USED FUEL DISPOSITION CAMPAIGN

Optimization of Hydride Rim Formation in Unirradiated Zr-4 Cladding

Fuel Cycle Research & Development

Prepared for
U.S. Department of Energy
Used Fuel Disposition Campaign

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EXECUTIVE SUMMARY

This report fulfills the M3 milestone M3FT-13PN0805057 under Work Package Number FT-13PN080505.

The purpose of this work is to build on the results reported in the M2 milestone M2FT-13PN0805051, document number FCRD-USED-2013-000151 (Hanson et al., 2013). In that work, it was demonstrated that unirradiated samples of zircaloy-4 cladding could be pre-hydrided at temperatures below 400°C in pure hydrogen gas and that the growth of hydrides on the surface could be controlled by changing the surface condition of the samples and form a desired hydride rim on the outside diameter of the cladding. The work performed at Pacific Northwest National Laboratory since the issuing of the M2 milestone has focused its efforts to optimize the formation of a hydride rim on available zircaloy-4 cladding samples by controlling temperature variation and gas flow control during pre-hydriding treatments. Surface conditioning of the outside surface was also examined as a variable. The results of testing indicate that much of the variability in the hydride thickness is due to temperature variation occurring in the furnaces as well as how hydrogen gas flows across the sample surface. Efforts to examine other alloys, gas concentrations, and different surface conditioning plan to be pursed in the next FY as more cladding samples become available.

ACKNOWLEDGMENTS

The authors thank Karl Mattlin and Nathan Canfield for their assistance with experimental setup and operations.

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ACRONYMS

ASTM	ASTM International
BWR	boiling water reactor
CFR	Code of Federal Regulations
DI	deionized
HF	hydrogen fluoride
ID	inside diameter
NRC	U.S. Nuclear Regulatory Commission
NUREG	publication prepared by staff of the U.S. Nuclear Regulatory Commission
OD	outside diameter
PH	pre-hydrided (cladding)
PNNL	Pacific Northwest National Laboratory
PWR	pressurized water reactor
RPD	Relative Percent Deviation
sccm	standard cubic centimeters per minute
scfm	standard cubic feet per minute
UFDC	Used Fuel Disposition Campaign
UNF	used nuclear fuel
U.S.	United States (adjective)
wppm	parts per million by weight
Zr-2	Zircaloy-2
Zr-4	Zircaloy-4

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1. INTRODUCTION

The U.S. Department of Energy Office of Nuclear Energy, Office of Fuel Cycle Technology has established the Used Fuel Disposition Campaign (UFDC) to conduct the research and development activities related to storage, transportation, and disposal of used nuclear fuel (UNF) and high-level radioactive waste. Within the UFDC, the storage and transportation task has been created to address issues of extended storage and transportation. The near-term objectives of the storage and transportation task are to use a science-based, engineering-driven approach to

- develop the technical bases to support the continued safe and secure storage of UNF for extended periods
- develop the technical bases for retrieval of UNF after extended storage
- develop the technical bases for transport of high burnup fuel, as well as low and high burnup fuel after dry storage.

Current regulations of the U.S. Nuclear Regulatory Commission (NRC) require (10 CFR 72.122(h)) that "spent fuel cladding must be protected during storage against degradation that leads to gross ruptures or the fuel must be otherwise confined such that degradation of the fuel during storage will not pose operational safety problems with respect to its removal from storage." Gross ruptures or breaches are defined in NUREG-1536 (NRC 2010) as any cladding breach greater than 1 mm. Thus, even though NRC does not explicitly consider cladding as a confinement barrier, as evidenced by failed fuel assemblies being allowed into dry cask storage systems as long as they are in a damaged fuel can, the state and material properties of the cladding currently are still important to licensing.

For the purposes of the UFDC program, retrievability and operational safety concerns also apply to the fuel after transportation so that, if necessary, the fuel can be transloaded into waste packages for disposal or handled in a reprocessing facility. In January 2013, NRC formally solicited public comment (Federal Register, Volume 78, Issue 12) for potential rulemaking changes that could affect retrievability, cladding integrity, and safe handling of spent fuel. The UFDC continues to pursue alternatives to individual fuel assembly retrievability (e.g., canning individual or small numbers of assemblies or direct disposal of canisters). Such alternatives may facilitate the demonstration of subcriticality in the case of cladding damage and fuel relocation or minimize the need to maintain cladding integrity. However, until regulations change and it can be demonstrated that for future waste management needs it is no longer necessary, fuel assembly retrievability remains a key feature for the UFDC.

The *Gap Analysis to Support Extended Storage of Used Nuclear Fuel* (Hanson et al. 2012a, also known as the UFDC Gap Analysis) was prepared to document the methodology for determining the data gaps and to assign an initial priority (Low, Medium, High) of importance for additional research and development to close the data gaps. The analysis was based on normal conditions of extended storage and informed by subsequent transportation needs. UFDC performed a second, more quantitative prioritization of the research to address the High and Medium priority gaps in the draft report *Used Nuclear Fuel Storage and Transportation Data Gap Prioritization* (Hanson et al. 2012b, also known as the UFDC Gap Prioritization). This prioritization report also considered anticipated high- and medium priority gaps associated with transportation and the design-basis phenomena and accident conditions during extended storage.

One of the gaps identified as a high priority is Hydrogen Effects: Embrittlement and Reorientation. The work of Billone et al. (2008, 2013) in support of NRC and the UFDC has shown that at high temperatures and stress, the normally circumferential hydrides found in high burnup (i.e., >45 gigawatt-days per metric ton uranium) cladding can reorient in the radial direction and result in brittle mechanical properties. Cladding that has cooled below this ductile-to-brittle transition temperature may be susceptible to breach when subjected to outside stresses such as during handling or normal conditions of transport.

Billone et al. (2013) compared the results of ring compression tests on unirradiated, pre-hydrided (PH) cladding with the cladding of high burnup fuel. They found that the PH samples had significantly higher ductility than the high burnup cladding. Two significant differences exist between the PH and high burnup cladding: 1) the PH cladding had a relatively uniform distribution of hydrides across the cladding radius whereas the high burnup cladding of approximately equal total hydrogen content exhibited a non-uniform distribution across the radius with a high concentration of hydrides at the outer diameter (i.e., a rim) and 2) the PH cladding had no irradiation damage whereas the radiation damage in high burnup cladding is known to significantly decrease ductility and increase hardness.

Multiple laboratories within the UFDC are performing separate effects tests to determine the role various parameters (hydride content and distribution, radiation damage, oxide thickness, etc.) play in determining the mechanical properties of irradiated cladding and how they change over time in extended storage. Billone et al. (2013) concluded that the difference in hydride distribution between PH and irradiated cladding was significant. Separate effects tests at Pacific Northwest National Laboratory (PNNL) were initiated to develop a means for pre-hydriding cladding such that the concentration, distribution, and morphology are the same as in high burnup cladding.

This report outlines the progress PNNL has made to optimize a PH process that produces a consistent hydride rim around the circumference of unirradiated as-manufactured cladding samples, and the factors that are important.

2. BACKGROUND

Alloys of zirconium (i.e., Zircaloy) have been used as cladding for nuclear fuel because of its low thermal neutron absorption cross section and their relatively good corrosion resistance and resistance to radiation damage. The typical alloying elements are tin (Sn), iron (Fe), chromium (Cr), niobium (Nb), nickel (Ni), and oxygen (O). The elements Sn and O are interstitial strengtheners whereas Fe, Cr, Ni and Nb tend to form intermetallics and are found as discrete second phases in the zirconium (Zr) matrix. The alloying elements and composition are varied to provide the proper corrosion resistance while maintaining strength and ductility to prevent failures during reactor operations.

For example, it was discovered that the presence of Ni in Zircaloy-2 (Zr-2) promoted absorption of hydrogen. Since Pressurized Water Reactors (PWRs) use an overpressure of hydrogen in the reactor coolant, Zircaloy-4 (Zr-4) was developed where the Ni was replaced with additional Fe to maintain corrosion resistance. Thus, most cladding in Boiling Water Reactors (BWRs) is Zr-2 whereas PWRs have used Zr-4. Recently, newer alloys such as ZIRLO™ and M5® have been developed to further reduce corrosion as the trend is to go to higher burnups and thus longer residence time of the fuel in the reactor core. These newer alloys use Nb to improve corrosion resistance. The common Zircaloy alloys used for commercial fuel cladding production are given in Table 2.1.

Table 2.1 Nominal compositions of Zirconium based nuclear fuel clad alloys.

Alloy	Sn (wt%)	Fe (wt%)	Cr (wt%)	Nb (wt%)	Ni (wt%)	O (wt%)	C (wt%)	Si (wt%)	Zr (wt%)
Zr- 2 (BWR)	1.5	0.12	0.1		0.05	0.13			Balance
Zr- 2 (BWR) Improved	1.3	0.17	0.1		0.06	0.13			Balance
Zr -4 (PWR) High Tin	1.55	0.22	0.12			0.12	0.015	0.01	Balance
Zr -4 (PWR) Improved	1.3	0.22	0.12			0.12	0.012	0.01	Balance
M5 ®		0.04		1		0.14			Balance
ZIRLO™	1	0.1		1		0.12			Balance
Optimized ZIRLO ™	0.7	0.1		1		0.12			Balance
UNS R60901 (Zr 2.5Nb) ATSM B353				2.6		0.13			Balance
Zircaloy 2 Specification ASTM B811	1.2 - 1.7	0.07 - 0.20	0.05 - 0.15		0.03 - 0.08	0.09 - 0.16	0.027 max	0.012 max	Balance
Ziracloy 4 Specification ASTM B811	1.2 - 1.7	0.18 - 0.24	0.07 - 0.13			0.09 - 0.16	0.027 max	0.012 max	Balance

These alloying elements influence corrosion behavior, including hydrogen uptake, as well as mechanical properties. Other factors that influence these behaviors are grain size, stress, texture, and cold work. An overview of the manufacturing process, literature review of hydrogen uptake, diffusion, redistribution, and reorientation are presented to help direct modelers and experimentalists determine which factors are important and the role they play.

