The Use of Surface Grinding and Polishing to Remove Etch Induced Noise Pitting in CR-39 Samples

Sean Reid

Fairport High School

Fairport, NY 14450

Advisors: Michelle Burke, Robert Boni, Dr. Stephen Jacobs

Laboratory for Laser Energetics

University of Rochester

Rochester, NY

Summer 2011

Abstract:

A time-efficient surface grinding and polishing procedure was developed to remove noise pitting associated with the etching of CR-39. CR-39 is a solid-state nuclear track detector used to measure and analyze ions produced by fusion implosions on the OMEGA and OMEGA EP laser systems. Using CR-39 thickness measurements acquired during the grinding process, surface removal rates of 68 μm/min and 5 μm/min were determined for 15-μm and 9-μm-grade abrasive pads, respectively. Removing 3-4 μm of surface material eliminated noise pits, as well as shallow 1-MeV and 2-MeV proton-induced pits. Exposing a CR-39 sample to UV light before etching was investigated as a method to deepen shallow data pits. CR-39 exposure to UV light for 48 hours increased 1-MeV and 2-MeV proton pit depth. The combination of CR-39 UV exposure and surface grinding and polishing was shown to eliminate noise pits while preserving data pits.

Introduction:

CR-39 is a solid-state nuclear track detector used to measure ion yield from OMEGA and OMEGA EP inertial confinement fusion implosions. Charged particles emitted from laser fusion reactions are scattered radially from the target, uniformly striking the CR-39 detector as shown in Figure 1. Ions such as protons, deuterons, tritons, and alpha particles incident on the CR-39 disrupt the polymer's chemical bonds, scattering electrons and forming latent tracks in the material. These tracks are invisible to an optical microscope, and are typically 3 nm to 10 nm in diameter[1].

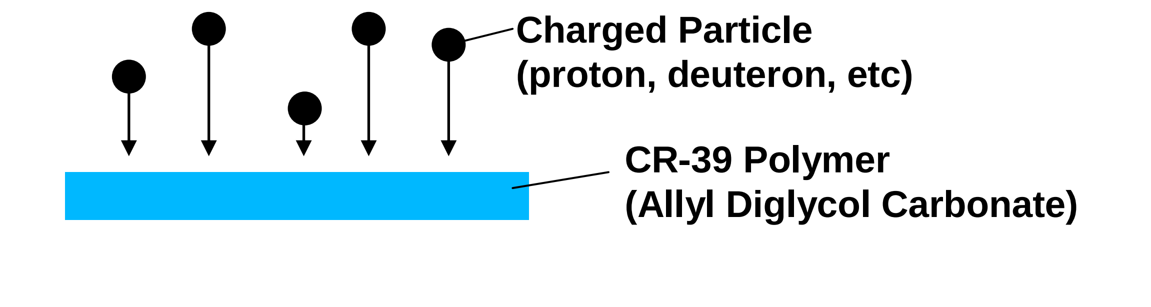

Figure 1:
CR-39 sample detecting ions emitted from inertial confinement fusion. The ions form latent tracks in the CR-39 polymer.

The irradiated CR-39 detector is exposed to a chemical etchant such as sodium hydroxide (NaOH) to establish latent track visibility. The etchant reacts with the polymer bonds of the detector, removing layers of material. The scattered bonds along the latent tracks of the CR-39 detector react faster than the bonds of the bulk material, resulting in conical-shaped pits as shown in Figure 2. These pits typically range from 5-25 μm in diameter, depending on particle type and etching conditions. A standard etch for the purposes of this experiment consists of a 6 hour exposure to 80°C, 6M NaOH.

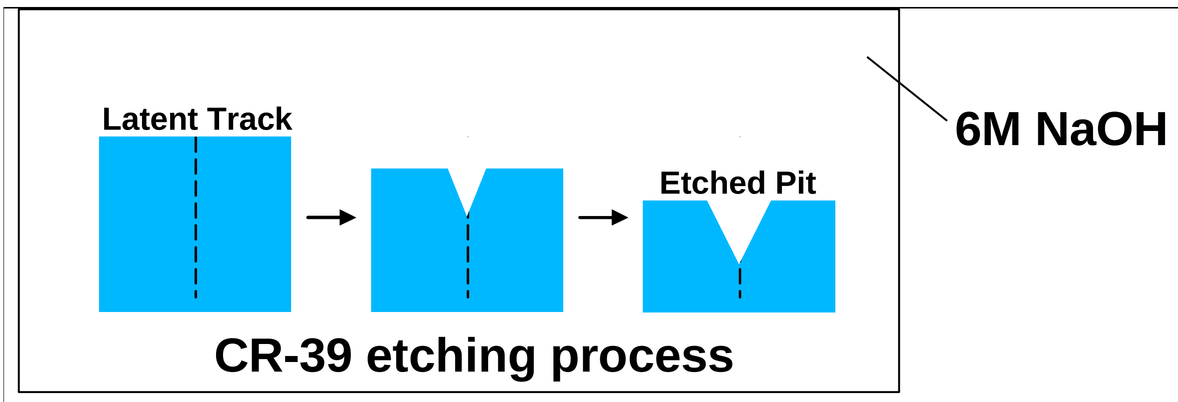

Figure 2:
The CR-39 etching process. NaOH etches along the latent tracks faster than the bulk material, forming visible conical pits.

The CR-39 is then scanned under an digital microscope to calculate ion yield from the etched tracks. A program developed by scientists at the Massachusetts Institute of Technology is used to facilitate this scanning process. The program analyzes images taken of the detector's surface to measure etched pit size and count, using contrast and eccentricity as parameters.

