A RAPID METHOD FOR MAINTAINING THE MOLARITY OF NaOH SOLUTION DURING CONTINUOUS ETCHING OF PM-355 DETECTOR

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A series of experiments have been performed to investigate the physio-chemical change that occurred in the 6 M NaOH solution after etching PM-355 detector, each for 3, 6, 9, 12 and 15 hr. Various physical parameters like concentration, electrical conductivity (EC) and density of the solutions have been measured after each etching interval. Results have been discussed in the light of stoichiometrical equation for the interaction of PM-355 with NaOH. It is found that the concentration, EC and the density of the solution, in which PM-355 has been etched, increase linearly with respect to the etching time. The inter-relationship of EC-concentration of the etchant can be maintained at certain desired value by adjusting the corresponding value of EC or density, measured through non-destructive and quick methods.

1. INTRODUCTION

Nuclear track detectors are a technique widely used [1]. Tracks of ionizing particles in solid state nuclear tracks detectors (SSNTDs) can be revealed by a variety of techniques [2]. Chemical etching is the simplest and most widely used technique for revealing the latent damage trials of ionizing particles in solids. The sensitivity of a detector depends a lot on the etchants temperature [3], concentration [4, 5, 6], and even on the conditions at the time of the exposures [7]. It has been shown, for example, that the etch rate along the bulk of SSNTDs, increases with the increase in concentration of the etchant [8–12]. It is common particles in various laboratories, using SSNTDs technique, to etch detectors in the same etchant again and again. Consequently, the concentration of the etchant increases, even if the evaporation of water is prevented. This increase in concentration is generally due to the dissolution of detector material, which also changes the other physics-chemical properties like electrical conductivity, density, turbidity and the number total dissolved solids.

The presence of ions in etchant solution is responsible for its electrical conductivity. Becker et al. [13] have used electrical conductivity for the calculations of track dimensions of heavy ions in PM-355. The increase of the electric current after

the breakthrough of an etched track yielded in information on the transversal track etch rate in the core of the latent track of the heavy ions. Similarly, pore sizes were calculated through the conductivity measurements by Danziger [14].

In the present work, the influence of some of the physical parameters such as concentration, electrical conductivity and density of solution on etching rate of PM-355 is presented. The inter-relationships of the EC-conductivity and density-concentration are further exploited to suggest a non-destructive and quick method for the determination of concentration at any time during the etching process.

2. EXPERIMENTAL

Ten pieces of PM-355 detectors, each having 2 cm × 2 cm size, were used in the present investigations. All the samples were etched in 200 ml of 6 M NaOH solution at (70 ± 1) for 3, 6, 9, 12 and 15 hr. After each etching time interval, in detectors the following physical quantities: concentration, density and electrical conductivity of the etchant were modified and in this paper the rates of modification are determined. The remaining 100 ml of 6 M NaOH solution, without containing any of the detectors (blank solution), was also placed in the same etching bath and the values of its concentration, electrical conductivity and density were also measured after each etching time interval.

Table 1

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Etching time(hr)</th>
<th>NaOH with PM-355 (solution 1)</th>
<th>NaOH blank (solution 2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration (mol/l)</td>
<td>0</td>
<td>6.02</td>
<td>6.02</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>6.22</td>
<td>6.11</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>6.42</td>
<td>6.21</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>6.65</td>
<td>6.32</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>6.87</td>
<td>6.46</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>7.10</td>
<td>6.57</td>
</tr>
<tr>
<td>Electrical conductivity (us/cm)</td>
<td>0</td>
<td>307</td>
<td>307</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>379</td>
<td>330</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>446</td>
<td>348</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>526</td>
<td>363</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>598</td>
<td>388</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>679</td>
<td>412</td>
</tr>
<tr>
<td>Density (g/ml)</td>
<td>0</td>
<td>1.21</td>
<td>1.21</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1.38</td>
<td>1.22</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>1.63</td>
<td>1.23</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>1.81</td>
<td>1.24</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>2.10</td>
<td>1.25</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>2.24</td>
<td>1.26</td>
</tr>
</tbody>
</table>
The concentration was measured by using Multi 350i/set (made in Germany) with an accuracy of ± 0.01 mol/l. Electrical conductivity was measured with the help of a conductivity meter (Multi 350i/set; made in Germany) having an accuracy of ± 0.01 µS/cm. Density was determined using density bottle, by measuring the mass and volume of the solution after every etching. Maximum error in determining the mass of the etchant was less than or equal to ± 2 mg. The net experimental errors in the measurement of concentration, electrical conductivity and density were therefore not more than 2%.