During reactor operations, the Zircaloy cladding is in contact with the high temperature and pressure reactor coolant (water and/or steam) and corrodes according to the reaction

$$Zr + 2H_2O \rightarrow ZrO_2 + 2H_2$$

Some fraction, called the hydrogen uptake ratio, of the hydrogen is absorbed in the cladding. The chemical composition of the alloy is a primary factor affecting the rate of corrosion and thus hydrogen generation. Newer alloys such as ZIRLO™ and M5® have been developed specifically to reduce the corrosion rate of the cladding. However, in PWRs, a hydrogen overpressure is applied to limit corrosion and, combined with radiolytically-generated hydrogen, can still supply a source of hydrogen for the cladding to uptake.

Texture in the manufactured cladding is primarily controlled by how tubing is cold worked reduced in size as defined by what is commonly called the Q-value. The Q-value is determined from the amount of wall thinning strain divided by the diameter strain during cold work reduction. Figure 2.1 (Schemel 1989) illustrates the impact of Q on final tube texture by showing two different reductions used to produce the same final tube. For both reductions shown in Figure 2.1, the reduction of area is equivalent. The reduction shown on the left side of Figure 2.1 uses a starting material with a smaller outside diameter and thicker wall than the reduction on the right. This reduction results in a larger amount of wall reduction versus diameter reduction, creating a relatively high Q. The reduction on the right side of Figure 2.1 uses a starting material with a larger diameter and thinner wall. This reduction results in a larger amount of diameter reduction versus wall reduction, creating a relatively low Q. The high Q reduction results in what is termed "radial texture" and low Q reduction results in what is termed "circumferential texture".

Texture of the final tube plays an important role in fuel-clad performance due the nature of hydrides formed in the tubes during service. In Zircaloy, hydrides tend to form on the basal plane of the hexagonal unit cell, shown in Figure 2.1 (Schemel 1989). If the tube has a high radial texture (meaning the basal planes are oriented normal to the radial direction), the hydrides, shown as dark lines in Figure 2.2(a), are oriented in the circumferential direction. If a tube was produced with low Q with circumferential texture, the basal planes are oriented in the circumferential direction and the hydrides will be oriented in the radial direction like the dark lines in Figure 2.2(b)). (In Figure 2.2(b) the hydrides were re-oriented by a different method and are only used here to illustrate the difference). The hydrides create a weak direction for fracture and if the hydrides are oriented radially, like the example of Figure 2.2(b), the hoop strength of the tube will be very low and poor reactor or dry storage life may be expected. If the tube is produced with a high Q, the hydrides will be oriented circumferentially creating a tortuous crack path and increased resistance to failure during reactor or dry storage operations.

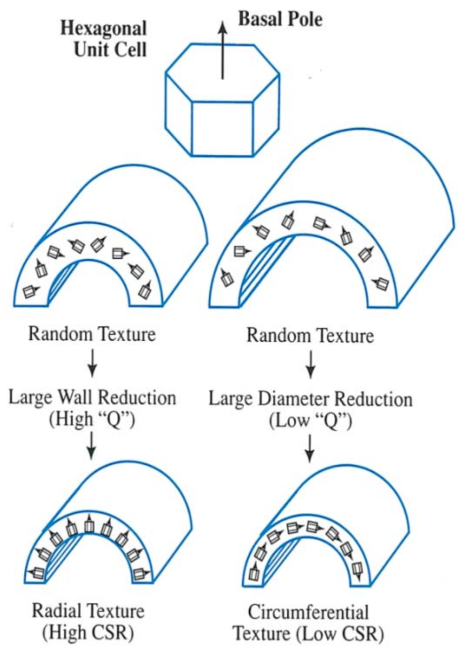


Figure 2.1 Illustration of two reductions used to produce the same tube (bottom) where the one on the left produces a radial texture and the one on the right produces a circumferential texture (Schemel, 1989).

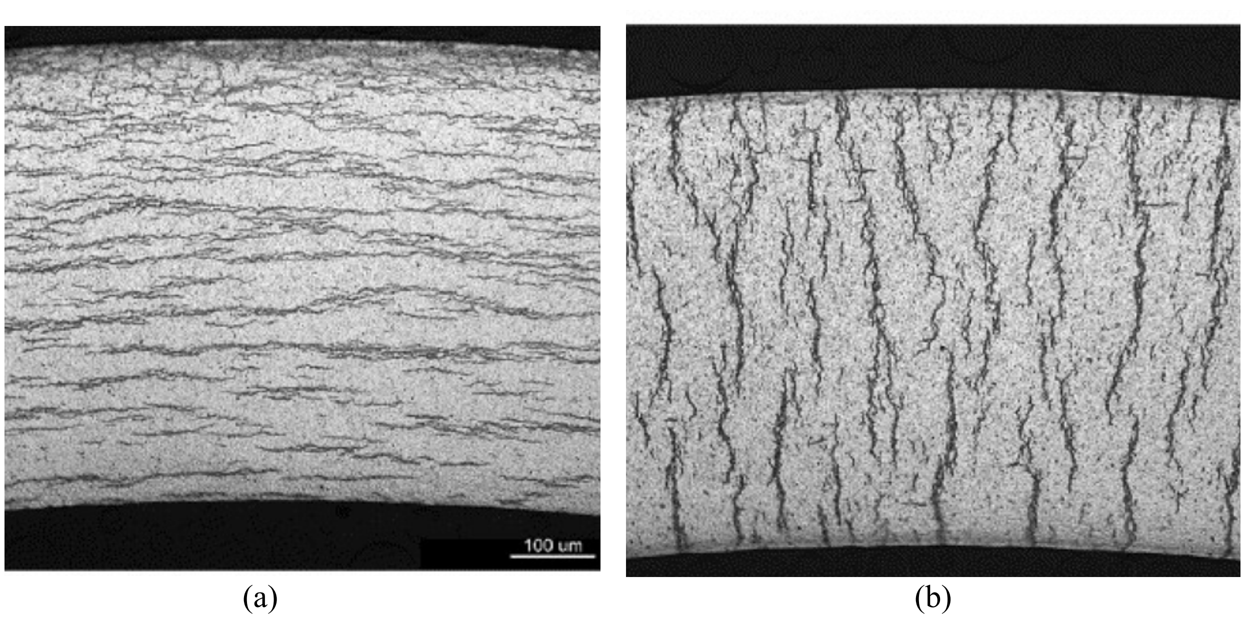
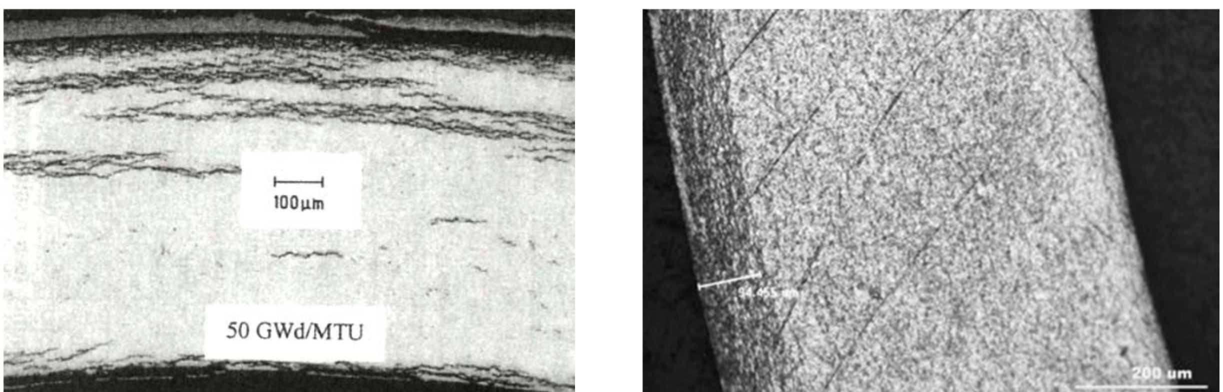


Figure 2.2 The tube on the left (a) was produced using a high Q reduction and resulted in the desirable circumferential orientation of hydride, as shown by the dark lines in the white zirconium matrix. The tube on the right (b) is an example of what the hydrides would look like if a tube were produced with a very low Q final reduction.

During the formation of the oxide layer, the initial designed texture of the cladding causes the hydrides to form in the circumferential direction near the oxide-metal interface, forming a hydride rim around the outside wall [Figure 2.3(a)]. This differs from the image in Figure 2.2(a) where samples were prepared according to ASTM B811-02 to create uniform hydrides across the cross section of the tube wall to validate the texture of the cladding. In that method, the tubing samples are prepared at temperatures at or above 400°C in hydrogen with equivalent surfaces conditions so the surface oxide layers on the outer and inner wall are the same. These two things allow hydrogen to enter the wall at equal rates and diffuse evenly into the wall before the saturation limit of zirconium is reached.

In the M2 milestone (Hanson et al. 2013), samples of Zr-4 were pre-hydrided to achieve heterogeneous deposits on the outside wall [Figure 2.3(b)] that appeared more like irradiated cladding shown in Figure 2.3(a), and not like cladding sample prepared according to ASTM B811 in Figure 2.2(a).

The scope of this document was to expand on the work that successfully produced a rim and attempt to resolve some of the variation issues reported in the M2 milestone (Hanson et al. 2013).