The Laboratory for Laser Energetics receives its shipments of CR-39 from Track Analysis Systems Ltd. Unfortunately, many of these shipments experience a noise pitting effect when exposed to NaOH, such that pitting appears on the CR-39 even in the absence of particle exposure. Other vendors such as Edmund Optics produce CR-39 samples which yield similar noise when etched, supporting the conclusion that noise pitting is not specific to Track Analysis Systems Ltd. Noise pitting differs from particle-induced pitting in formation and structure. It is not a result of particle irradiation, and therefore is not caused by latent particle tracks. Although MIT's scanning program is usually able to differentiate between noise and data, some noise may exhibit a similar contrast and/or eccentricity as charged particle-induced pits when analyzed, as seen in Figure 3.

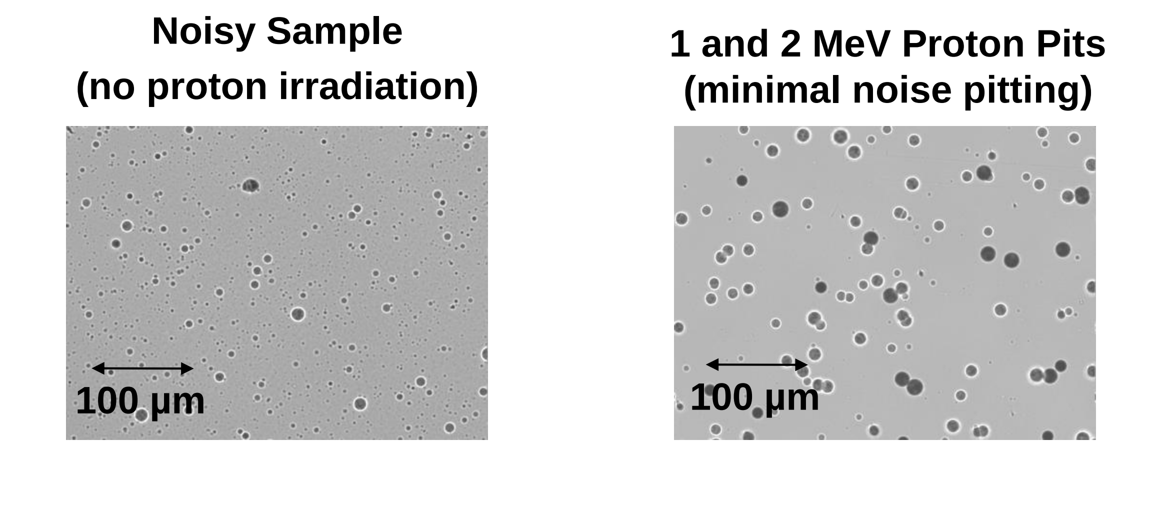

Figure 3:
Images of CR-39 samples at 40x magnification. CR-39 shows noise pitting, on the left, visually similar to data pits, on the right.

CR-39 Grinding and Polishing Procedures:

The majority of noise pitting is characterized by high eccentricity and low contrast, which prompted the CR-39 noise pitting experimental hypothesis that shallow noise pits could be easily ground and polished away while leaving the deeper charged particle induced pits remaining, as graphically represented in Figure 4.

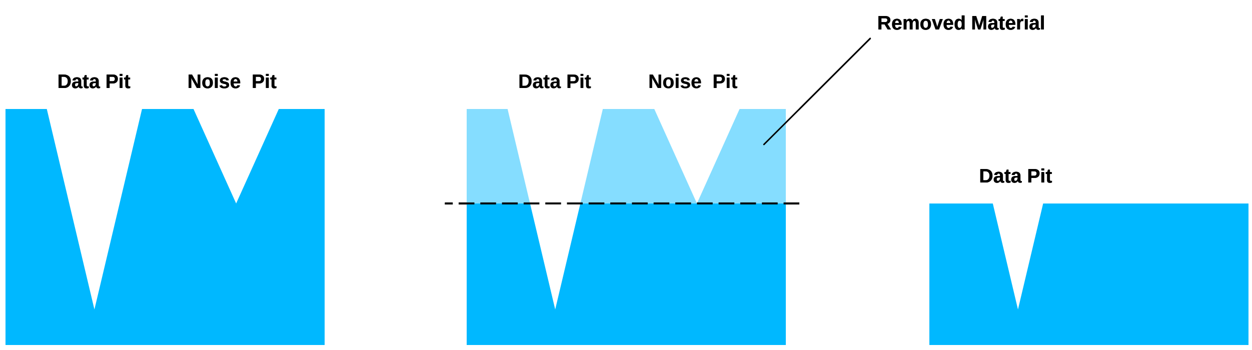

Figure 4:
The grinding and polishing method of noise pit reduction in CR-39. Surface material is removed, eliminating shallow noise pits and leaving deeper data pits.

Three types of abrasive pads were purchased from Universal Photonics Incorporated, for the purposes of grinding and polishing CR-39: a 15-μm rough-grade fixed abrasive pad for bulk removal, a 9-μm fine-grade fixed abrasive pad for surface smoothing, and a velveteen polishing pad to be used with an aluminum oxide solution for further surface smoothing.

A polishing rotating spindle was used to facilitate the grinding and polishing process. The CR-39 is mounted in a custom fabricated aluminum holder, with protective masking tape applied to the mounted side. An abrasive pad is then applied to the polishing spindle using the pad's adhesive backing. The polishing spindle and pad are moistened with water to flush residue during the grinding process; water must be continuously applied during grinding. To grind CR-39 on a moving spindle, the mounted sample is moved circularly on the surface of the abrasive pad, in the opposite direction of rotation of the spindle. The mounted sample is rotated often to create even abrasion.

