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BULK ETCHING CHARACTERISTICS OF CR-39 TRACK DETECTORS IN HYDROXIDE SOLUTIONS†

by

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Abstract

A systematic study of the bulk etch rate of CR-39 track detectors in KOH and NaOH aqueous solutions is presented. A number of unirradiated and non-thermally treated CR-39 samples were chemically attacked in KOH and NaOH solutions of concentration and temperature in the range 2-10 N and 50-90°C, respectively. From measurements of the thickness of layers removed as a function of the etching time, the bulk etch rate v_B and the induction time t_o for surface removal were obtained for each etching condition. For both NaOH and KOH solutions the activation energy of the process was derived as $E = 0.76 \pm 0.05$ eV. It was observed that the induction time decreases both with increasing normality and temperature of the solution.

Key-words: Bulk etch rate; CR-39 track detectors; Hydroxide solutions; Induction time; Surface removal; Activation energy.

1. Introduction

In the simplest picture of etched nuclear-track formation, the track geometry and track evolution have been described usually by a mechanism of competition: the action of the chemical attack along the particle track at a rate v_T and the general chemical dissolution on the detector surface and the interior particle-track surface at a rate v_B [1]. Although the assumption is made that v_B at the undamaged detector surface and the interior particle-track surface are about equal for short etching times in a number of nuclear-track detectors, this assumption seems to be invalid in the case of CR-39 track-detectors*, particularly for incident heavily ionizing charged particles such as low-energy alpha particles and fission fragments.

The present experiment has been carried out to determine values of v_B on the undamaged CR-39 track-detector surface from measurements of the thickness of layers removed as a function of the etching time. Results have indicated values for the induction time t_o for surface removal (t_o is defined as the time between the immersion of the detector into the solution and the start of the chemical attack process) as large as 1-20 h, which strongly depends upon the etching conditions. In the present work we describe and discuss such results.

2. Experimental

A number ($\sim$ 70) of unirradiated and non-thermally treated CR-39 samples (15 × 10 × 1mm^3) obtained from American

*A polymer produced from the liquid monomer allyl diglycol carbonate [2,3].

Acrylics & Plastics Inc., were immersed in KOH and NaOH aqueous solutions of varied concentrations (2-10N) and temperatures (50-90^{o}C). For each chemical attack condition, samples of CR-39 track detectors were attacked without stirring during different time intervals. The etching equipment consisted of a 40-ℓ pyrex vessel filled with comestible oil into which the bottles containing the solutions (usually $\sim$ 400 mℓ) and the samples were dipped. Temperature gradients were avoided by constant stirring of the oil bath, and the temperature was kept constant ($\pm$ 0.3^{o}C of maximum uncertainty) by an automatic thermal regulator. Such a device enabled us to prolong etching times up to $\sim$ 150 h under controlled etching bath temperatures. The thickness of layers removed from one surface, $\Delta\ell = (\ell_o - \ell)/2$, was obtained by taking half of the difference between the original thickness ℓ_o and the thickness ℓ at an etching time t. Both ℓ_o and ℓ were measured by using a precise micrometer. Having determined $\Delta\ell$ at various etching times, values of v_B and t_o were derived from the slope and the intercept, respectively, of a plot of $\Delta\ell$ *versus* t.

3. Results and Discussion

The data for NaOH aqueous solutions are shown in Figs. 1-2. Good fits to a straight line with non-zero intercept have been obtained, thus indicating a constancy of v_B with depth after some induction time t_o to allow for surface removal. Similar results have been obtained also for KOH aqueous solutions. Least-squares analysis has been used from these plots in deducing the values of v_B and t_o for each attacking

condition.

The variation of v_B with normality of NaOH solutions at 70°C is shown in Fig. 3. For normalities greater than about 3 somewhat higher v_B-values have been obtained in the present work (1.5 times on the average) as compared with those of other authors. This may be due to either differences in the method used in obtaining v_B [4,5] or differences in manufacturing of CR-39 samples [6]. When the method of fission-fragment diameters measurement is chosen instead of the thickness change method, the quantity which is actually determined is the general chemical attack rate on the interior particle-track surface.