(a) Irraditated Zr-4 Cladding Sample with Typical Hydride Rim

(b) Prehydrided Unirradiated Zr-4 Cladding Sample from Hanson et al. 2013

Figure 2.3 Example of Irradiated Zr-4 cladding Compared to Unirradiated Cladding Pre-Hydride (Hanson et al. 2013)

3. EXPERIMENTAL SETUP

The work at the laboratory since issuing of the M-2 milestone report (Hanson et al. 2013) has focused on optimizing rim consistency on unirradiated Zr-4 cladding. Efforts have continued to concentrate on using low (≤300°C) temperature methods so that this process could be used to hydride cladding that is pre-irradiated at Oak Ridge National Laboratory without annealing out the induced radiation damage that is annealed out in relatively short times at temperatures above 400°C.

3.1 Hydrogen Loading

Unirradiated Zircaloy-4 samples were pre-hydrided using two furnaces:

1. A cold wall hydrogen gas furnace capable of running both argon and hydrogen continuously into the furnace to maintain a constant gas concentration to heterogeneously load samples with hydrogen at temperatures up to 300°C.
2. A tube furnace heated up to 300°C also capable of running both argon and hydrogen continuously into the furnace to maintain a constant gas concentration to heterogeneously load samples as well.

The cold wall furnace (see Figure 3.1) is manufactured by CAMCo Incorporated (San Carlos, CA) and is capable of high vacuum or flowing either hydrogen or argon at 100%, or mixed with each other, during the heat cycle at atmospheric pressure. The furnace is capable of higher heat treatments (2400°C), but is capable of heating samples between 200-350°C. Gas enters the retort through a diffusion plate on the bottom of furnace and exits through a flame arrestor that burns the hydrogen discharge. Gas flow is controlled by manual rotometers with a 30 standard cubic feet per minute (scfm) maximum readout. The furnace is capable of profiling its heating and cooling ramps with a digital controller, as well as electronically recording additional survey thermocouples inside the furnace. Typically, samples between 40-50 mm long were placed in a stainless steel pan in the bottom of the furnace or hung at an elevated height during the heat treatment with a thermocouple nearby to confirm the temperature.

Figure 3.1 CAMCo Hydrogen/Inert gas furnace.

The tube furnace (see Figure 3.2) used was a 24” Thermolyne tube furnace (Model 79400) that also was setup to run with both 100% hydrogen or argon into the annealing chamber continuously during operation. Samples were placed on a ceramic block inside the tube furnace that could place 2” samples vertically (upright) or horizontally into the heat zone of the furnace. The differences between the two furnaces are:

- The CAMCo furnace chamber was a larger diameter furnace designed to reduce materials in a hydrogen gas environment.
- The Thermolyne furnace was smaller, but has a tighter control on the heat zone (±1-2°C), and better control of gas flow.

For all the work done in this document, the outside diameter of the Zr-4 cladding was blasted prior to heat treatment to strip the oxide layer off the surface to increase the flux of hydrogen into the outside wall of the cladding faster. Samples were finished on the outer wall using a grit blasting box (Figure 3.3) using glass frit beads. Samples were placed into the furnace either within an hour of conditioning or after waiting 24 hours. Oxide layer growth can form in air immediately after finishing, so the impact on the time period between conditioning and pre-hydriding was of interest.

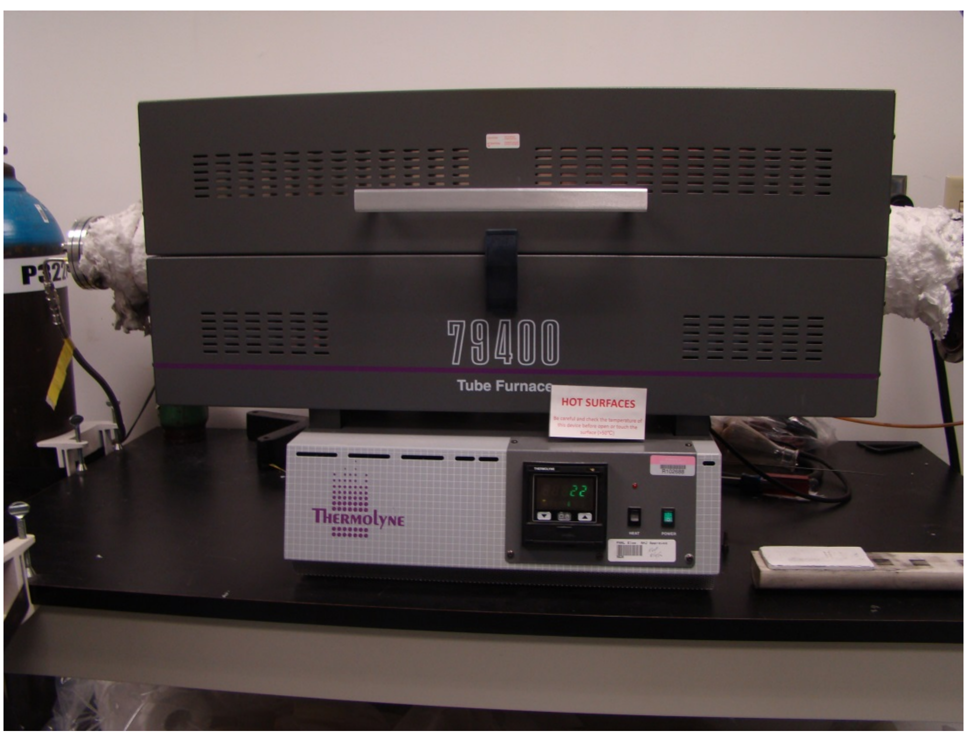


Figure 3.2 Thermolyne Model 79400 Tube Furnace

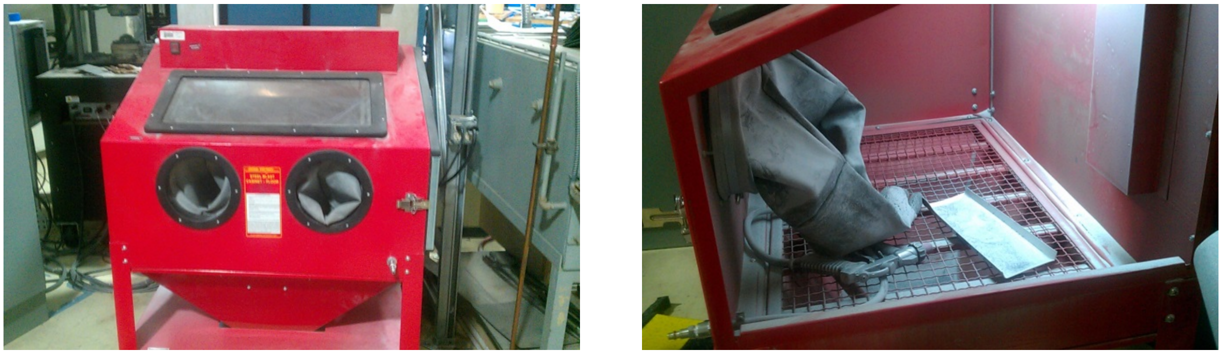
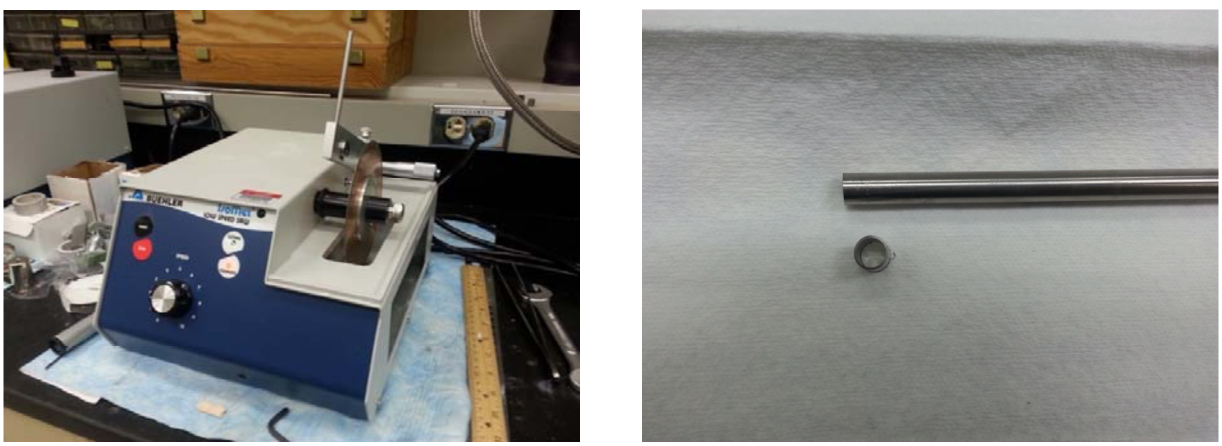


Figure 3.3 Grit Blasting Box used to Condition Cladding Surfaces

3.2 Post-test Examinations

Radial cut tubing samples approximately 8 mm in length were taken from the test specimens for destructive and optical examination. A metallographic saw (Figure 3.4) wet-cut subsamples with a diamond-impregnated copper blade using deionized (DI) water as the blade coolant.



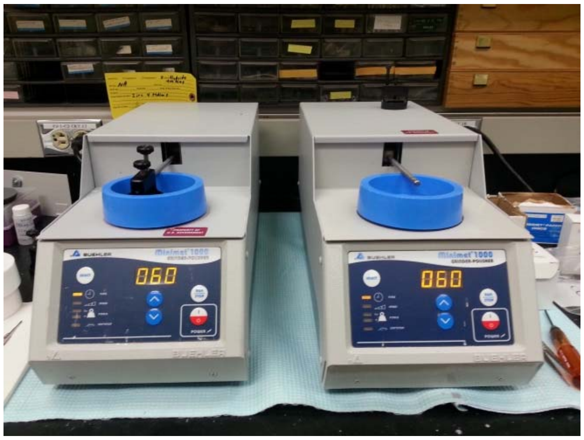
(a) Buehler Isomet® Sample Cutter (b) Typical 8 mm long radial cut tubing sample

Figure 3.4 Metallographic sample saw and radial cut specimen.