To develop a time-efficient procedure for the grinding and polishing of CR-39, the grinding and polishing pads were tested for their surface removal rates. Each CR-39 sample used in the surface removal rate experiment was measured in ten invariant locations by a digital micrometer every minute during the grinding and polishing process. The average surface thickness was plotted against time to determine the rate of bulk material removal, as illustrated in Figure 5.

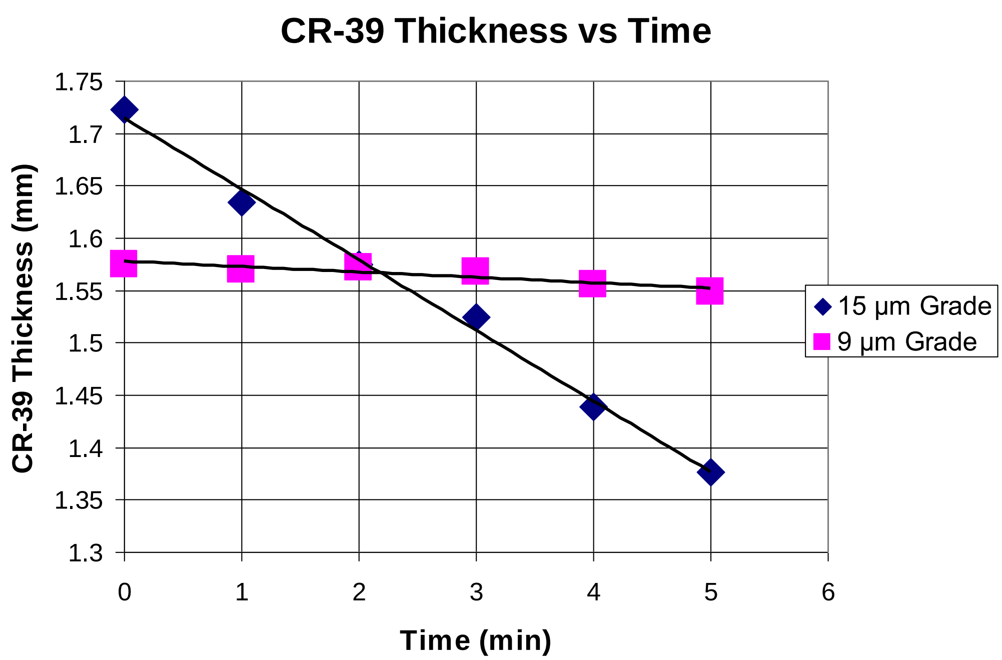

Figure 5:
The thickness of CR-39 samples plotted against time for two different fixed-abrasive pads. The slopes of the graphs represent the rate of surface material removal for the two pads: 5 µm/min for the 9 µm grade pad, 68 µm/min for the 15µm grade pad.

Rates of 68 µm/min and 5 µm/min were determined for the respective 15-µm-grade and 9-µm-grade pads. Due to its high rate of material removal, the 15-µm grade pad was removed entirely from the grinding process. The polishing velveteen with its aluminum oxide solution did not show any measurable rate of material removal, and was therefore not used for bulk removal estimates. Figure 6 shows this graphically.

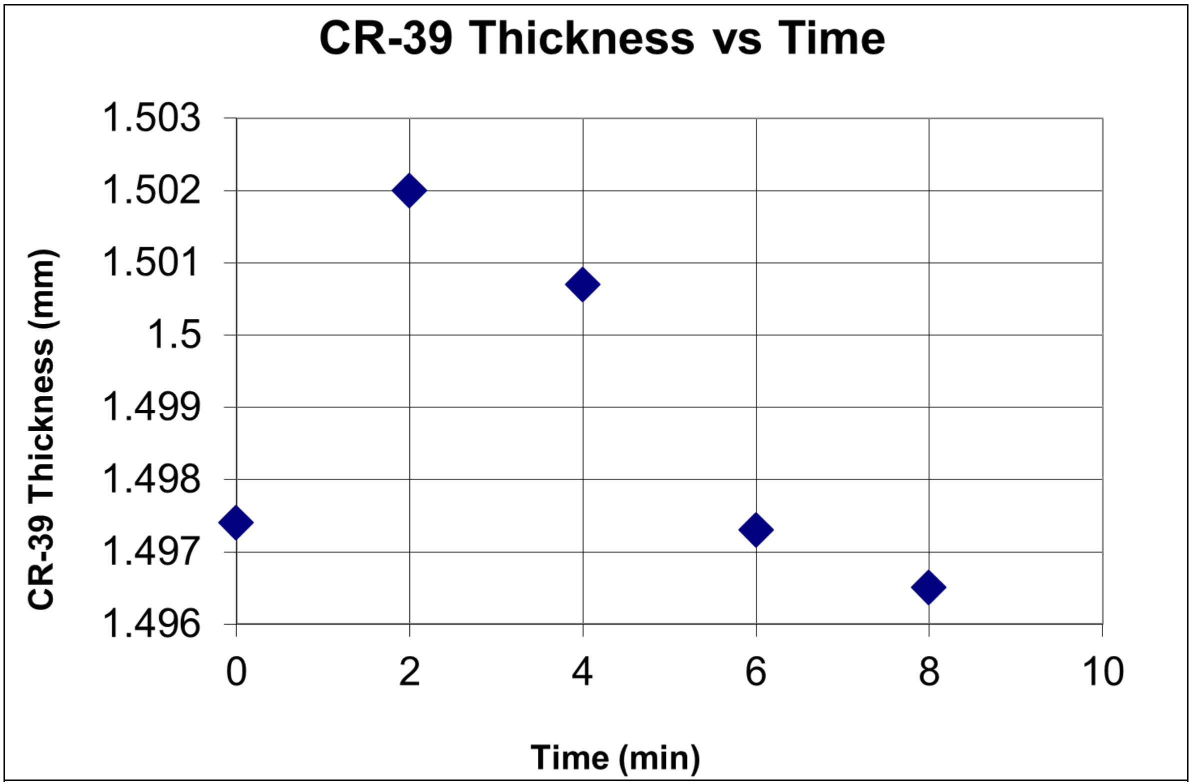

Figure 6:
CR-39 removal using a velveteen polishing pad and aluminum oxide polishing solution. The surface thickness could not be measured with sufficient accuracy to determine the material removal rate.