The induction time for surface removal has been shown to decrease both with increasing concentration and temperature of the attacking solutions. Some results are presented in Fig. 4. Such relatively high values for t_o (1-20 h in the concentration- and temperature-range studied here) may result from the highly cross-linked-like polymer structure which makes CR-39 track--detectors a much "harder" material than other commonly used plastic track-detectors. Thus the process of degradation of the detector surface is retarded during the early stages of chemical attack. On the contrary, as highly ionizing particles hit the detector surface a damage trail is formed making the interior particle-track surface along the particle trajectory more reactive than the detector surface. Therefore, low-energy alpha-particle and fission-fragment tracks, for instance, can be revealed within the induction time for surface removal [7].

Finally, the temperature-dependence of the bulk etch rate for 6.25 N KOH and NaOH solutions is depicted in Fig. 5. The data have been fitted to an equation of the type

$v_B = A \exp(-E/kT)$, where A is a constant (it includes the dependence of v_B on etchant concentration), k is Boltzmann's constant, T is the absolute temperature and E is the activation energy of the process. For both NaOH and KOH solutions the activation energy was found to be $E = 0.76 \pm 0.05$ eV, which compares well with the E-values of Green and co-workers [5] and Cartwright and co-workers [2]. Some disagreement is noted with the work of Khan and Khan [6]. For the case of NaOH solutions, the constant A could be expressed as $A = CN^{\alpha}$, where $C = (7.75 \pm 0.86) \times 10^9$ μm/h and $\alpha = 1.89 \pm 0.09$.

4. Conclusion

In conclusion, we point out the observation of large induction times for surface removal of CR-39 track detectors as compared to the time for etched-track formation by highly ionizing particles. We hope that this result may contribute to a better description of the etched particle-track geometry and the mechanism of track evolution for CR-39 nuclear-track detectors.

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References

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FIGURE CAPTIONS

FIG. 1. Bulk attack of CR-39 track detector by a 6.25 N NaOH etchant at different temperatures. The straight lines are least-squares fits to the data.

FIG. 2. Bulk attack of CR-39 detector by NaOH etchants of different concentrations at 70°C. The straight lines are least-squares fits to the data.

FIG. 3. Bulk etch rate (v_B) of CR-39 track detector as a function of normality (N) of NaOH etchants at 70°C. Least-squares treatment of our data gives $v_B = 0.064N^{1.89}$ (full line).

FIG. 4. Variation of the induction time for surface removal of CR-39 with etching conditions. The curves were drawn by eye to indicate the trends.

FIG. 5. Temperature dependence of bulk etch rate for 6.25 N KOH and NaOH etchants. The straight lines are least-squares fits to the data.