Cut radial samples were destructively analyzed for the total hydrogen content in the Zircaloy samples by inert gas fusion, using an analyzer (Figure 3.5) made by the LECO Corporation (St. Joseph, MI). Analyses were performed both at PNNL and at ATI Wah Chang Analytical Services (Albany, OR). Instruments were calibrated prior to each analysis using manufacturer-supplied standards. Cut samples were divided again either into 2 or 4 additional samples to produce analytical duplicates or to verify the uniformity of the hydrogen concentration around the circumference.

Figure 3.5 LECO Series 400 inert gas fusion analyzer.

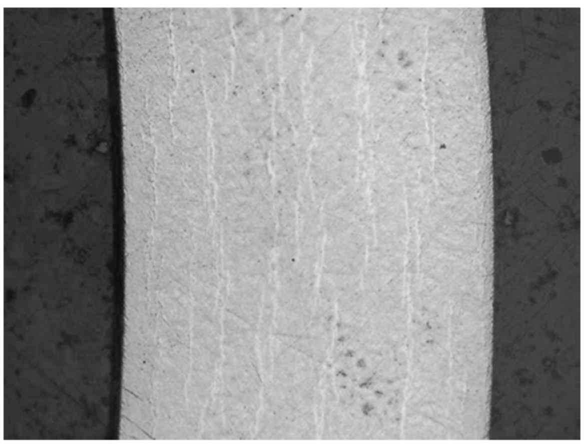
Samples were also examined in the radial direction by metallographic optical microscopy to confirm the results of the gas fusion analysis and qualitatively assess how the hydrides were deposited on the samples. Samples were placed in epoxy circular mounts and wet-polished using a metallographic polisher (Figure 3.6). Silicon carbide (SiC) grit paper with DI water was used, starting with 400 grit and repeating with ever finer grit sizes up to 1200. The number of polishes required varied depending on how scratches were removed during polishing. In some cases, a vibratory polisher was used instead. After polishing, hydrides should be visible but can be difficult to discern from the metal (Figure 3.7). After polishing was completed, a hydrofluoric-nitric acid etch polish was used to preferentially etch the hydrides over the metal. The hydrides appear as dark crevices in the metal surface in an etched sample. This metallographic etch is typically performed while rubbing the mount onto a cotton swab that contains the etchant and is quickly rinsed afterwards. Most of the literature discusses using higher concentrations (4-5 wt%) of hydrofluoric than what was used on this task. However, samples were easily over-etched at this concentration making discerning the hydride present difficult. It was discovered that lowering the hydrofluoric concentration to between 0.5 – 1 wt% was a more robust etching solution and minimized over-etching of samples afterwards.



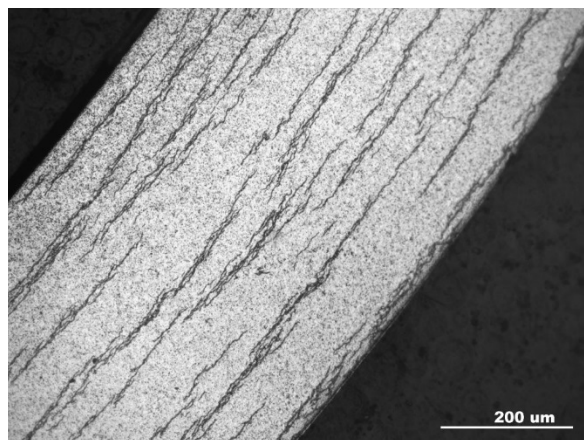
(a) Buehler MicroMet® 1000 Polisher

(b) Buehler Vibratory Polisher

Figure 3.6 Metallographic polishing equipment used.



(a) Polished with SiC up to 1200 Grit



(b) Followed with HF/HNO_3 Etch

Figure 3.7 Metallographic images of hydrided Zircaloy (a) polished with SiC 400-1200 grit paper and (b) followed by a 0.5 wt% HF – 30 wt% HNO_3 etch while polishing with cotton swab and rinsed in DI water.

After etching, each metallography sample was marked into four quadrants, as shown in Figure 3.8 . Pictures of each area were captured at 100x and 200x magnification and consolidated in the appendix of this report.

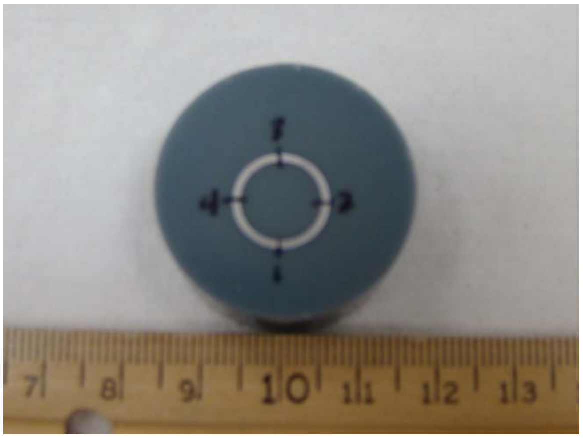


Figure 3.8 Metallography sample mounted, polished, etched, and marked

4. RESULTS

This section describes and summarizes the results of the pre-hydriding (hydrogen loading) tests to produce an optimal hydride rim. Additional information for each sample discussed is found in the Appendix of this report.

4.1 CAMCo Furnace Testing

The testing from the M2 milestone (Hanson et al. 2013) found that cladding samples whose outside diameter were grit blasted, but maintained a manufactured chemical etched inside diameter, could preferentially form hydrides on the outside diameter at furnace control temperatures between 270° - 300°C. However, variation in the rim thickness and in the hydrogen concentration was evident in the samples. This section discusses PNNL's efforts to determine the source of variability in the process and if it was related to the furnace itself.

4.1.1 Thermo-profiling of CAMCo

Upon completion of the M2 milestone testing (Hanson et al. 2013), the capability of the CAMCo to pre-hydride longer samples was tested. A cladding sample eleven inch long (furnace depth of 12 inches) was run at a control temperature of 280°C for 24 hours in 100% hydrogen, with no surface conditioning preparations, to see how it would behave. Upon opening of the furnace, the sample was discovered to be overly hydrided on one end and sitting in the bottom of the furnace (Figure 4.1). Examination of the tube showed that the end that was hanging from the top of the furnace was the end that was overly hydrided and had fallen off the hanging wire.

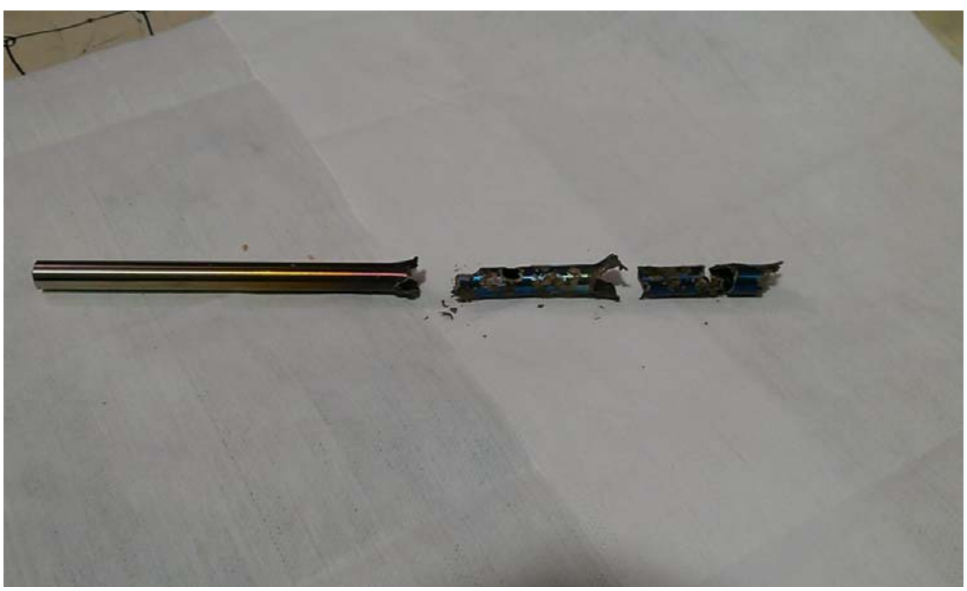


Figure 4.1 Cladding sample (11 inches) hung vertically in CAMCo for 24hr in 100% Hydrogen

The results indicated that a thermal gradient was present in the furnace which could explain the variation in rim thickness seen in previous work. A vertical profile of the furnace was performed while the furnace was running at 300°C with hydrogen, using four thermocouples spanning the height of the furnace (Figure 4.2). While the one middle thermocouple located near the thermocouple used to control the furnace temperature was near 300°C, the other showed a span of almost 40°C difference from the bottom tray where most of the coupons sample work was performed (280°C) to the top of the furnace (320°C). This confirmed that the over hydriding occurring at the top of the furnace was due to temperature variation, and indicated that past temperature results may be off by 20°C.