The order in which CR-39 etching occurs in the grinding and polishing process was also tested. A CR-39 sample was ground, polished, and then etched to determine the effects of CR-39 surface alterations on the etching process. Despite attempts to smooth the surface of the polymer with the polishing pad and solution, small scratches in the CR-39 surface were deepened during the etching process, becoming indistinguishable from noise as seen in Figure 7.

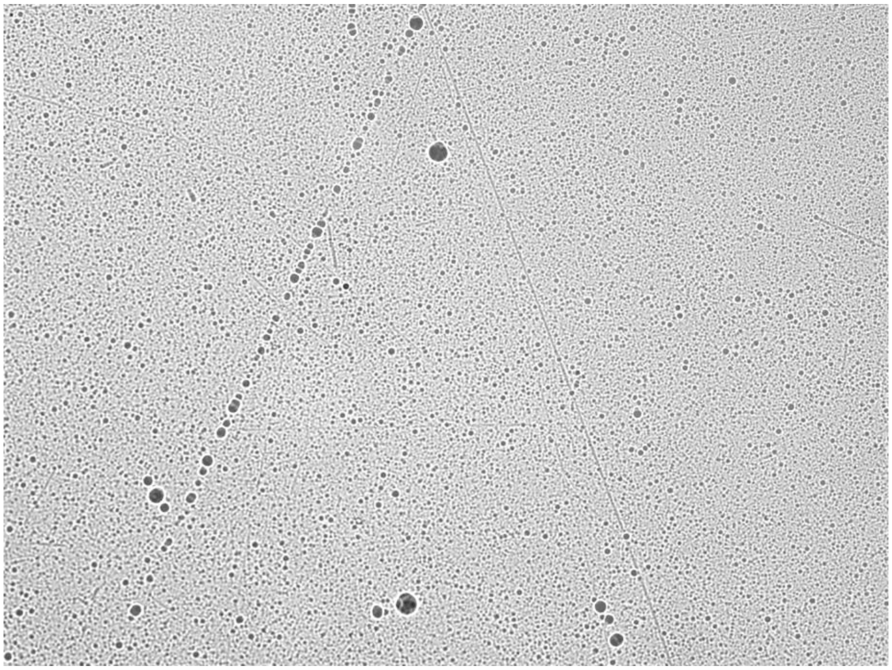

Figure 7:
CR-39 sample etched after grinding and polishing. Scratches and pits left by surface grinding are deepened during the etching process, creating etched noise.

Using the measured removal rates, a time-efficient procedure was developed to grind and polish noisy CR-39 samples. First, the etched sample is mounted in the aluminum holder with protective masking tape. It is ground for 30 seconds on the 9-μm-grade pad and spindle, removing approximately 3-4 μm of bulk material. Then it is moved to the polishing pad, where the surface is smoothed for 6 minutes.

After grinding, the surface of the CR-39 sample is scratched and unable to be scanned. The polishing pad and solution eliminate those scratches. The root-mean-square (RMS) surface roughness was measured every 2 minutes during the polishing process using a NewView 100 white-light interferometer to determine the most efficient polishing time. The results of this experiment are shown in Figure 8; after 6 minutes, the surface was smooth enough to be scanned.

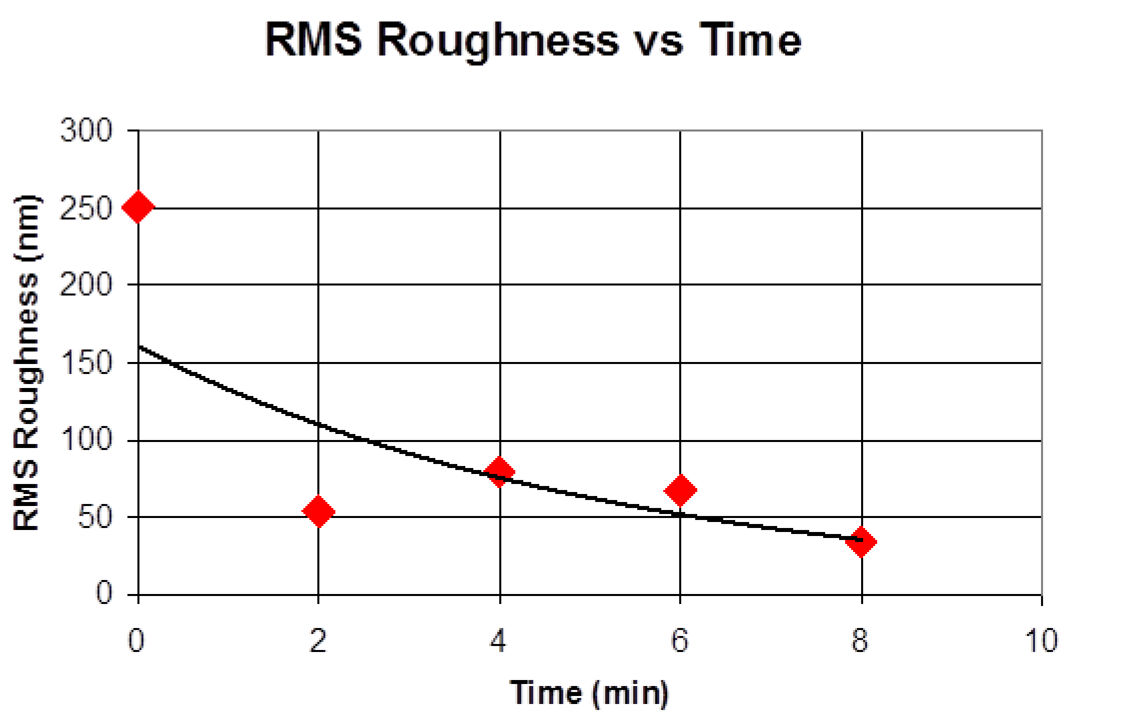

Figure 8:
RMS roughness deviation of a CR-39 sample plotted against polishing time. As the sample approaches the roughness grade of the polishing solution, the RMS roughness plateaus.

