Available online at www.sciencedirect.com
ScienceDirect
Nuclear Instruments and Methods in Physics Research B 254 (2007) 160–164
NIM B
Beam Interactions with Materials & Atoms
www.elsevier.com/locate/nimb

Investigation of the breakthrough point of ion track etching by capacitometry

J. Chen [a,*], D. Fink [a], S. Dhamodaran [b]

[a] Hahn-Meitner-Institut Berlin, SE2, Glienicker Str. 100, D-14109 Berlin, Germany
[b] School of Physics, University of Hyderabad, Central University (P.O), Hyderabad 500 046, India

Received 24 May 2006; received in revised form 10 October 2006
Available online 22 November 2006

Abstract

Nanoscale size definition and flexible device structures are presently among the most ambitious development goals in the semiconductor field. One approach to attain these goals is based on the use of hybrid structures, combining the flexible etched ion track templates as substrates with the device functions of filled inorganic semiconductor.

It is a critical issue to determine precisely the moment of breakthrough in order to use the etched ion track templates to fabricate vertical nano electronic device.

This study tries to shed some light upon these processes by means of a novel approach, the so-called capacitometry measurement for investigating the breakthrough moment and etching processes. It is shown that the capacitometry is a simple but quite reliable technique to determine precisely the moment of track etching breakthrough.

PACS: 29.27.Ac; 61.80.−x; 81.07.−Bc; 85.35.−p

Keywords: Swift heavy ions; Ion tracks; Polymers; Cleaning; Sensitization; Etching; Incubation time; Track breakthrough

1. Introduction

The fabrication of nano electronic devices has spurred considerable activity in material science. Recently it became clear that ion tracks can also be used in nanotronic technology since the etched ion tracks have just the right size for nanostructuring: the etched ion track diameter is in the order of a few tens of nm and the track length can be varied from a few nanometers up to several micrometers by choosing the appropriate sample thickness [1–3].

The identification of the moment of breakthrough is a key point for producing the hollow channels in nanoscale within irradiated materials, which is an important step for fabrication of this kind of nano electronic devices.

One of the authors tried some years ago to get some more insight into these processes, by following the depth distribution of the etchant and neutral solution when penetrating into ion-irradiated polymer foils. A summary of these studies is given in [4].

A capacitometer records correct capacity values only if the measured capacities are not shortcut by parallel Ohmic resistances. The latter occurs, however, at the moment of breakthrough of the etchant. From that moment on, the conducting connections exist through out the tracks, which show up in their equivalent circuits as Ohmic resistances parallel to the track capacity. Consequently, the capacitometer does no longer indicate the true capacity value, but another one which we denoted as "effective capacity". Typically, the values of "effective capacity" recorded by the capacitometer may deviate strongly from the true capacities. As concerning the point of etchant breakthrough of the tracks, it is marked clearly by a spontaneous decrease

* Corresponding author.
E-mail address: jie.chen@hmi.de (J. Chen).

0168-583X/$ - see front matter © 2006 Elsevier B.V. All rights reserved.
doi:10.1016/j.nimb.2006.10.064

in effective capacity. Please note that the exact value of the effective capacity depends sensitively on the type of capacitometer used. Specifically, for the capacitometer used by us, the effective capacity indicated for a test capacity of 1 μF changed by a factor ~3, and for 10 μF even by a factor ~10, when shortcutting the test capacities by an Ohmic resistance of 1 kΩ. This point will become important in the subsequent discussion.

In spite of these ambiguities, capacitometry is excellent to explore the processes going on in the early stages of ion track etching [5] and to find precisely the etching breakthrough point. Here we report our recent research results on this topic and discuss the procedure of the actual etching.

2. Experimental

We took 8 μm thick polyethylene terephthalate (PET) foils obtained from two different companies: the DuPont Teijin Films Luxembourg SA (named as foil-**I** in the following paper) and the Oxyphen GmbH (named as foil-**II** in the following paper). These foils had been irradiated homogeneously by 390 MeV Xe^{21+} ions up to a fluence of $\sim 3.8 \times 10^7$ cm^{-2} at the swift heavy ion accelerator "ISL" of our institute. Thereafter the foils were cut into ~3 cm × 3 cm large pieces and subject to some of pre-treatments.