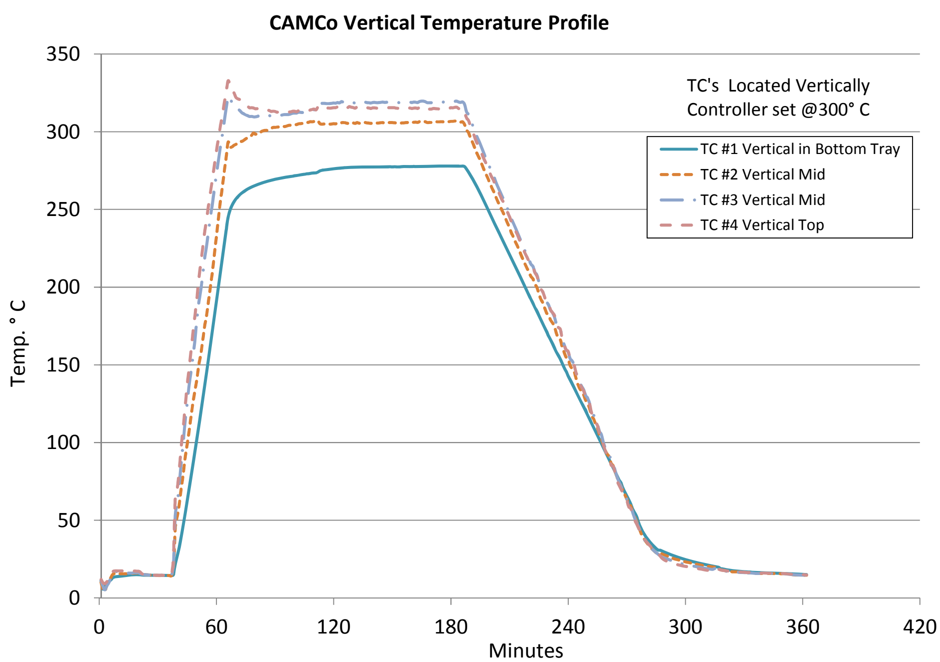


Figure 4.2 Vertical Profile of CAMCo Furnace Running Hydrogen Gas at 300°C

The next temperature profile test performed was to understand how gas flow variation may cause variation in temperature. This time, a profile of the bottom tray was performed while controlling the temperature of the furnace at 300°C while the system was under vacuum and while hydrogen gas was flowing (Figure 4.3). Four thermocouples were placed at each of the four corners of the 6 inch square pan used to hold samples at the bottom of the furnace. While variation between each of the four thermocouples was minimal, the temperature variation between the vacuum and hydrogen gas test was high. While in vacuum, the sample tray was running about 50°C higher than the furnace controller temperature. But when hydrogen at 2 scfm was pumped through the furnace, the sample tray was now 20-30°C lower than the furnace control temperature. Because the gas flows enters the retort from the bottom, it is not too surprising that it is the gas heating up as it flows upward into the retort to the discharge that is causing the source of the temperature gradient.

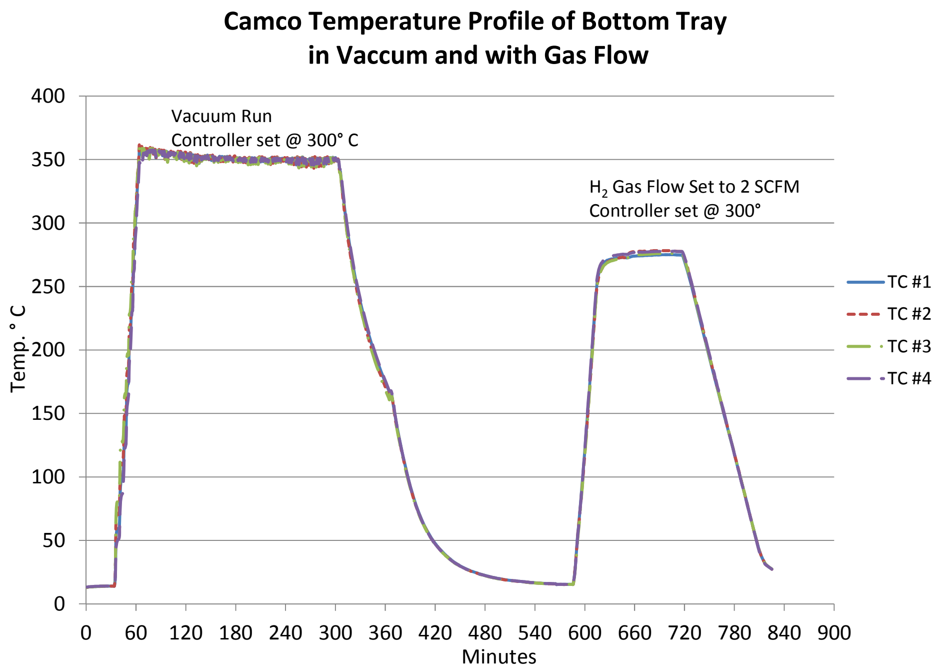


Figure 4.3 Bottom Profile of CAMCo Furnace Running in Vacuum and with Hydrogen Gas at 300°C

4.1.2 Samples in Bottom Tray

To understand how temperatures vary on the bottom tray, two samples were heat treated using a control set point of 300°C and the furnace for a 24 hour period. Two thermocouples were placed on each sample on opposite sides to track the thermal differences. Both samples were blasted on the OD; sample 11 was blasted the day before entering the furnace while sample 12 was blasted within an hour of heat treatment. The results of the thermo-profile of the samples during the run (Figure 4.4) showed that the sample temperature varied between 282°C to 287°C.

The hydrogen analysis results were performed on four radially cut subsamples from the cladding sample at different positions along the length. As discussed in Section 3.2, each ringlet cut was divided into 3-4 smaller sampled for analysis to also examine the difference in the hydrogen concentration circumferentially. The result of the analysis summarized in Table 4.1 showed that significant hydrogen was absorbed at the temperature range shown in Figure 4.4. However, the variation seen axial (between the four subsamples per tube) and circumferentially (relatively high standard deviation compared to the average) was not desirable to achieve longer samples with consistent concentrations.

Optical examination of Samples 11 and 12 are shown in Figure 4.5 and Figure 4.6. While the pictures all show a significant hydride rim forming on all the samples, variation circumferentially is seen that confirms the results seen from the hydrogen analysis. In some cases, regions became overly hydrided producing a hydride blister (Quadrant 1 of Sample 11 in Figure 4.5) which cause radial hydrides to branch out through the tube wall. Such a formation for this program is very undesirable for a final surrogate, so total hydrogen concentrations need to be lower.

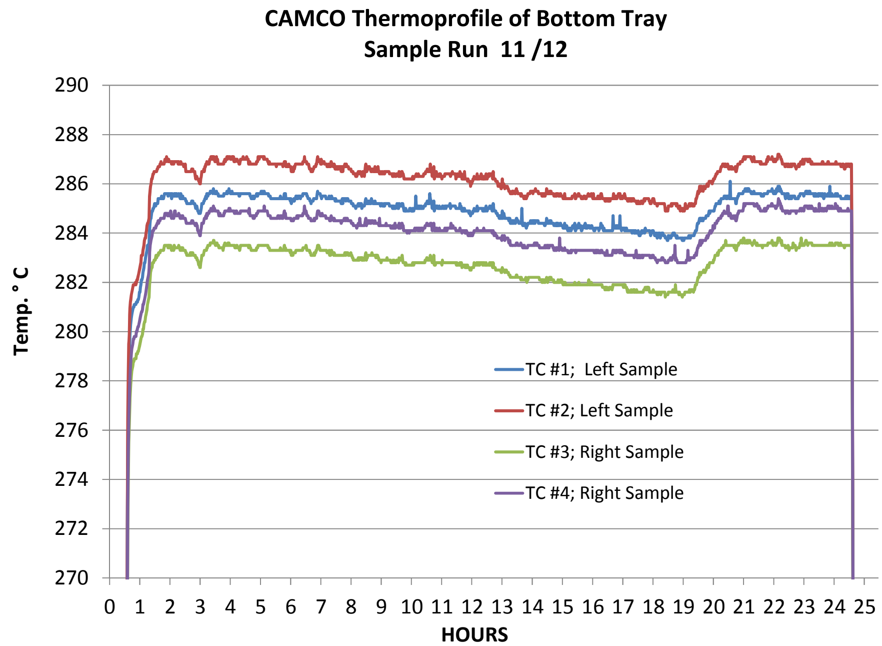
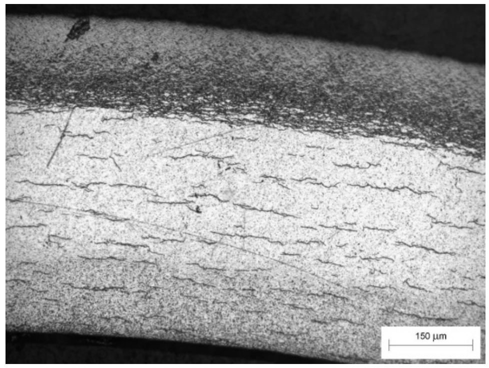


Figure 4.4 Temperature Profile of Samples 11 and 12

Table 4.1 Hydrogen Concentration by Gas Fusion of Samples 11and 12

Sample ID	Subsample Location	Hydrogen Concentration (wppm)		RPD (%)
		Average	Standard Deviation	
11	Top	4267	153	4%
	Middle-Top	2175	222	10%
	Middle-Bot	2225	793	36%
	Bottom	667	172	26%
12	Top	2567	611	24%
	Middle-Top	1480	1050	71%
	Middle-Bot	975	862	88%
	Bottom	1833	306	17%



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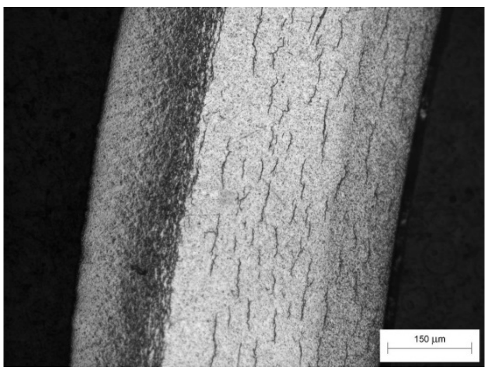

