Bulk Etch Rate Properties of NaOH/Ethanol as a CR-39 Nuclear Track Detector Etchant

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Abstract

The bulk etch rate properties of NaOH/ethanol as a CR-39 nuclear track detector etchant were investigated. It was discovered that NaOH/ethanol is an aggressive bulk etchant that has a bulk etch rate greater than the standard NaOH solution etchant. The bulk etch rate properties of NaOH/ethanol were tested by etching CR-39 (polyallyl diglycol carbonate) in NaOH/ethanol of various temperatures and molarities. It was found that as temperature increased the bulk etch rate increased. However, the molarity of the NaOH/ethanol solution did not have a significant effect on the bulk etch rate. 1.5M NaOH/ethanol at 60°C yielded the fastest bulk etch rate of 27.3 μm/hr. The results obtained in this experiment will support a new CR-39 processing method that will include a background noise subtraction technique intended to enhance the accuracy of CR-39 diagnostics.

Introduction

CR-39 nuclear track detectors are used at the Laboratory for Laser Energetics (LLE) to record the products of inertial confinement fusion reactions conducted on the OMEGA laser system[1]. As a part of the Magnetic Recoil Spectrometer,[2] CR-39 detectors are positioned around the OMEGA laser system fuel capsule. When the lasers are fired, intense heat and pressure implode the fuel capsule and charged particles are emitted. These charged particles, which include protons, alpha particles, and deuterium and tritium atoms, bombard and damage the CR-39 detectors. This results in latent tracks of damage typically ranging in diameter from 3 nm to 10 nm,[1] too small to be visible with an optical microscope.

The latent tracks are made visible by etching the CR-39 in a 6N NaOH at 80°C for 6 hours. During the etching, conical pits are created along the particle tracks because the damaged plastic along the track has a higher etch rate than the undamaged (bulk) plastic.[1] These pits range from 10 μm to 20 μm in diameter, large enough to be visible under an optical microscope. Etched CR-39 detectors are scanned under an optical microscope using the Charged Particle Spectroscopy (CPS) program developed by scientists at MIT. The CPS program uses the pit eccentricity, diameter and resulting contrast to calculate an energy spectrum of the particles ejected during the implosion. This information is then used to calculate the areal density[3] of the target and to quantify the success of the reaction.

Unfortunately, data collected from CR-39 detectors can be compromised by defects (background noise) found in the bulk of the plastic created during the CR-39 manufacturing process. During the etching process, these defects are revealed as pits with diameters comparable to those created along particle tracks (valid data pits) as shown in Figure 1. The CPS program cannot differentiate between defect pits and valid pits which can result in inaccurate data collection. A new etching process is being investigated at LLE and MIT that would enable scientists to identify background noise from data.

