POST IRRADIATION EXAMINATION RESULTS OF THE NT-02 GRAPHITE FINS NUMI TARGET*

K. Ammigan†, P. Hurh, V. Sidorov, R. Zwaska, FNAL, Batavia, IL 60510, USA
D. M. Asner, A. M. Casella, D. J. Edwards, A. L. Schemer-Kohrn,
D. J. Senor, PNNL, Richland, WA 99352, USA

Abstract

The NT-02 neutrino target in the NuMI beamline at Fermilab is a 95 cm long target made up of segmented graphite fins. It is the longest running NuMI target, which operated with a 120 GeV proton beam with maximum power of 340 kW, and saw an integrated total proton on target of 6.1×10^{20}. Over the last half of its life, gradual degradation of neutrino yield was observed until the target was replaced. The probable causes for the target performance degradation are attributed to radiation damage, possibly including cracking caused by reduction in thermal shock resistance, as well as potential localized oxidation in the heated region of the target. Understanding the long-term structural response of target materials exposed to proton irradiation is critical as future proton accelerator sources are becoming increasingly more powerful. As a result, an autopsy of the target was carried out to facilitate post-irradiation examination of selected graphite fins. Advanced microstructural imaging and surface elemental analysis techniques were used to characterize the condition of the fins in an effort to identify degradation mechanisms, and the relevant findings are presented in this paper.

INTRODUCTION

The NT-02 neutrino target in the NuMI beamline at Fermilab produced neutrinos for the MINOS and MINERVA high energy physics experiments. The NT-02 target, 95 cm long and composed of segmented graphite fins as shown in Fig. 1, was bombarded with 340 kW beam of 120 GeV protons. During operation from 2006 to 2009 and again in 2011, it was subjected to an integrated total of 6.1×10^{20} protons on target with a Gaussian beam sigma of 1.1 mm and peak fluence of 2.5×10^{22} p/cm^2. With the 10 µs beam pulse length and cycle time of 1.87 s, the target experienced rapid temperature cycling from 60 °C to 330 °C during operation. The target is operated in a helium environment and the graphite fins bonded to water cooling tubes, as shown in Fig. 1.

Over the last half of the NT-02 target's lifetime, a gradual decline in neutrino yield of 10-15% was observed [1]. This performance degradation was not detected in preceding NT targets, although their lifetimes were roughly half that of the NT-02 target. The probable causes for the target performance degradation was therefore attributed to material radiation damage, cracking caused by reduction of thermal shock resistance, or localized oxidation in the heated region of the target. The peak radiation damage in the graphite fins was estimated to be 0.63 displacements per atom (DPA), as calculated by the MARS Monte Carlo code [2].

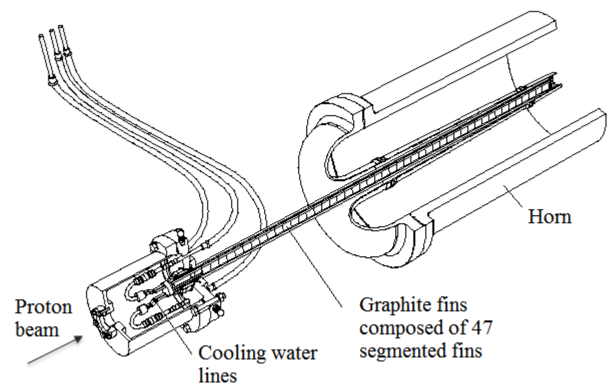

Figure 1: NuMI graphite target.

During the removal and disassembly of the NT-02 target at Fermilab, several graphite fins were discovered to be cracked near the centerline or broken away from the cooling water tubes as shown in Fig. 2. It was unclear whether the separation and fracture of the fins had occurred during the removal and disassembly operations. As a result, studies were initiated to explore the NT-02 graphite fins. The main objectives of the investigation were to determine whether the neutrino yield degradation was a result of radiation damage, by measuring bulk swelling, evaluating the fin fracture surfaces to determine whether they occurred in service or during disassembly, and finally by evaluating the microstructural condition to assess the extent of radiation damage of the POCO ZXF-5Q [3] graphite.

Figure 2: Boroscope images of NT-02 graphite fins during target autopsy. Broken fins from cooling tubes (left) and fractured fin at centerline (right).

POST IRRADIATION EXAMINATION

Four NT-02 fins were retrieved and shipped in a Type-A container to Pacific Northwest National Laboratory (PNNL),

* Work supported by Fermi Research Alliance, LLC, under Contract No. DE-AC02-07CH11359 with the U.S. Department of Energy.
† ammikav@fnal.gov

which has well established capabilities for Post Irradiation Examination (PIE) studies of activated specimens [4]. The four fins consisted of one intact fin and one fractured fin taken from an upstream location in addition to one intact fin and one fractured fin taken from the downstream location of the target. The preliminary findings from the PIE work are presented in the subsequent sections.