Etched CR-39 samples were observed after grinding and polishing, to establish a level of surface material removal that would eliminate noise and preserve the charged particle-induced etched pits. Images from an optical microscope in Figure 9 show that the grinding and polishing procedures remove shallow data pits, as well as noise pits. A method was therefore needed to increase the depth of the etched data pits without affecting noise pits.

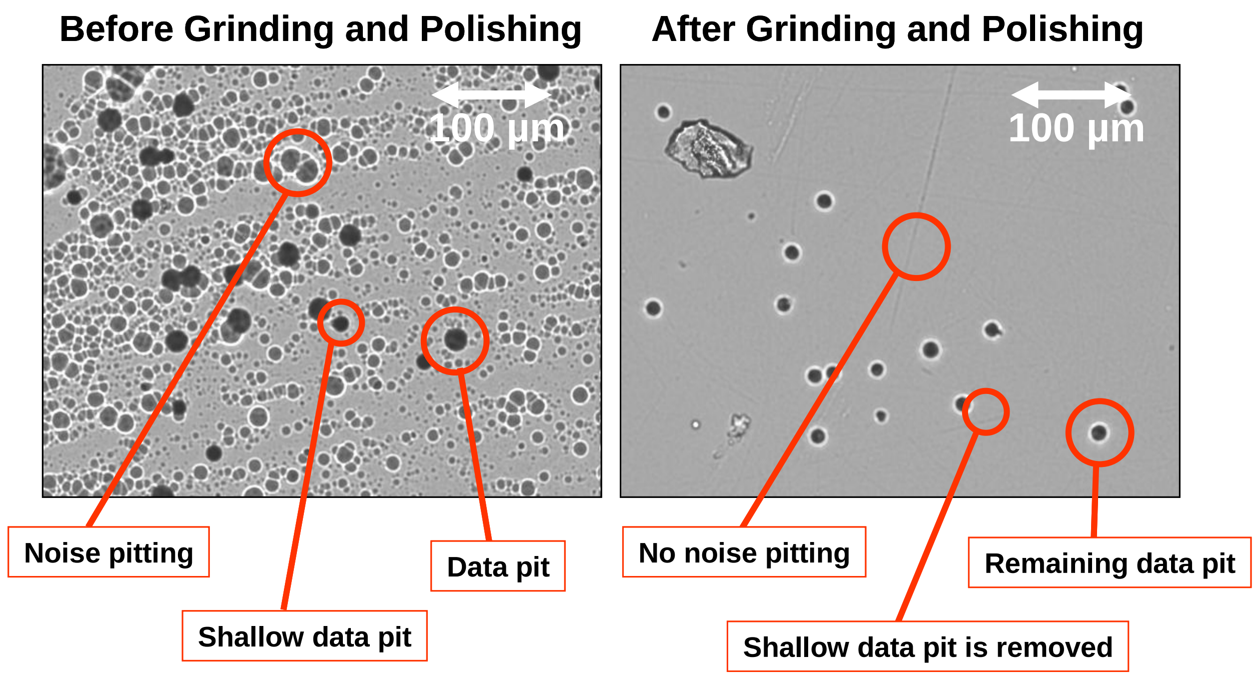

Figure 9:
CR-39 sample imaged at the same coordinates before and after grinding and polishing off 3-4 µm of surface material. Shallow data pits have been eliminated as well as noise pits.

Ultraviolet light exposure techniques:

When a CR-39 sample is etched, the NaOH etchant disrupts chemical bonds at the material surface, as well as along the latent tracks left by charged ions. The rate at which CR-39 is etched along the surface is known as the bulk etch rate. The rate at which material is removed from the latent tracks is called the track etch rate. The ratio of these rates, known as the track-to-bulk etch ratio, determines the resultant pit depth and diameter. Figure 10 shows how the etch ratio affects the pit shape. Exposure of CR-39 to UV irradiation has been confirmed to increase the track to bulk etch ratio of the polymer[2]. UV irradiation incident on a sample disrupts the broken chemical bonds of latent tracks such that, when etched, more track material in proportion to bulk material is etched.

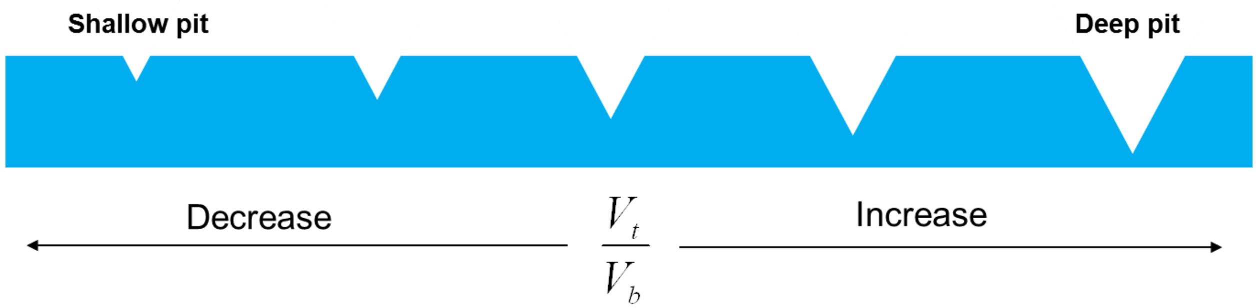

Figure 10:
The effect of the track-to-bulk etch ratio of CR-39 on the etched pit shape. UV exposure increases the track-to-bulk etch ratio, increasing the pit depth and diameter.