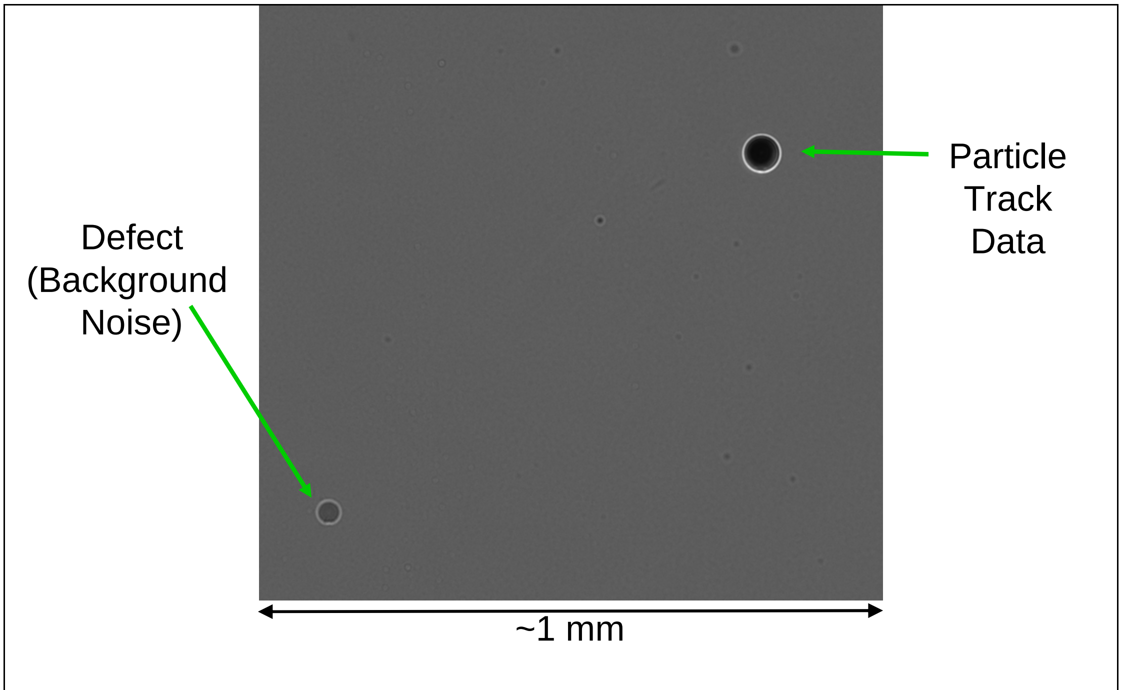

Figure 1: Image of CR-39 under an optical microscope after standard etching. The defect pit appears similar to the valid pit and would be identified as a valid pit by the CPS program.

Background Noise Reduction Technique

This process uses an accelerated, more aggressive etchant to remove some of the bulk material, and noise within it, while preserving the data tracks. This process begins with a standard etch of CR-39 in 6N NaOH at 80°C for 6 hours. The CR-39 detector is then scanned using the CPS program and an image of the CR-39 surface is recorded.

Next, the CR-39 is etched in NaOH/ethanol. NaOH/ethanol removes a large amount of bulk and a relatively small amount of particle track.[4] Defects created during the manufacturing process are significantly shallower than particle tracks[5] and are located intermittently throughout the CR-39 bulk. Therefore, the defect pits established after the initial standard etch are removed during the NaOH/ethanol etch, but the valid data track remains intact.

The CR-39 detector is etched again in 6N NaOH at 80°C for 6 hours to reestablish valid pit shape, and scanned by the CPS program. During this final NaOH etch, defect pits are also established, but can be differentiated from valid data because defect pits appear in different locations on the initial and final scan while valid pits appear in the same location.

For this new process to be viable, the bulk etch rate properties of NaOH/ethanol must be known. A substantial amount of bulk must be removed during the NaOH/ethanol etch to eliminate defects seen in the first scan. However, if too much CR-39 is removed, particle tracks are eliminated completely and the valid data are lost. In this experiment, the bulk etch rate of various NaOH/ethanol solutions were studied in an attempt to find a NaOH/ethanol solution with an efficient and reliable bulk etch rate.

Methods and Materials

In the present experiment, the bulk etch rate of CR-39 was tested in NaOH/ethanol solutions ranging from 1M to 4M at temperatures of 50°C, 55°C, and 60°C. 31 CR-39 detectors were tested, all of which had not been exposed to radioactive particles.

NaOH/ethanol solutions were prepared in a beaker using 10M NaOH and distilled water. Beakers were placed in a water bath to acquire and maintain the desired temperature, and covered to prevent evaporation.

Prior to etching, the thickness of each CR-39 detector was measured in five locations using a micrometer. CR-39 detectors were placed in NaOH/ethanol, and measured every 30 minutes for 3 hours. Prior to measurement, CR-39 detectors were rinsed with distilled water and dried. Before detectors were returned to the NaOH/ethanol, the solution was stirred.

The five measurements taken at each 30 minute interval were averaged to find the average thickness of the CR-39 detector at each interval. For each interval, the average thickness was subtracted from the initial average thickness to find the amount of CR-39 removed in millimeters. These values were plotted on a graph with x-values representing time in hours and y-values representing the amount of CR-39 removed in millimeters. A line of best fit was determined for the data (see Figure 2).

