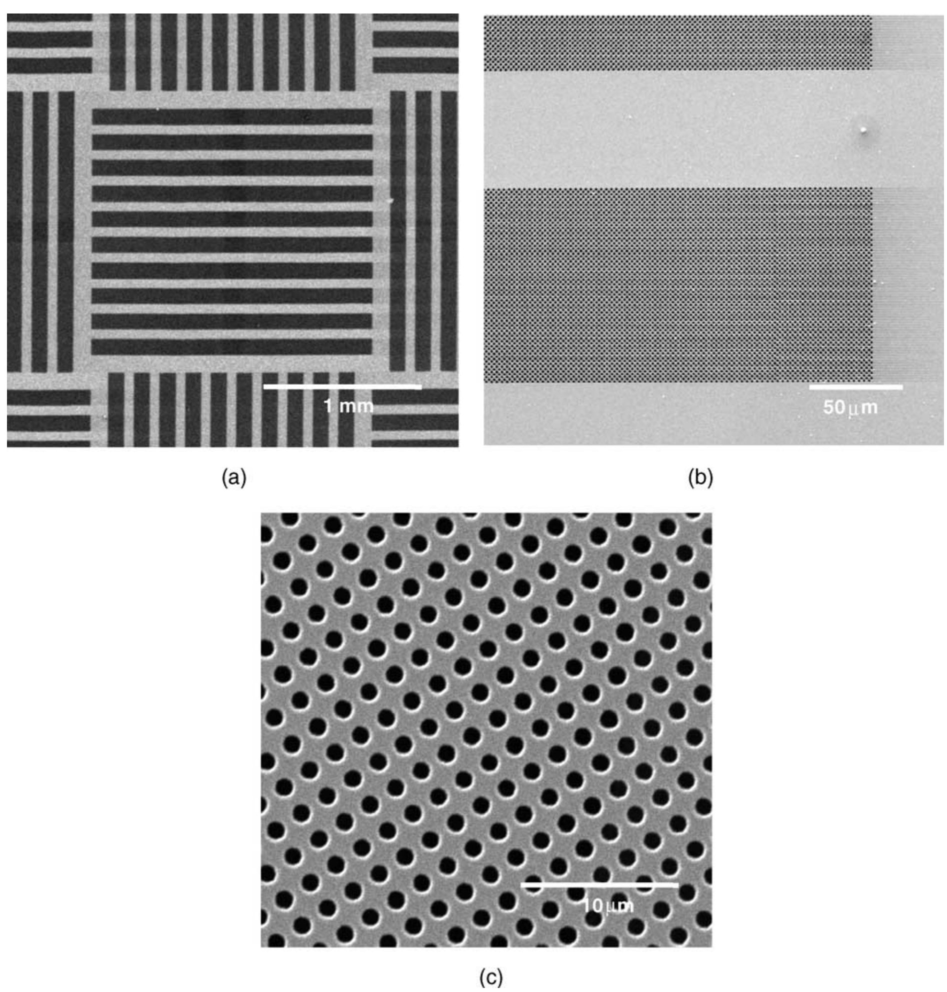

Fig. 1. SEM images of a silicon nitride microsieve surface, (a) sieve bed, (b) pore grid and (c) close-up of pores.