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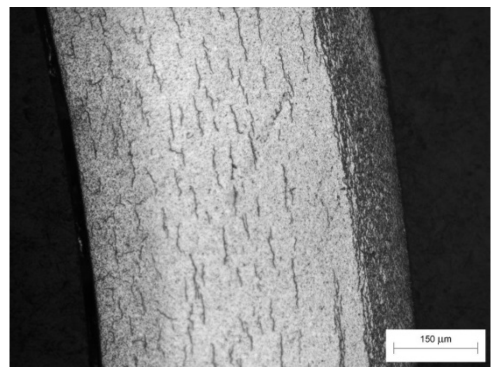

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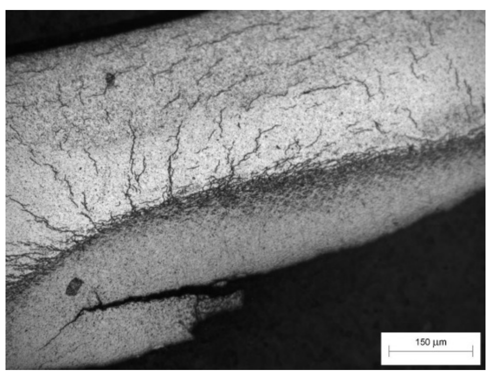

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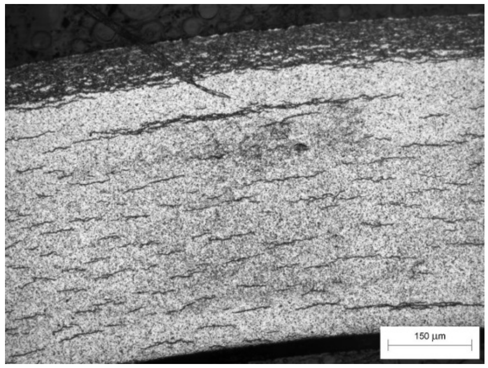
Figure 4.5 Optical Images of Sample 11 at 100x Magnification



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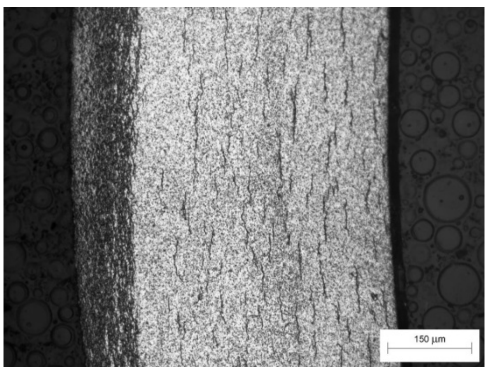

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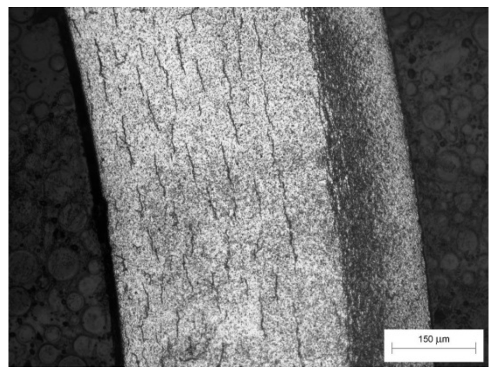

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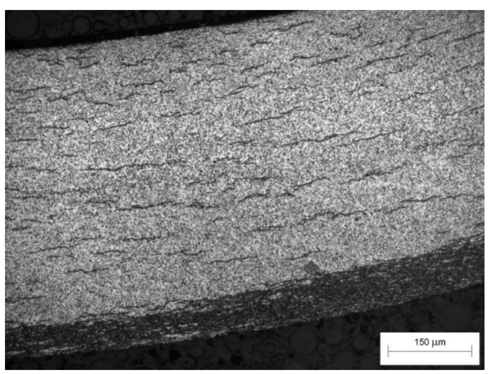


Quadrant 1

Figure 4.6 Optical Images of Sample 12 at 100x Magnification

The conditions for the previous samples were re-run again as Sample 13 and Sample 14 for 24 hour in 100% hydrogen using a furnace set point of 300°C. This time, both samples were run separately to see if the temperature and flow conditions of the furnace were repeatable. Thermocouples were located near each sample to confirm that the same temperatures (between 282°C-287°C) were seen using the furnace control set point of 300°C. Surface conditioning of Sample 13 was matched to Sample 11 and Sample 14 was matched to Sample 12.

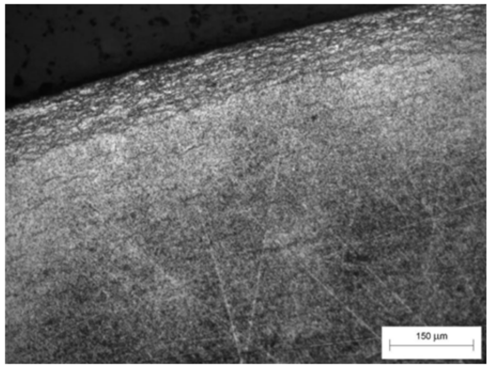
After pre-hydriding each sample, two radially sub-samples were cut from each sample and split into four sections to be analyzed for hydrogen by gas fusion. The results are summarized in Table 4.2. Comparison to the results in Table 4.1 (for Samples 11 and 12) shows that the results are less than half. Also, variability in hydrogen concentration within each sample is still high for both Sample 13 and 14. Although Sample 13 indicates that the sample has low variability circumferentially, the average concentration of the top of the sample is 1.8 times higher than near the bottom. However, Sample 14 show higher variability circumferentially with a higher relative deviations while the difference between the average top and bottom subsamples is only 4%.

Optical examination of Samples 13 and 14 are shown in Figure 4.7 and Figure 4.8. The decrease in rim thickness observed in these samples correlate well with the decrease in the hydrogen concentrations measured. Also, variability visually observed in the rim thickness also correlates well to the relative deviation measured in the hydrogen analysis in Table 4.2, showing it to be a useful tool to delineate variability in the rim thickness.

Overall, the results from this testing indicated that while rim formation was possible as shown in the M2 milestone (Hanson et al. 2013), placing samples at the bottom of the furnace was not an optimal location for this work and could not provide repeatable results.

Table 4.2 Hydrogen Concentration by Gas Fusion of Samples 13 and 14

Sample ID	Subsample Location	Hydrogen Concentration (wppm)		RPD (%)
		Average	Standard Deviation	
13	Middle-Top	815	44	5%
	Middle Bot	458	96	21%
14	Middle-Top	285	107	37%
	Middle Bot	273	69	25%



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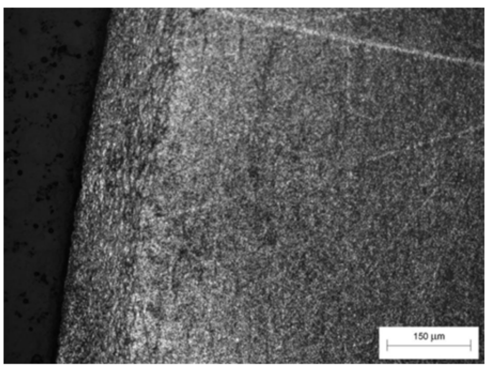

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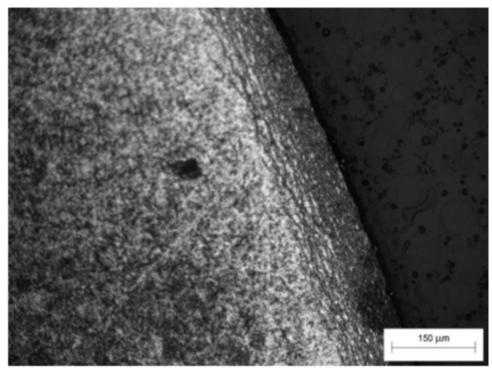

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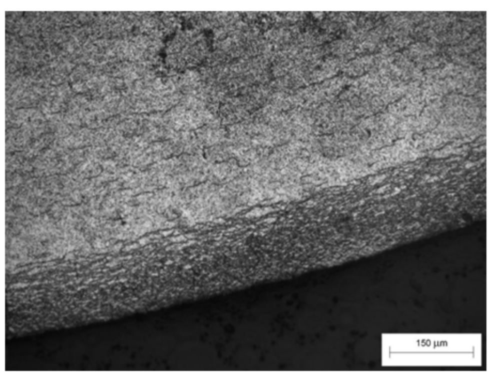
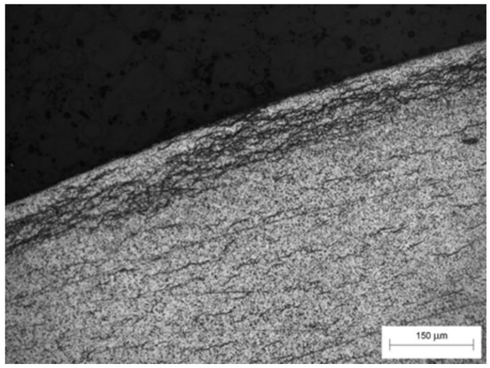


Quadrant 1

Figure 4.7 Optical Images of Sample 13 at 100x Magnification



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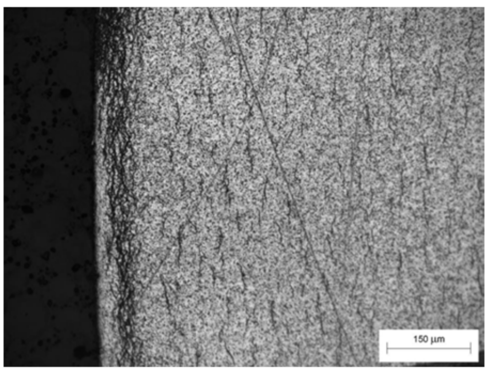