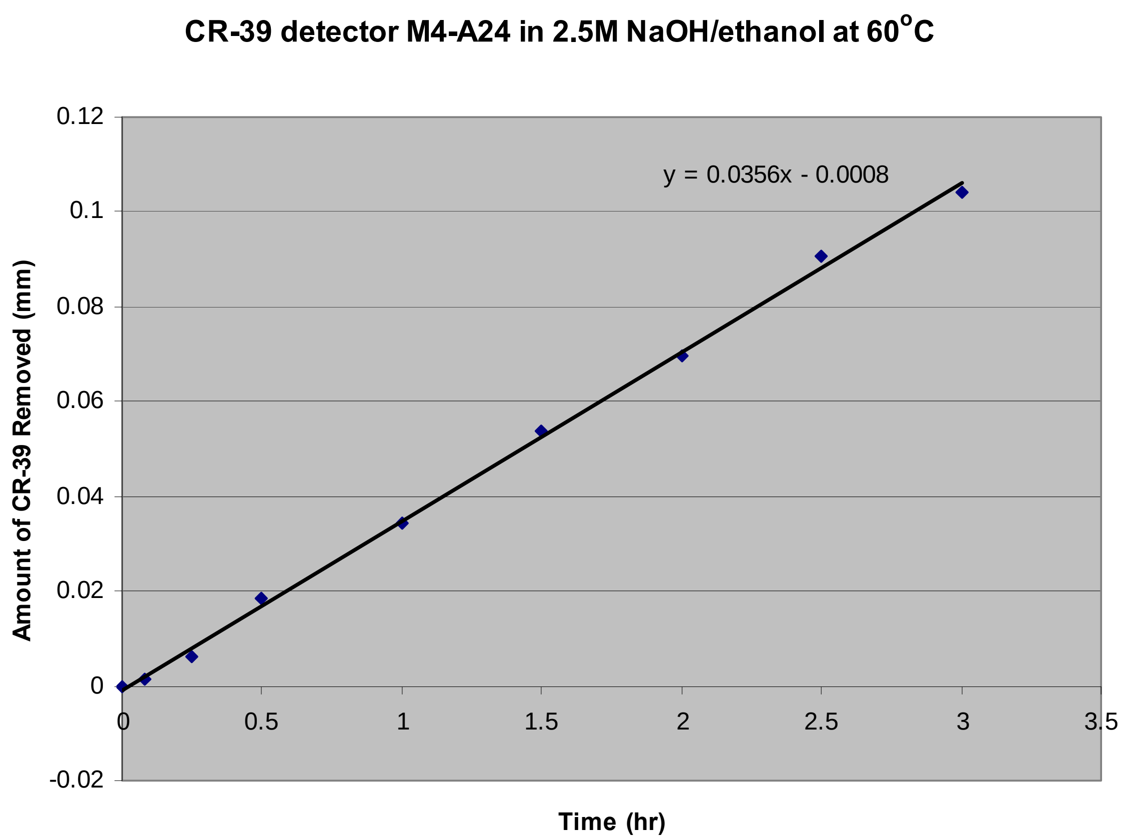

Figure 2: The amount of bulk removed from CR-39 detector M4-A24 in 2.5M NaOH/ethanol at 60°C over time. X-values represent time in hours. Y-values represent the amount of bulk removed in hours. A line of best fit was determined and its equation is shown.

The slope of the line of best fit was entered into the following equation

$$\frac{1000m}{2} = V_b$$

where m represents the slope of the line of best fit and V_b represents the bulk etch rate in μm/hr. The value m was multiplied by 1000 to convert from millimeters to μm and then divided by 2 to account for measuring both sides of the CR-39.