We used an ISO-Tech9023 capacitometer from RS Components GmbH in our experiments. This instrument delivers positive rectangle pulses of 2 V height and 1 ms, 10 ms or 100 ms width and 1.2 ms, 12 ms or 120 ms repetition time each for the measuring ranges {200 pF–2 μF}, 20 μF, and {200 μF–20 mF}, respectively. The sample response is then transferred to the corresponding capacity values.

Additionally, we measured the current through the tracks after breakthrough with a home-made nA-V meter model HMI EM40 and a 1.5 V source, by which one can record currents from less than 0.1 nA till 100 μA.

The different pre-etching procedures included cleaning with 4 M HNO_3 in an ultrasonic bath for 2 min, rinsing the sample with distilled water, cleaning it with ethanol in an ultrasonic bath for 2 min, and UV treatment for 4 min by running a RF glow discharge in rough vacuum ($\sim 10^{-2}$ Pa) with 30 W 10 MHz applied. As in the glow discharge plasma both UV irradiation and ion sputtering are acting simultaneously on the polymer foil, we tried to separate these effects from each other by shielding the target foil in one experiment from the energetic ions by depositing on top of it a thin quartz glass.

The pre-treated foils were then subject to track etching in 1.2 M or 7 M NaOH at 55 °C from both sides of the foil. The temperature was stabilized in a water bath. This etching process was followed capacitometrically. For this sake the samples were mounted in the middle of an etchant-filled Teflon vessel so that they separate the vessel leakage-free into two equivalent compartments according to Apel [6]. In each chamber a ~1 cm^2 large Pt electrode was immersed which was connected to the above-mentioned capacitometer. The latter has an accuracy of about ±4% for small (200 pF–20 μF), and up to ±20% for large (⩾200 μF) capacities. After the onset of etching, every half minute the capacity of the etching vessel was recorded.

3. Results and discussion

There is an obvious difference in dielectricity coefficients ε of the polymers ($\varepsilon \sim 2 \ldots 3$) and the etchant solution (i.e. primarily of water with $\varepsilon \sim 80$). Whenever some free volume in the polymer, may it be intrinsic or formed by etching, is filled by the etchant, then the capacity slightly increases during the incubation time which indicates the transport of etchant into the polymer's interior. This is clearly seen in the measurements of Figs. 2(a) and (b) for times till ~3.5 min, and of Fig. 3 for times till ~2.5 min, and also in Fig. 4 for times till ~8 ... 9 min, respectively.

The sudden appearance of the parallel resistor (R_T) in the equivalent circuit (Fig. 1) discharges the capacities. Suppose at the moment of breakthrough the sum of the values of the paralleled bulk capacity (C_B) and the track capacity (C_T) is C_0, then the charge Q is written by:

$$\frac{Q}{C_0} = -R_T \frac{dQ}{dt}.$$

At the moment of breakthrough, $Q = UC_0$, then the discharge (Q_1) is given by $Q_1 = UC_0 \exp(-\frac{t}{R_T C_0})$, where t is time. From this moment the charge of the etching circuit is given by:

$$Q = UC_0 - UC_0 \exp\left(-\frac{t}{R_T C_0}\right).$$

As the applied circuit voltage is constant, the capacity of etching vessel becomes

$$C = C_0 - C_0 \exp\left(-\frac{t}{R_T C_0}\right).$$

It is recognized from the above expression that the capacity of etching vessel will decrease after the moment of breakthrough. Though the capacitometer does not record the true capacity because of the additionally emerging parallel resistor (R_T), nevertheless it indicates the very moment of breakthrough precisely. The exact knowledge of that moment is very important in track etching technology.

Figs. 2(a) and (b) show the capacity and current measurement for irradiated and nonirradiated 8 μm thick

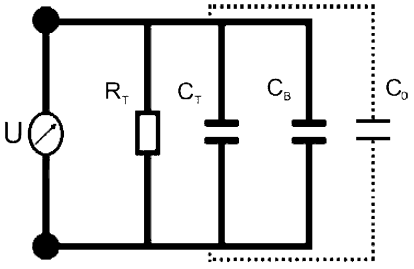

Fig. 1. The circuit of the etching vessel for the moment of breakthrough.