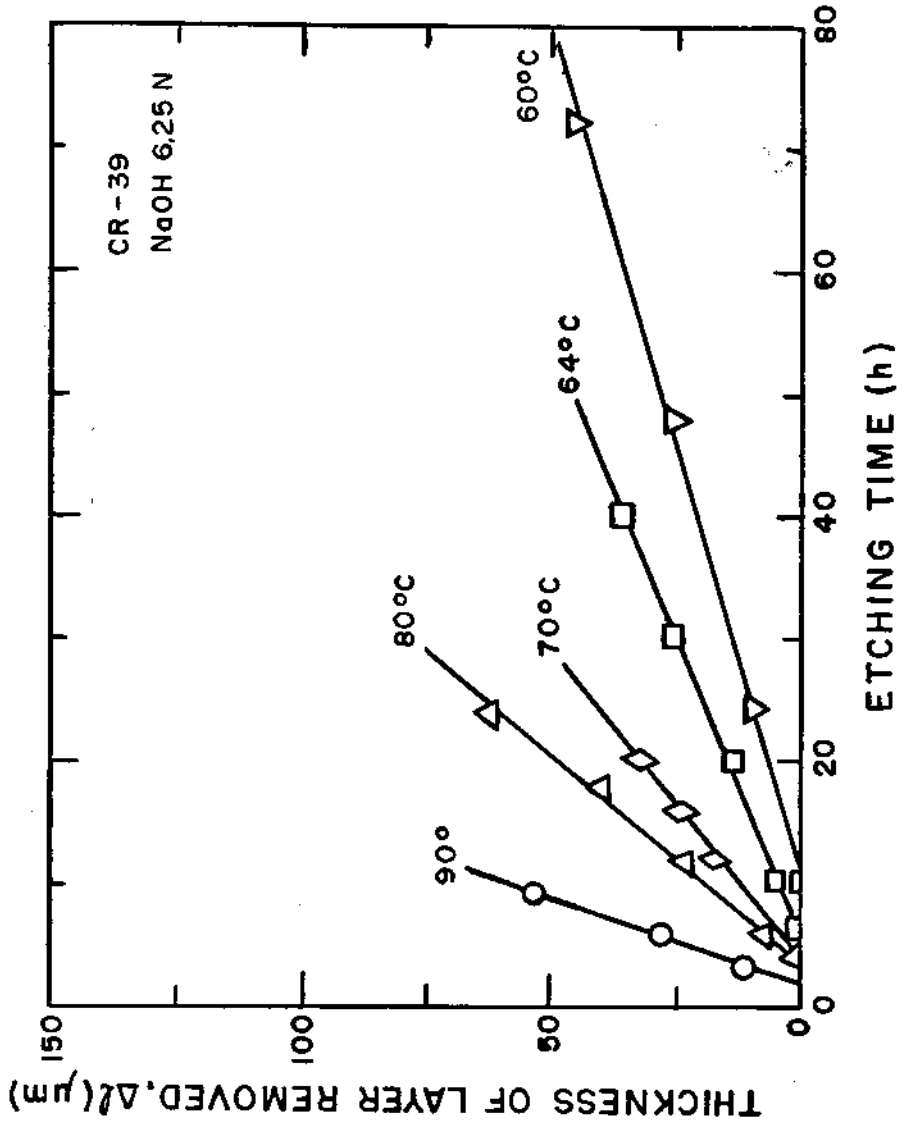
CR-39
NaOH 6,25 N
60°C
64°C
70°C
80°C
90°
ETCHING TIME (h)
THICKNESS OF LAYER REMOVED, Δl (μm)
0
20
40
60
80
50
100
150