Dimensional Measurements

Measurements of the physical dimensions of the fins were first performed to determine if there was any indication of swelling. The thickness and width of each fin was measured with a micrometer and caliper respectively, in the pattern indicated in Fig. 3. The three thickness measurements in the center (4-6 for full fin and 1-3 for half fin) are along the proton beam axis and therefore in the high fluence region, whereas the measurements at the ends are in the lower fluence region.

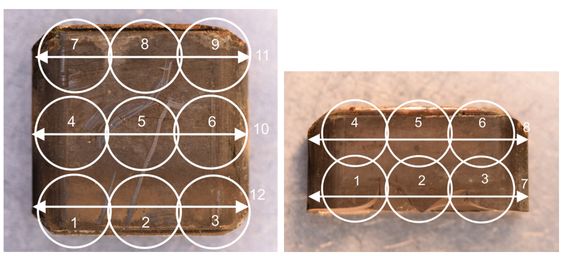

Figure 3: Dimensional measurement locations on full fins (thickness: 1-9, width: 10-12) and half fins (thickness: 1-6, width: 7-8).

Results indicate bulk swelling, with greater swelling measured in the middle of the fins versus the ends and in the upstream fins versus the downstream ones. Relative swelling (middle-to-end) of up to 2% was measured for the upstream half fin. The results are consistent with the higher proton fluence in the middle of the fins as well as the greater damage rate at the upstream end of the target. Another trend observed in the measurements is larger swelling in the half fins versus the full fins, possibly indicating stress relief after fracture.

SEM Analyses

Optical and Scanning Electron Microscopy (SEM) images of the fin fracture surfaces were taken to analyze how the fractures initiated. Figure 4 shows SEM images of the fracture surfaces of the upstream half fin (top image), the downstream half fin (middle image), and of an intentionally fractured upstream full fin (bottom image) with pressure from a razor blade. The preliminary conclusion from these images is that the fracture likely initiated from the center of the fins in the region of peak proton fluence. The evidence for this is in the symmetry propagation pattern exhibited by the half fractured fins. Even though the patterns are not entirely symmetrical, the contrast to the asymmetrical fracture pattern of the intentionally fractured upstream full fin is very clear. For the half fins, the fracture propagation ripples can be seen from the central fracture initiation point.

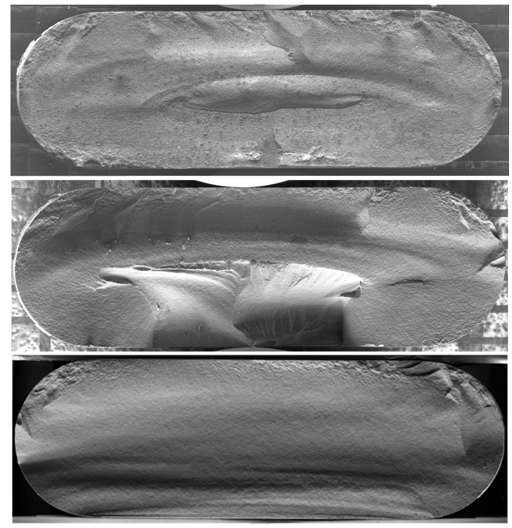

Figure 4: SEM images of fracture surfaces of upstream half fin (top), downstream half fin (middle), and intentionally fractured upstream full fin (bottom).

Also evident from SEM images of the fractured surfaces are loose particulate dispersed across the fracture surfaces of the already fractured fins. On the other hand, debris are not observed along the fracture surface of the intentionally fractured full fin. This observation again supports the notion that the fins likely fractured during operation. Additionally, discolorations and structural formations that are optically visible on the fractured fins are not visible on the fracture surface of the intentionally fractured fin, as shown in Fig. 5. This is additional evidence of material transport along the fracture surface during operation, further suggesting that the fins fractured during operation.

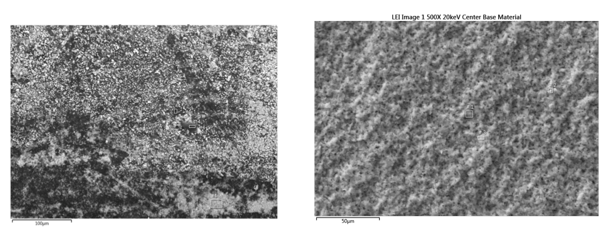

Figure 5: Magnified SEM images of fracture surfaces of upstream half fin (left) and intentionally fractured upstream full fin (right).

EDS and TEM Analyses

Energy Dispersive Spectroscopy (EDS) and Transmission Electron Microscopy (TEM) analyses were also performed to obtain elemental maps and probe the microstructure of the graphite fins at different locations. The first Focused Ion Beam (FIB) lamella lift out for TEM and EDS analyses came from the side of the upstream half fin close to the fracture surface. Figure 6 shows the SEM image of the FIB extracted lamella (left) and the TEM image of the microstructure. The microstructure is seen to be a mix of larger crystalline regions and smaller nanoscale crystalline regions. Mrozowski cracks that commonly exist in as-fabricated graphites are also visible. These cracks are identifiable by the brighter lamellar bands on the TEM image and usually develop parallel to the graphite basal planes during cool-down after graphitization. The microstructure observed is highly consistent with as-fabricated microstructure.