Results

1.5M NaOH/ethanol at 60°C yielded the fastest bulk etch rate of 27.3 µm/hr. As the temperature of NaOH/ethanol increased the bulk etch rate increased. Change in molarity had a greater effect on the bulk etch rate as temperature increased, but there is no apparent relationship between molarity and bulk etch rate for all temperatures. The bulk etch rate of NaOH/ethanol at 50°C was consistent despite changes in molarity. These results are summarized in Figure 3.

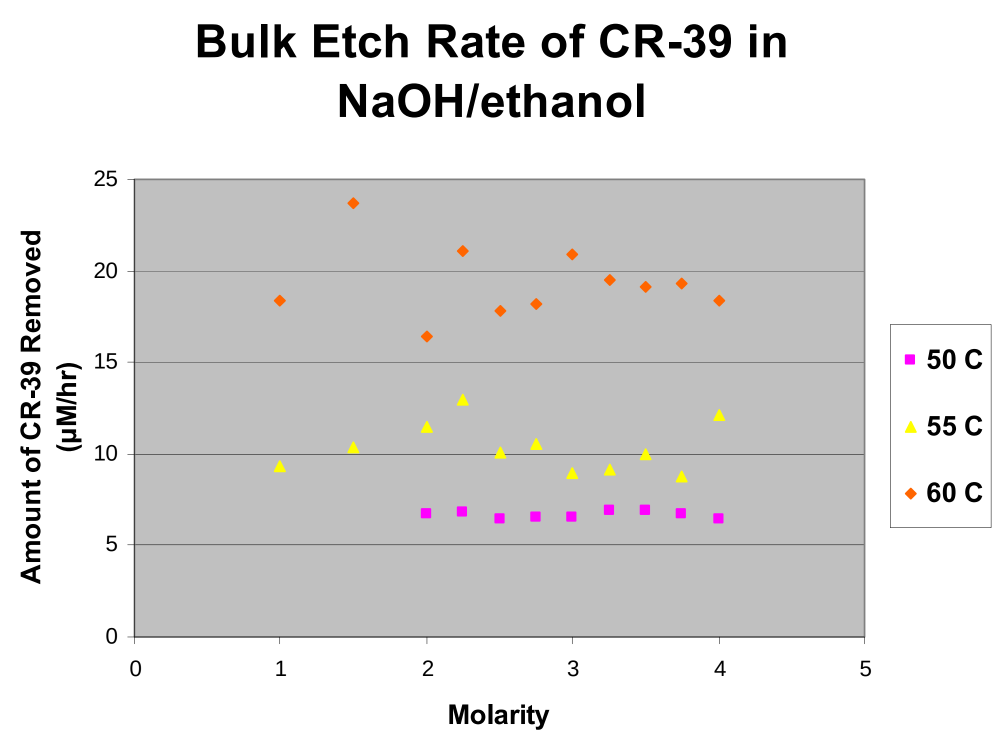

Figure 3: The bulk etch rate of CR-39 in NaOH/ethanol solutions. Values on the x-axis represent NaOH/ethanol solution molarity. Values on the y-axis represent the bulk etch rate in µm/hr. Each data point represents a CR-39 detector etched in NaOH/ethanol at the indicated temperature and molarity.

Discussion

The drastic variation in bulk etch rate at higher temperatures is suspected to be caused by sodium carbonate precipitate formed during etching.[6] As temperature increased more precipitate formed, which may have insulated the CR-39 surface and hindered etching.[4] At 55°C, the bulk etch rate increased with molarity from 1M to 2.25M then began to decrease. This finding correlates with data collected by K.F. Chan et al. who reported the bulk etch rate of CR-39 in 55°C NaOH/ethanol to increase with the molarity of NaOH/ethanol, reach a maximum at ~2.5N, and start to drop beyond 3N. [4]

The results obtained in this experiment demonstrate that the ideal NaOH/ethanol temperature for the proposed processing method is 50°C because it produced the most consistent bulk etch rates. This discovery of NaOH/ethanol at 50°C as a reliable and efficient bulk etchant supports the viability of the proposed background noise subtraction technique that will enhance the accuracy of CR-39 diagnostics on the OMEGA laser system.

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