FIG. 1

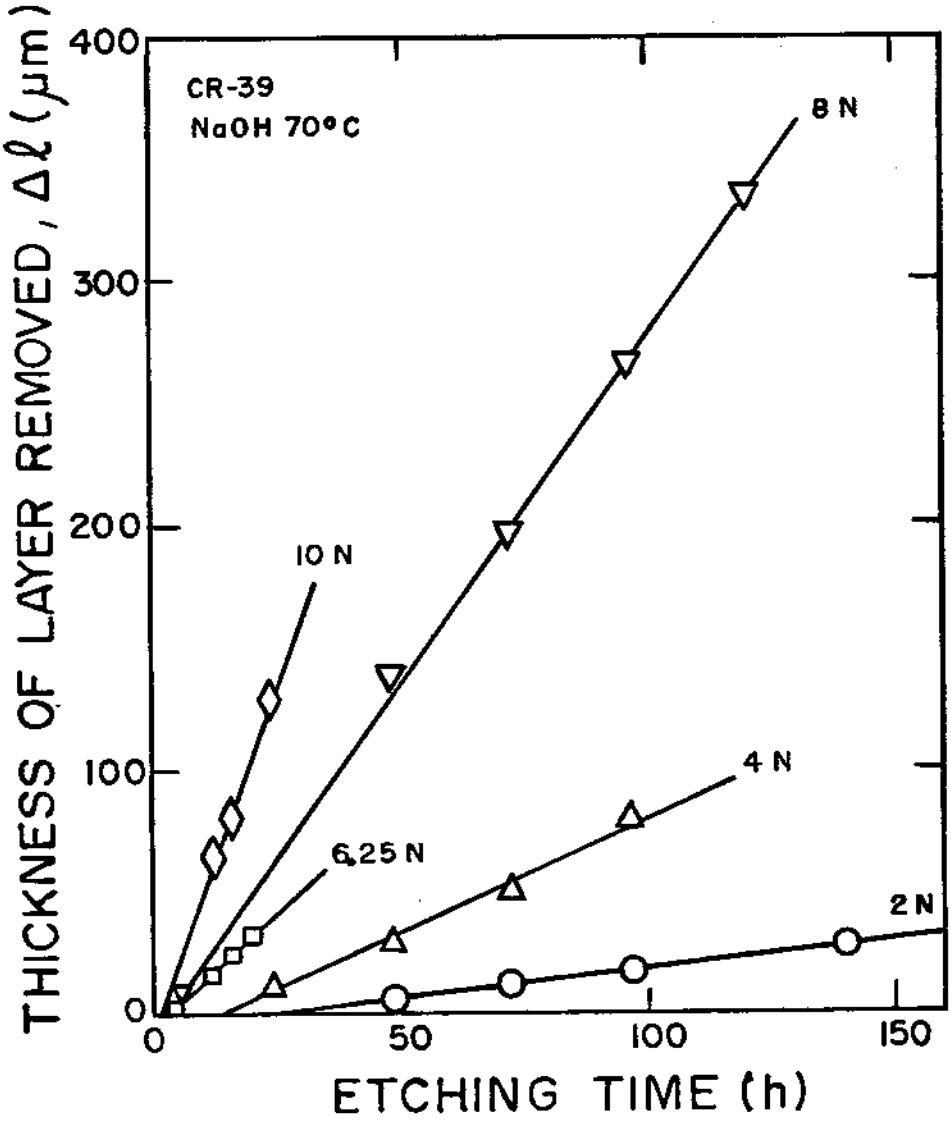
CR-39
NaOH 70°C
8 N
10 N
6.25 N
4 N
2 N
THICKNESS OF LAYER REMOVED, Δl (μm)
ETCHING TIME (h)
0
100
200
300
400
0
50
100
150

FIG. 2.