UV irradiation was therefore tested as a possible method for increasing the data pit depth and diameter in CR-39, such that data pits would not be removed as a result of grinding and polishing. Three light-emitting sources were tested for their effects on increasing the track-to-bulk etch ratio in CR-39. To optimize UV exposure, UV spectral emission was measured with a spectrometer. The spectra of the three tested sources are represented graphically in Figure 11.

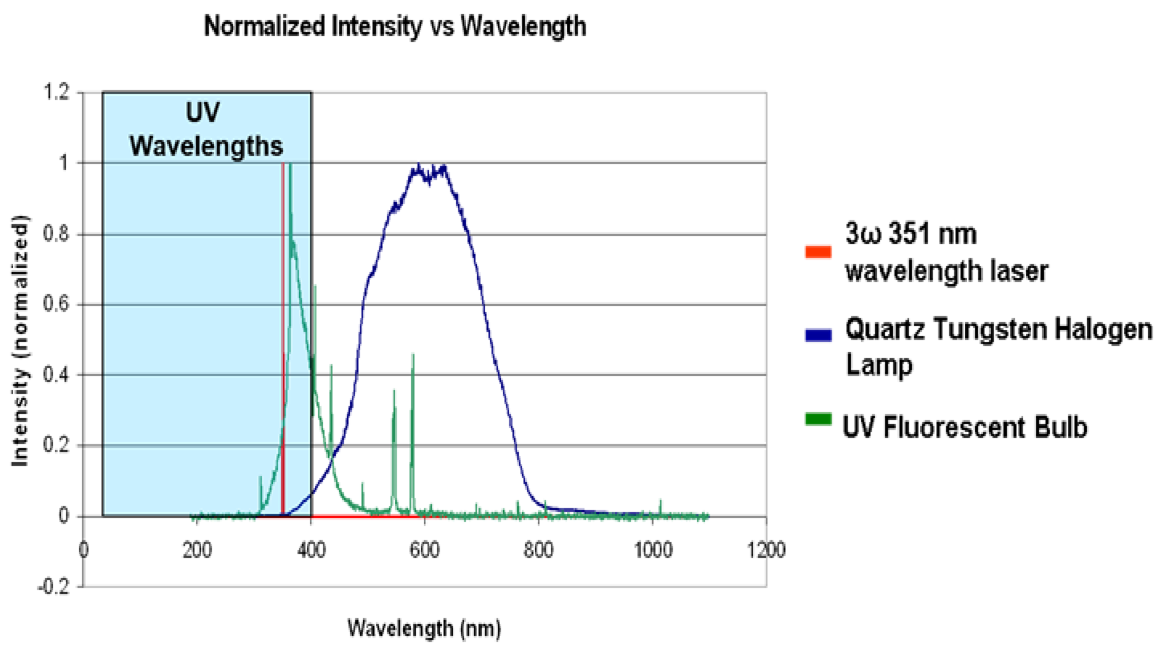

Figure 11:
The full spectral graphs of three tested UV sources, normalized intensity plotted against wavelength. The UV fluorescent bulb shows the broadest UV spectral emission, with 43.3% of its power in the UV spectrum.

A quartz halogen tungsten lamp with a power input of 150 W at 21 V and a power output of 0.87 W was determined to emit only 0.614% of its detected frequencies in the ultraviolet range. When exposed to the halogen source for six hours, alpha particle-induced latent tracks showed only a 10% increase in pit depth after etching. A UV fluorescent bulb array was also tested for its spectral output, and was determined to emit 43.3% of its detected frequencies in the ultraviolet range. As shown in Figure 12, 48 hour exposure to the UV fluorescent bulb array increased 1-MeV proton-induced etched pit diameters by 40.9%, indicating an increase in pit depth as well.