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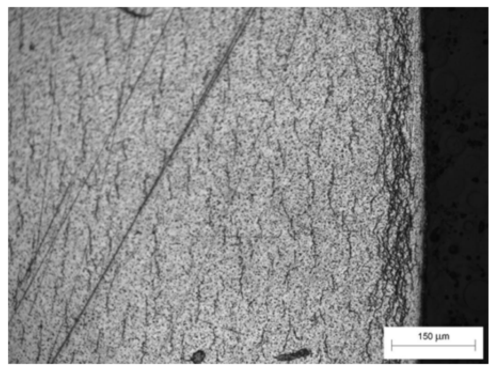

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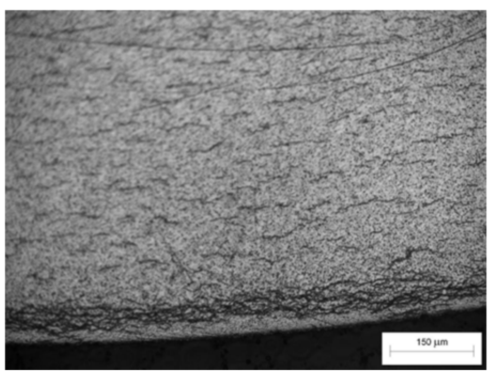


Quadrant 1

Figure 4.8 Optical Images of Sample 14 at 100x Magnification

4.1.3 Hanging Samples

Knowing that the location of the thermocouple used by the furnace controller was 5-6" from the bottom of the furnace, it was proposed that the sample would be hung in the middle of the furnace in the same location. Samples were positioned in the furnace as shown in Figure 4.9 for a series of tests run between 250°C and 300°C.

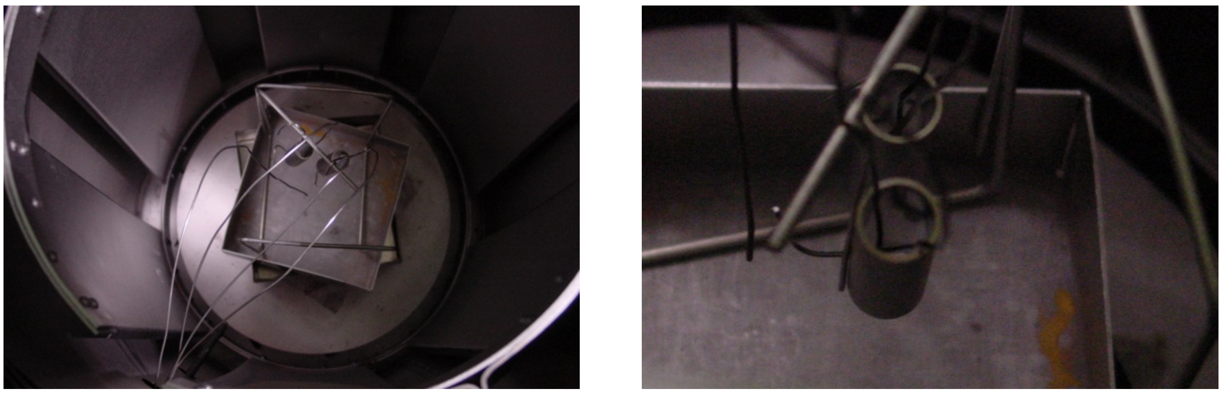


Figure 4.9 Samples 15 and 16 Hanging in CAMCo Furnace 5-6 Inches from Bottom

The first test run was run at a furnace control temperature of 300°C for 24 hours with two samples (Samples 15 and 16) that matched the conditioning of Sample 11 and 12. Four thermocouples were placed near the two samples to measure the temperature variation during the run and see how well it tracked to the thermocouple the controller used. Figure 4.10 shows the temperature measurements during the pre-hydriding of Samples 15 and 16. The new furnace position now showed good agreement with the control temperature setting and that thermal variation in temperature for the samples was ~1°C.

Images of Samples 15 and 16 after pre-hydriding are shown in Figure 4.11. The samples were heavily hydrided with a great deal of loss of mechanical integrity. The increase in temperature and likely gas flow from hanging in the center of the furnace versus sitting at the bottom were the likely cause for the increase in reaction rate. Hydrogen analysis and metallography were not performed.

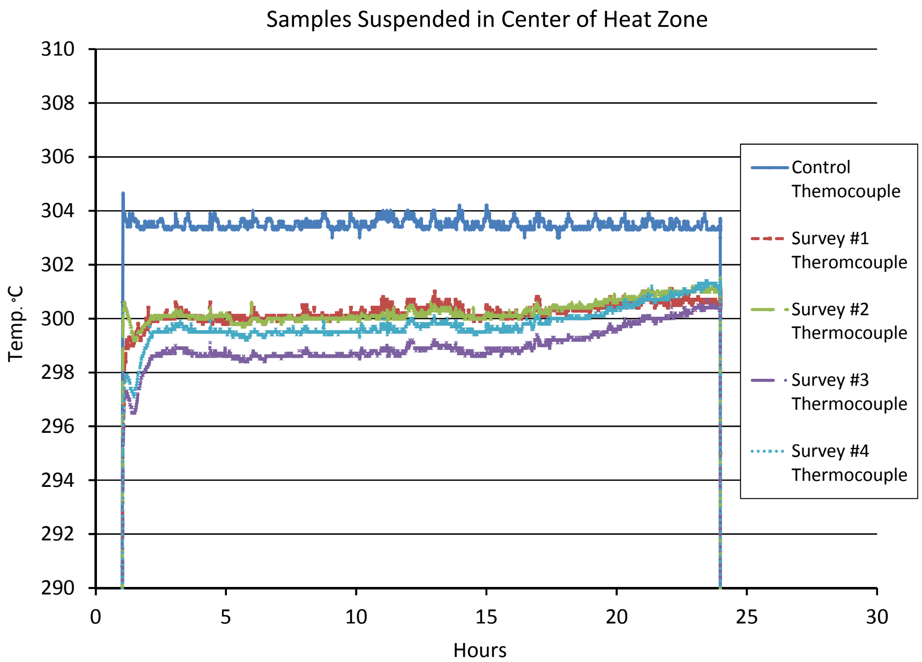


Figure 4.10. Thermo profile of Sample 15 and Sample 16 Run

(a) Sample 15

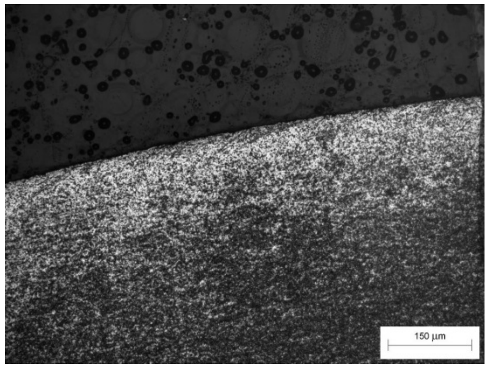
(b) Sample 16

Figure 4.11 Images of Sample 15 and 16 after Pre-hydriding

Samples 17-20 were repeat runs of Sample 15 and 16 run at control temperatures of 250°C (Samples 17 and 18) and 280°C (Samples 19 and 20). Those samples ran in the furnace successfully and were analyzed for hydrogen by gas fusion (Table 4.3) and by optical metallography (Figure 4.12 thru Figure 4.15). Samples 17 and 18 were run at 250°C, so they were not expected to pick up much hydrogen (59-67 wppm) and therefore not expected to see any rim formation in Figure 4.12 and Figure 4.13. Samples 19 and 20 have higher hydrogen concentrations (325-428 wppm) and started to show rim formation as seen in Figure 4.14 and Figure 4.15. While the total hydrogen concentration was not much greater, the rim thickness was greater for Sample 20 versus Sample 19, which was blasted 24 hours earlier that Sample 20. However the circumferential variation in the rim thickness and the hydrogen concentration for both samples was higher than expected.

Table 4.3 Hydrogen Concentration by Gas Fusion of Samples 17 through 20

Sample ID	Subsample Location	Hydrogen Concentration (wppm)		RPD (%)
		Average	Standard Deviation	
17	Middle-Top	67	10	14%
	Middle Bot	63	6	9%
18	Middle-Top	66	8	12%
	Middle Bot	59	7	11%
19	Middle-Top	345	99	29%
	Middle Bot	428	111	26%
20	Middle-Top	403	200	50%
	Middle Bot	355	70	20%



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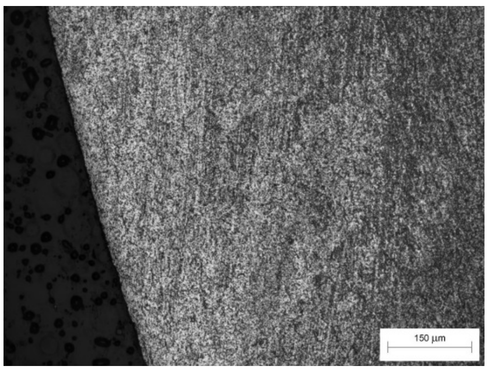

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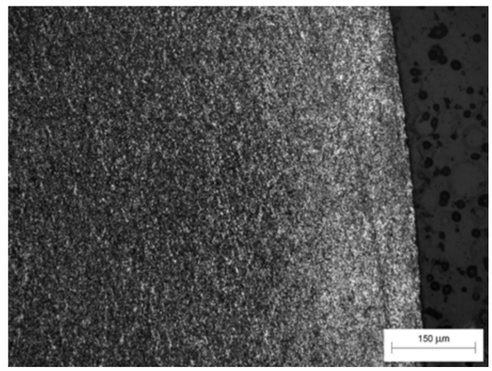