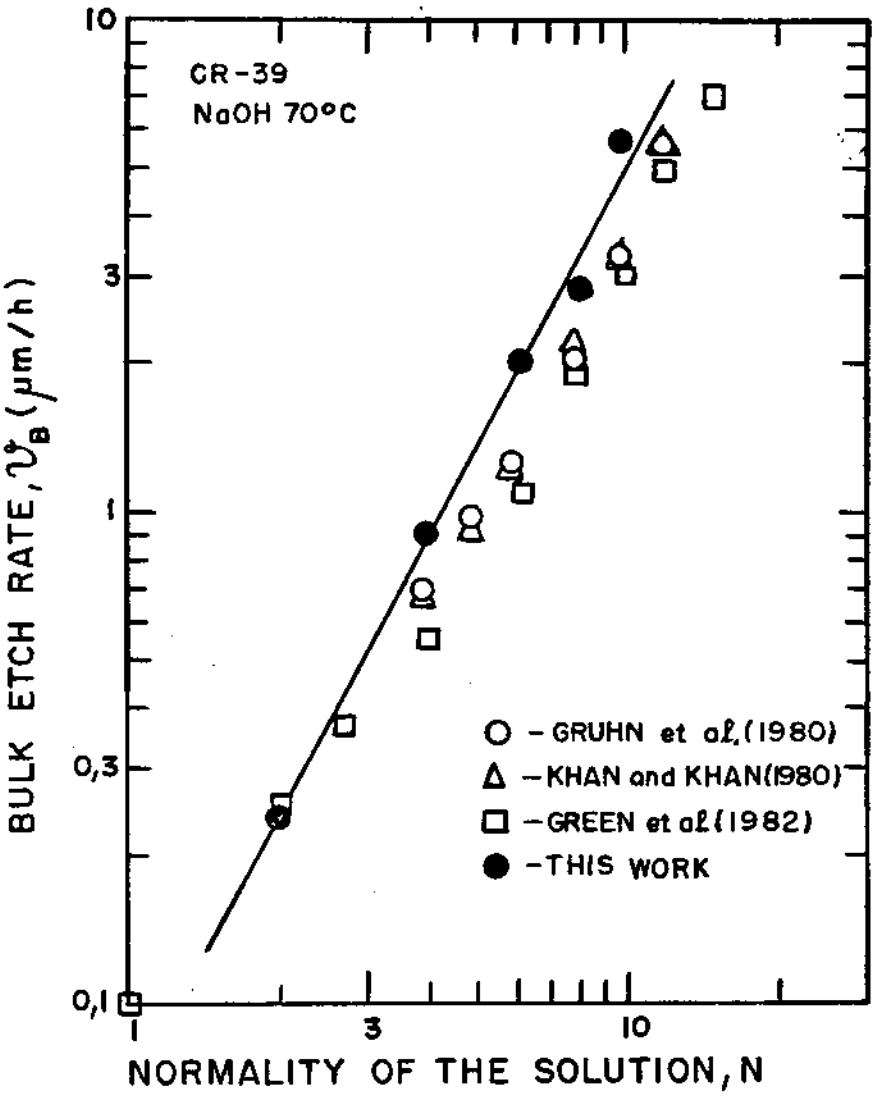
CR-39
NaOH 70°C
BULK ETCH RATE, v_B (μm/h)
NORMALITY OF THE SOLUTION, N
10
3
1
0,3
0,1
1
3
10
O - GRUHN et al.(1980)
Δ - KHAN and KHAN(1980)
□ - GREEN et al(1982)
● - THIS WORK

FIG. 3

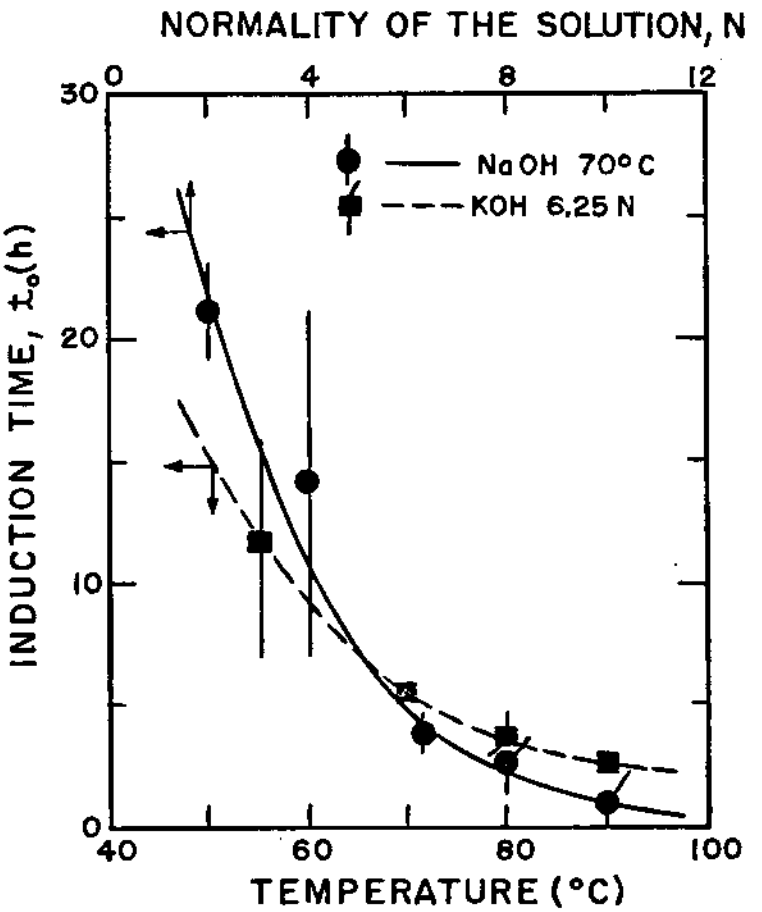
NORMALITY OF THE SOLUTION, N
0
4
8
12
30
20
10
0
NaOH 70°C
KOH 6,25 N
INDUCTION TIME, t_o(h)
40
60
80
100
TEMPERATURE (°C)