3. RESULTS AND DISCUSSION

Table 1 shows the values of concentration, electrical conductivity and density of NaOH solution after etching PM-355 samples in it for different time intervals. The deviation from the value of each physical quantity determined for solutions in which PM-355 have been etched (solution 1), was not more than 1% in any of the measurements. The values of the same parameters for NaOH solution without containing any of the SSNTDs (solution 2) are also shown in Table 1. In the following, we discuss the changes that occurred in the values of concentration, electrical conductivity and density at successive etching intervals. The chemical reaction of PM-355 with NaOH solution is [15]

\[ \text{C}_{12}\text{H}_{18}\text{O}_7 + 4\text{NaOH} \rightarrow 2\text{Na}_2\text{CO}_3\cdot\text{H}_2\text{O} + 2\text{C}_3\text{H}_6\text{O} + \text{C}_4\text{H}_{10}\text{O}_3 \]  

3.1. CONCENTRATION

Fig. 1 shows the values of the concentration (C) of the etchant for the solutions in which PM-355 (solution 1) have been etched and of the un-used NaOH solution (solution 2) kept under the same conditions as solution 1.

![Fig. 1. Change of concentration (C) of NaOH solution with respect to the etching time. Error bars include statistical as well as measurements uncertainties.](image-url)
It has been observed that concentration increases linearly with respect to the etching. The concentration of solution 1, after 15 hr of etching is 8% higher than solution 2, where the change in the concentration is only due to water evaporation. This increase in C values in solution 1 is therefore due to etched products in the solutions as given by Eq. (1).

3.2. ELECTRICAL CONDUCTIVITY

The main source of electrical conductivity ($\sigma$) in the polar solutions is the presence of ions. Electrical conductivity in the case of NaOH solution in which plastic detectors have been immersed, however, depends on the degradation of plastic detectors via cleavage of ester linkages, into different inorganic and organic polar compound.

Fig. 2 represents the values of $\sigma$ in the case of solution 1 and solution 2, which show a linear increase with etching time. The value of electrical conductivity of solution 1 is increased by $\sim 65\%$ over the value of solution 2 after 15 hr of etching. High $\sigma$ values for the etchant in which PM-355 has been etched (solution 1) is due to the presence of higher number of carbonate ions, with respect to the un-used etchant (solution 2) as indicated by Eq. (1).

![Graph showing electrical conductivity vs. etching time](image)

Fig. 2. Change of electrical conductivity ($\sigma$) of NaOH etchant with respect to the etching time. "$\sigma$" in the case of fresh solution also increase with time. Error bars include statistical as well as measurements uncertainties.

3.3. DENSITY

Dissolution of detector in the etchant increases the weight of the solution per unit volume, resulting in the increase in density ($\rho$). Densities of solution 1 and solution 2 have been plotted in Fig. 3. The value of density of solution 1 is increased by $\sim 78\%$ over the value of solution 2 after 15 hr of etching. Like C and $\sigma$, density of solution 1 is a linear function of etching time. The density of blank (solution 2), however, is independent of the etching time.
3.4. INTER-RELATIONSHIP OF CONCENTRATION, ELECTRICAL CONDUCTIVITY AND DENSITY

The values of electrical conductivity (σ) for any solution can be measured directly, on-line and more accurately as compared to the values of concentration and density. We have therefore plotted the relationship of “σ” with respect to the concentration for solution 1 in Fig. 4. The experimental points have been fitted (confidence level 99.99%) with a straight line having a slope of 337.75 (µS/mol cm) and a constant of -1719.53 (µS/cm). Rearrangement of the values allows us to use the measured value of σ (µS/cm) directly in the following expression which yield the value of C (mol/l):

\[
C = 0.002969\sigma + 5.09
\]  

(2)
This indirect procedure of determining concentration is easier, quicker and more accurate. As the electrodes of the conductivity meters are generally made of glass with plastic cover, it is not advisable to use them to measure the \( \sigma \) of the etchant having the molarity and temperature in the region of the present studies. Hence meters having electrodes made of Teflon or quartz or some other suitable material may be used for the purpose. Alternately, density, the parameter which is not measured as accurately as \( \sigma \), but can be measured during etching without destroying any of the material, may be correlated with concentration as given in Fig. 5. The experimental points here too have been fitted (confidence level 99.64\%) with a straight line having a slope = 0.95 (Kg/mol) and a constant = -4.67 (Kg/l). The value of \( C \), therefore, can be obtained in mol/l, by direct substitution of the measured of \( \rho \) (Kg/l) in the following expression:

\[
C = 1.026\rho + 4.77 \tag{3}
\]

![Fig. 5. Plot of density versus concentration of the used NaOH solution at 70°C. Error bars include statistical as well as measurements uncertainties.](image)

### 4. CONCLUSION

During the etching of PM-355, the concentration, electrical conductivity and density of the etchant change linearly with respect to the etching time. The increase in the etch product of PM-355 in NaOH solution increases its concentration by 8\% after 15 hr of etching. After the same etching intervals, the EC and density, however, increase by 65\% and 78\%, respectively. The linear relationship of EC-concentration and density-concentration has been suggested to be used as the standard calibration for maintaining the concentration of the solution during the continuous prolonged etching process by measuring and adjusting the EC or density of the etchant on-line.
REFERENCES