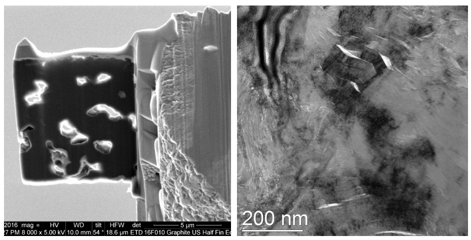

Figure 6: SEM image (left) and TEM image (right) of the lamella lift out from the side of the upstream half fin.

The elemental map in Fig. 7 shows impurities decorating the interior surface of a pore. Sn, Zn and Cl impurities are likely associated from the solder and flux used to join the graphite fins to the cooling water tubes. The location of these impurities suggests that vapor created during fabrication or operation penetrated the open porosity and deposited within the pores of the graphite.

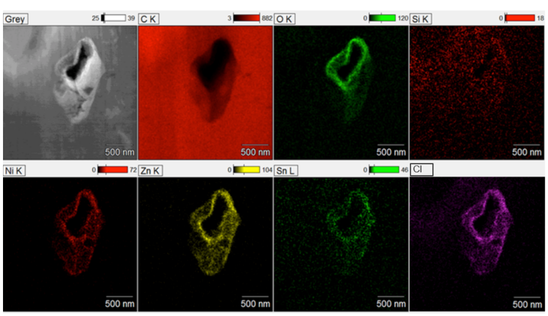

Figure 7: EDS elemental mapping of a pore located on TEM sample from the side of the upstream half fin.

Another TEM sample was lifted out from the center of the fracture surface of the downstream half fin to examine the microstructure of the graphite exposed to the peak proton fluence. As shown in Fig. 8, the lamella has mixed regions of what appear to be amorphous (encircled yellow diffraction pattern) and nanocrystalline microstructure (encircled red). Mrozowski cracks are also present at the interfaces between the two regions, suggesting that lattice swelling is moderate. Therefore, the bulk swelling measured may be a result of a combination of moderate lattice swelling and localized amorphization caused by radiation damage and thermal effects.

TEM analyses were also carried out on other specimens lifted out from multiple locations on both upstream and downstream fins, and all reveal a mixture of crystalline and nano-crystalline regions throughout the samples. From the TEM images, it is not clear whether the microstructure of the graphite is significantly altered between regions of the fins that were exposed to high proton fluence and those that were not. The crystalline and nano-crystalline regions observed is consistent with as-fabricated graphite and no clear evidence of any significant change in the microstructure due to proton irradiation is seen.

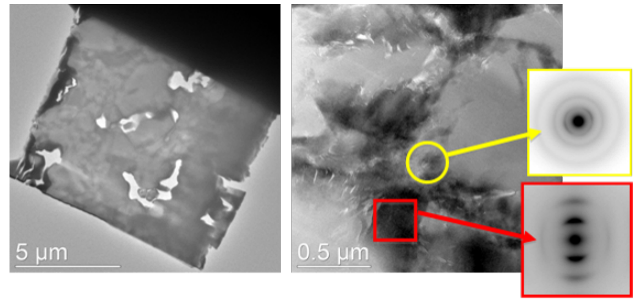

Figure 8: TEM bright field image of TEM specimens taken from center of fracture surface of downstream half fin.

CONCLUSION

PIE activities to investigate the selected NuMI NT-02 graphite fins were carried out successfully [5]. Dimensional measurements confirmed bulk swelling of the fins which was seen to be more prominent in the peak proton fluence region along the centerline of the fins. Fracture surface analyses also support the idea that fracture initiated in the middle of the fin along the proton beam axis during operation. No strong evidence of radiation damage to the microstructure of the graphite was observed. As such, preliminary conclusions are that the reduced density induced by swelling as well as the fracture along the centerline of some fins during operation may be partially responsible for the neutrino yield degradation observed during the target's lifetime.

REFERENCES

[1] J. Hylen, "NuMI Target Info for MINERVA Run Plan Review", unpublished.

[2] N. V Mokhov, "The MARS Code System User's Guide", Fermilab-FN-628, 1995.

[3] POCO ZXF 5Q, `http://poco.com/MaterialsandServices/Graphite/IndustrialGrades/ZXF5Q.aspx`

[4] D. J. Senor, "NSUF Partner Facility Capabilities at the Pacific Northwest National Laboratory", presented at the RaDIATE Meeting, Brookhaven National Laboratory, Upton, NY, Nov. 2015.

[5] A. M. Casella *et al.*, "Results from Post-Irradiation Examination of Graphite from the NuMI NT-02 Target", presented at the RaDIATE Collaboration Meeting, Pacific Northwest National Laboratory, Richland, WA, Sept. 2016.