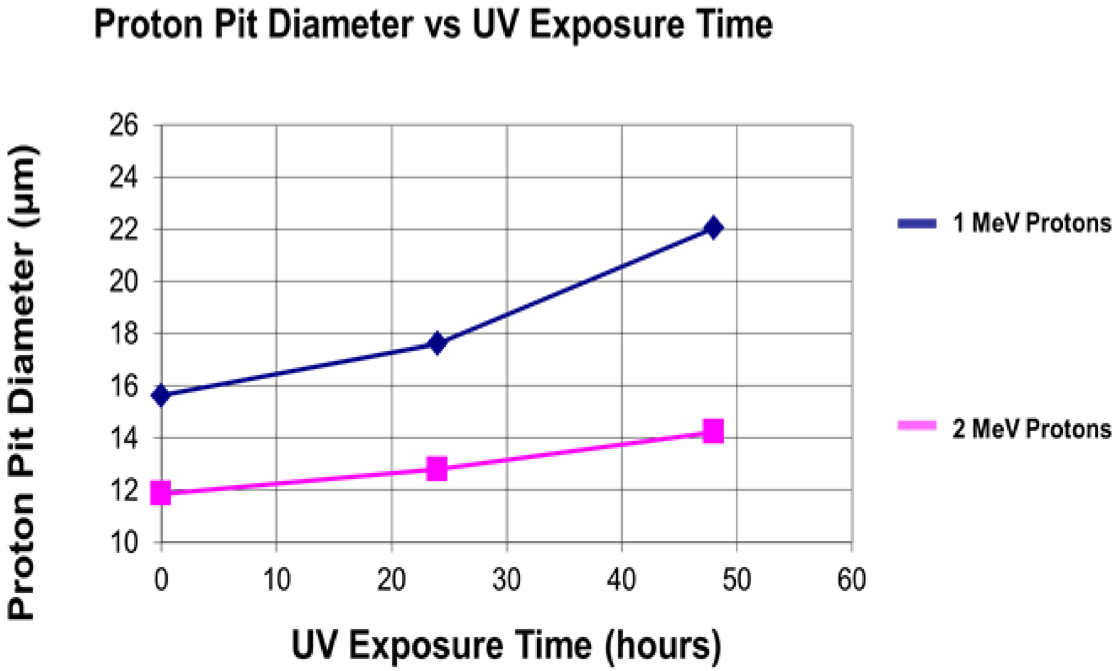

Figure 12:
Proton-induced etched pit diameters as a function of exposure to an ultraviolet bulb array. After 48 hours of exposure and a 6 hour standard etch, 1-MeV proton pits are 40.9% wider in diameter.

To expand upon the success of the UV bulb array, a 3ω 351-nm wavelength laser was tested for its effects on CR-39 etched pits. The 3ω laser was hypothesized to affect CR-39 latent tracks quicker and more efficiently than the UV fluorescent bulb array, due to its controlled, focused emission of a single wavelength of ultraviolet light as shown in Figure 11. However, as shown in Figure 13, CR-39 samples exposed to the 351-nm wavelength laser at intervals of 1, 10, and 100 minutes showed no discernable correlation between alpha particle-induced pit depth and exposure time. This suggests that the wavelength of the 3ω laser, 351nm, is not the optimal wavelength for UV exposure.

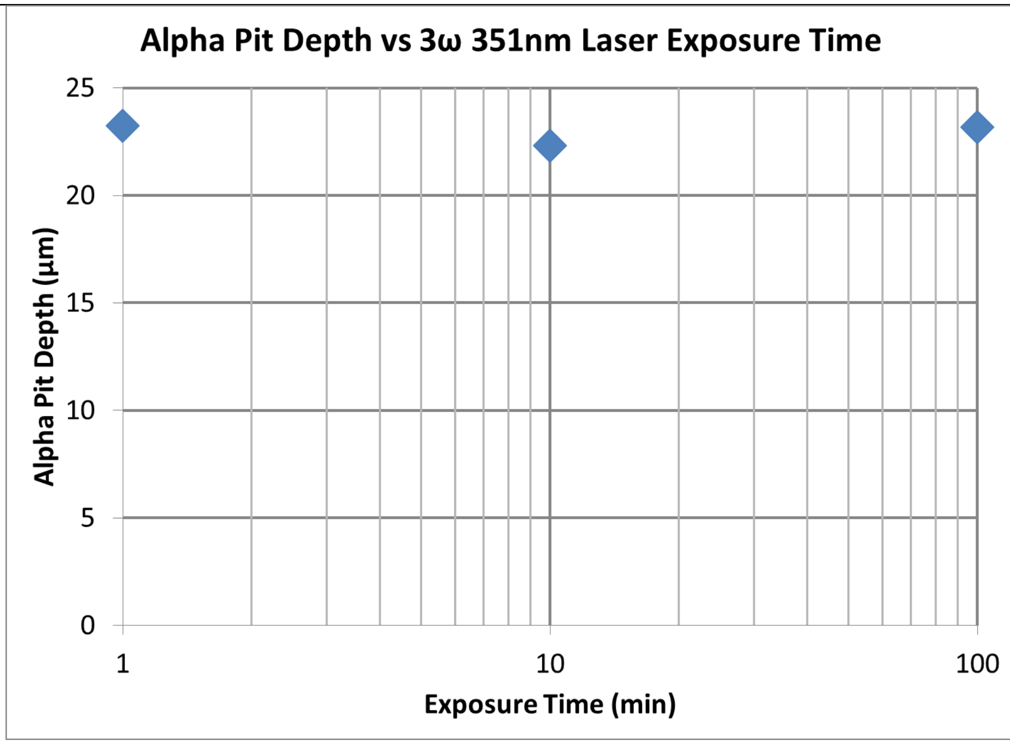

Figure 13:
Alpha particle-induced etched pit depth plotted against 3ω UV laser exposure time, with a logarithmic x-axis scale. The control CR-39 sample with 0 minutes of UV exposure had etched alpha pits averaging 22.5 μm in depth. As the pit depth remained relatively constant throughout each trial, it was concluded that there is no correlation between 351-nm wavelength UV and etched pit depth.

It was hypothesized that a wavelength exists at which the scattered particles in the latent tracks of CR-39 would resonate, such that the etching process would produce deeper, wider pits. The broad-spectrum halogen lamp did not include enough UV intensity in its spectrum to feasibly affect pit depth and diameter. The broad-spectrum UV bulb array included this wavelength in its spectrum, and therefore increased the resulting pit depth and diameter. Despite its high-powered UV emission, the 3ω UV laser did not emit the correct wavelength to create a discernable effect. Future experimentation, as shown in Figure 14, will determine the optimal wavelength for UV exposure. A broadband UV source will be refracted through a prism and onto an exposed sample of CR-39. Due to the refractive properties of the prism, the wavelength of light incident on the sample will change with position. When etched, the position of the deepest pits on the sample will correspond to the optimal wavelength for UV exposure.