FIG. 4

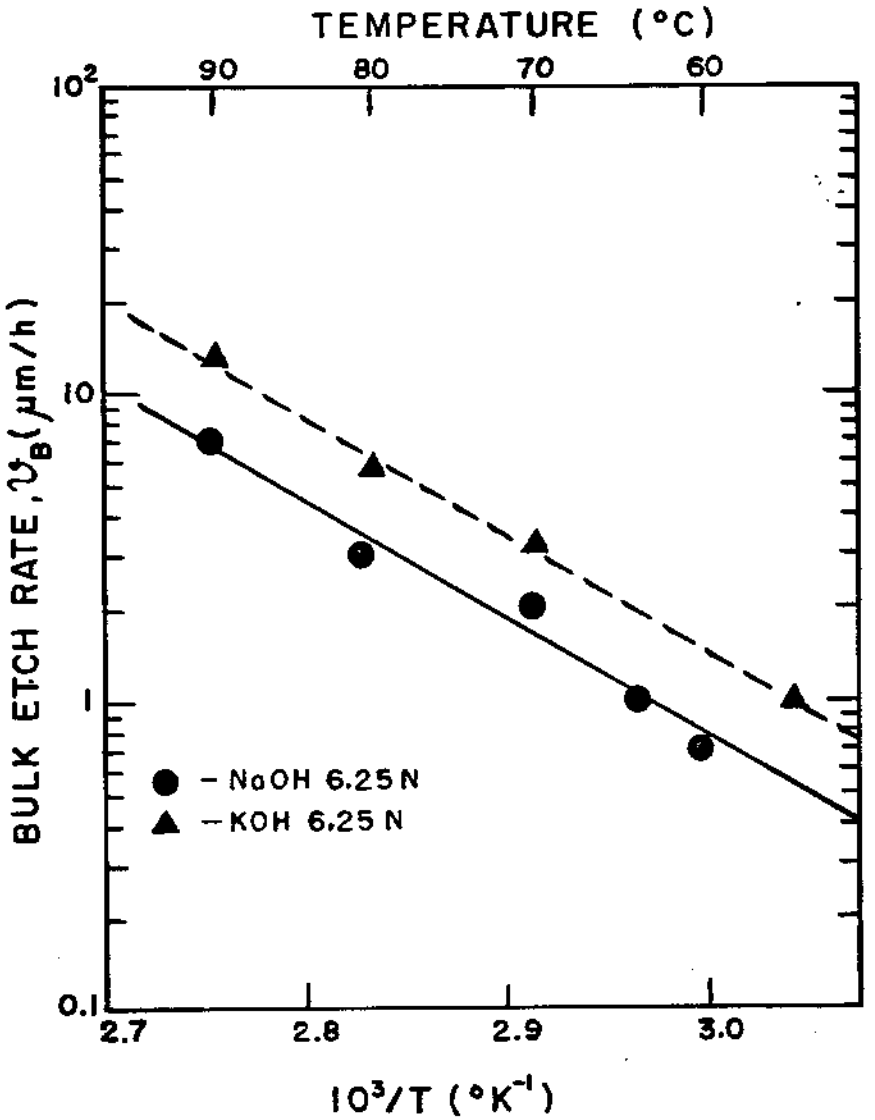
TEMPERATURE (°C)
90
80
70
60
10^2
10
1
0.1
BULK ETCH RATE, v_B (μm/h)
● – NaOH 6.25 N
▲ – KOH 6.25 N
2.7
2.8
2.9
3.0
$10^3/T$ (°K^{-1})

FIG. 5