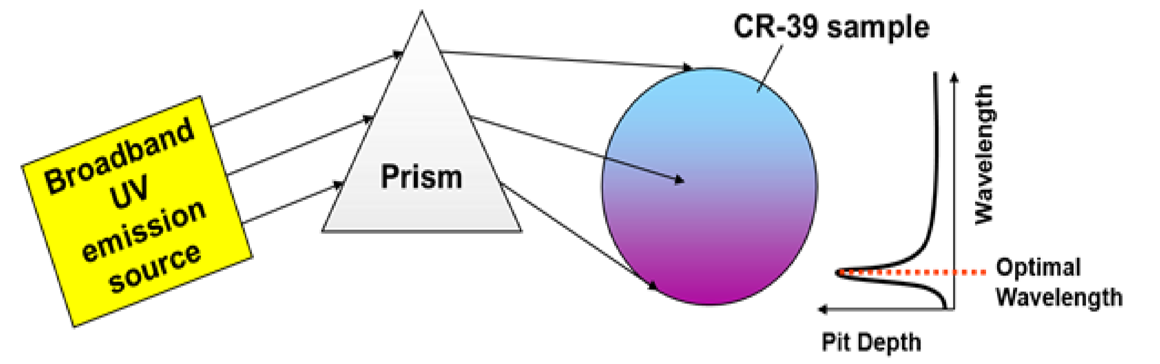

Figure 14:
Future schematic for optimization of UV exposure of CR-39. A broad spectrum of UV light is refracted through a prism such that the wavelength of light incident on a CR-39 sample will change with position. After etching, the deepest pits will correspond to the optimal wavelength.

Application of UV exposure:

A noisy CR-39 sample which had been irradiated with 1-MeV and 2-MeV protons was exposed to UV light for 24 hours from the UV fluorescent bulb array. This sample was etched under standard conditions, and successively ground and polished for 30 seconds and 6 minutes, respectively. To test the application of CR-39 UV exposure to the optimized noise removal process, the sample was imaged at the same place before and after grinding and polishing. As shown in Figure 15, most of the proton pits remain after grinding and polishing, indicating that UV exposure is valid as a technique to increase proton pit depth.

Before Grinding and Polishing | After Grinding and Polishing

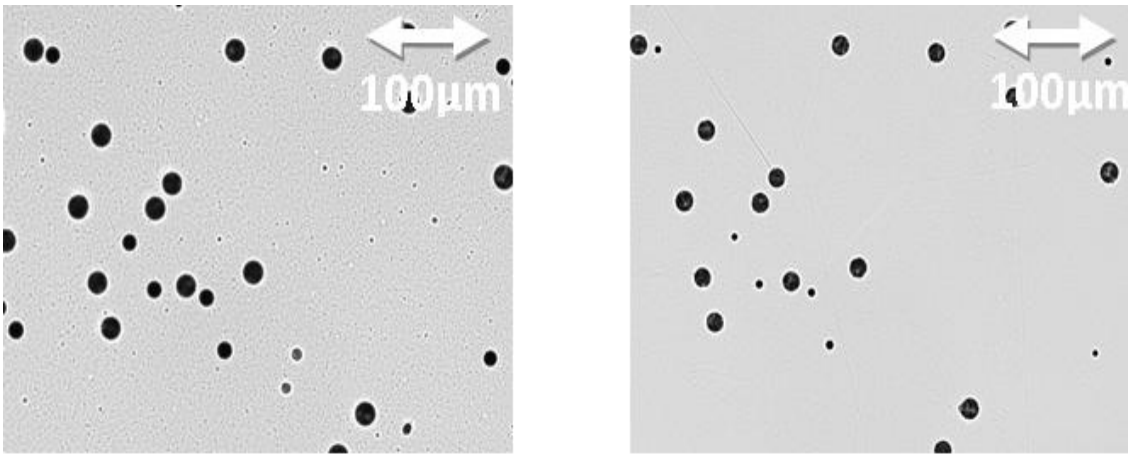

Figure 15:
Images of a CR-39 sample taken at 40x magnification before and after grinding and polishing. The sample was exposed for 24 hours under a UV fluorescent bulb to increase the pit depth. The majority of proton-induced data pits remain as a result.

Conclusions:

Grinding and polishing procedures were tested and confirmed as a feasible method to reduce noise in CR-39 diagnostics. The grinding and polishing process was established to remove noise pits in etched samples; unfortunately, the procedure also removed shallow data pits. To deepen the shallow data pits without affecting noise pits, UV exposure of CR-39 was investigated. When exposed to a broad-spectrum UV bulb array for 48 hours, CR-39 exhibited a 40.9% increase in 1-MeV proton pit diameter, indicating an increase in depth due to UV light. Future investigation of UV exposure will determine the optimal laser wavelength to increase data pit depth; the broad-band UV source is very inefficient. The time-efficient grinding and polishing process was used on a noisy CR-39 sample which had been exposed to 24 hours of broadband UV light; the noise was eliminated and most of the data pits remained. This confirms UV irradiation, grinding, and polishing as a viable set of procedures to remove etch-induced noise pitting in CR-39 samples.

Acknowledgements:

I would like to express my gratitude to my advisor Michelle Burke for her persistent enthusiasm and support. Also, I would like to thank Robert Boni, Joe Katz, and Dr. Stephen Jacobs for their invaluable insight and guidance. Finally, I would like to thank Program Director Dr. Stephen Craxton for providing this unique research opportunity.

References:

[1] Hicks, Damien G. Charged Particle Spectroscopy: A New Window on Inertial Fusion. Ph. D. thesis, Massachusetts Institute of Technology (1999).

[2] Z. Arif, M. Saiyid-Uz-Zafar, G. Hussain, H. A. Khan, K. Jamil, I. M. Siddiqui. "Improvement in the sensitization of the plastic track detectors by ultraviolet irradiations." International Journal of Radiation Applications and Instrumentation (1985).