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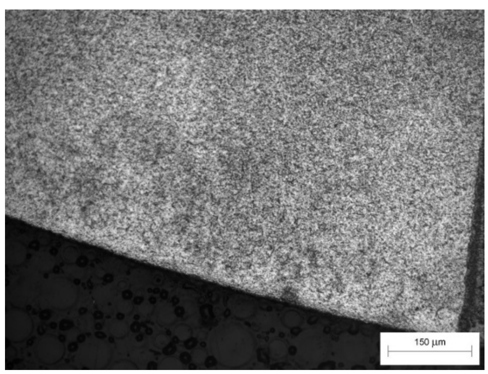
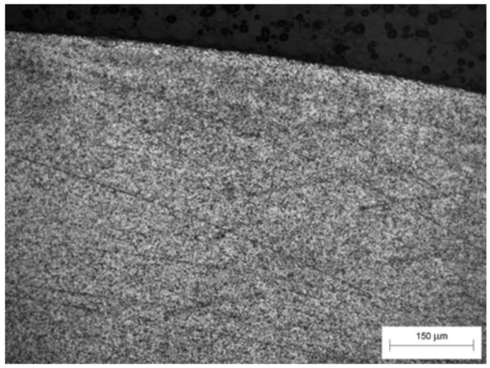


Quadrant 1

Figure 4.12 Optical Images of Sample 17 at 100x Magnification



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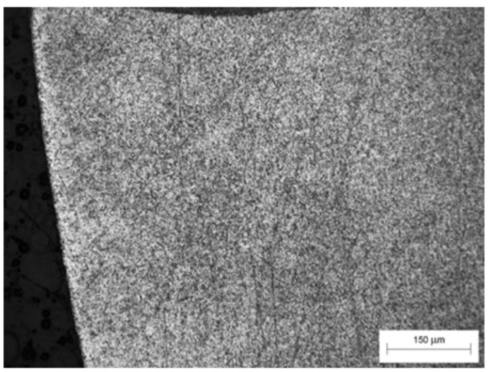

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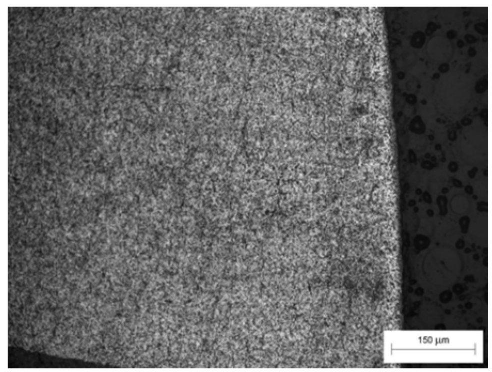

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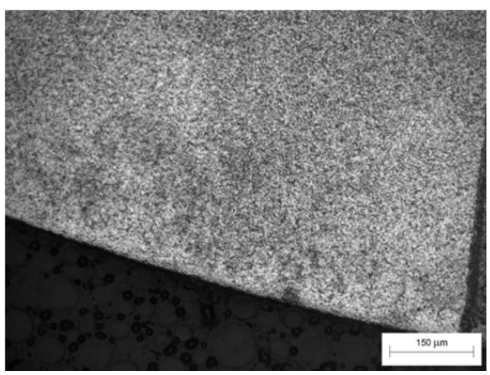
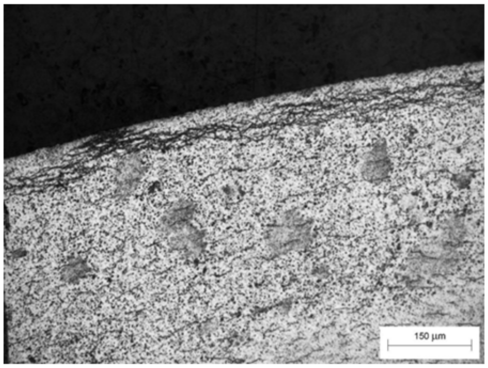


Quadrant 1

Figure 4.13 Optical Images of Sample 18 at 100x Magnification



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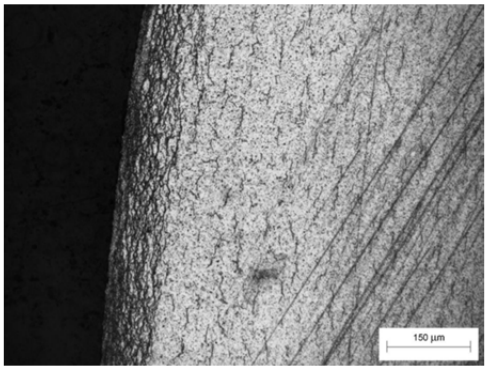

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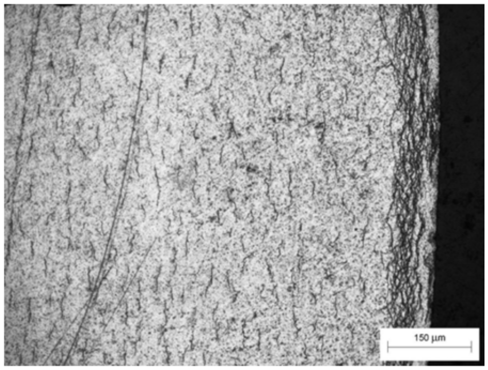

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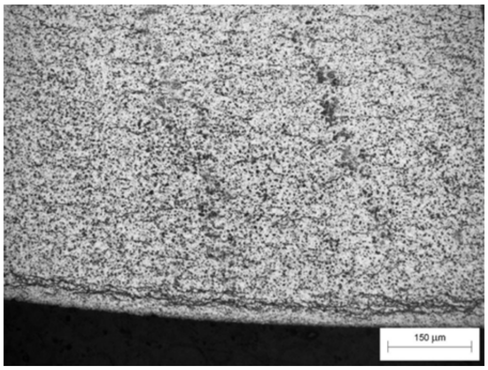
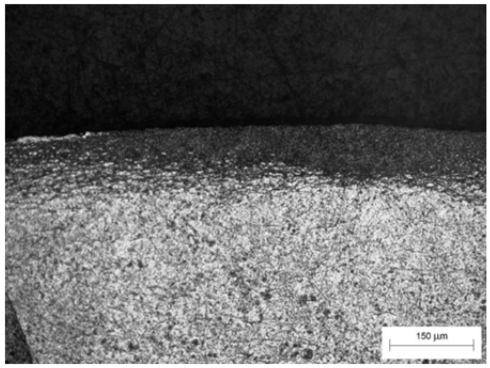


Quadrant 1

Figure 4.14 Optical Images of Sample 19 at 100x Magnification



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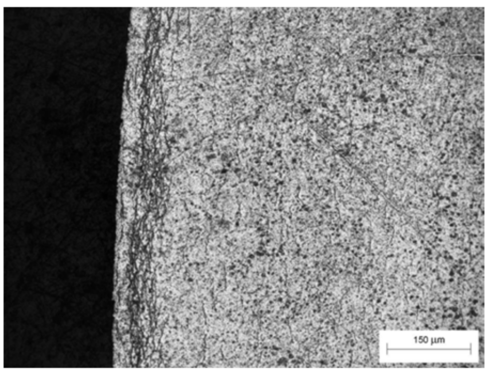

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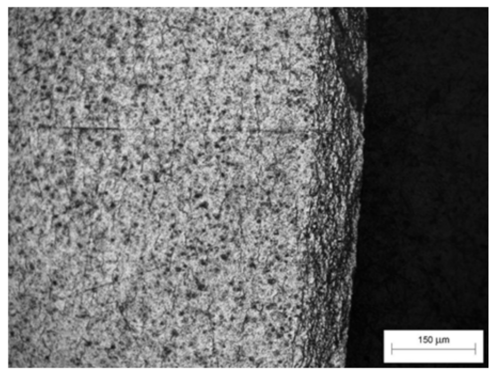

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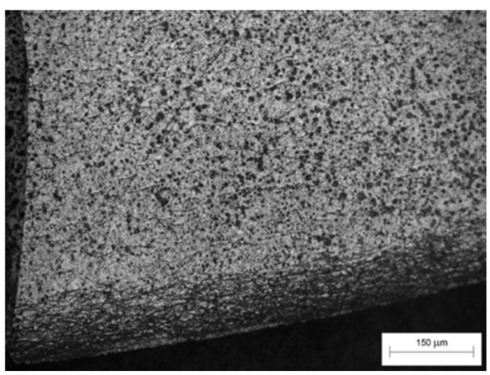


Quadrant 1

Figure 4.15 Optical Images of Sample 20 at 100x Magnification

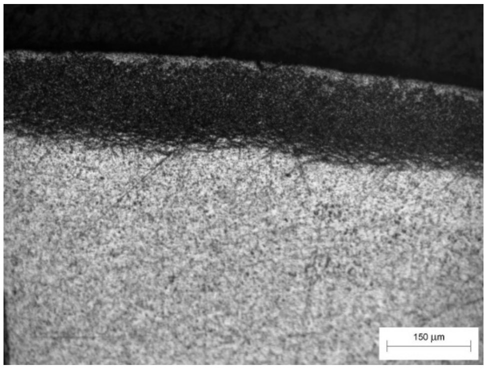
After the CAMCo was used for another project, the furnace was setup to run Samples 21 and 22 at 270°C for 24 hours in hydrogen. Like before, one sample's outer wall was blasted 24 hours prior to pre-hydriding (Sample 21) and the other within an hour of entering the furnace (Sample 22). Gas flows were needed to be re-adjusted to 2 scfm for the run prior to the start of the test and no additional thermo-profiling was performed. Samples were subsampled for hydrogen gas analysis and optical metallography as performed on the previous data set.

The hydrogen concentration results are summarized below in Table 4.4. Examination of the data set shows high variability in the axial and circumferential regions of each sample. More striking is that the average concentrations for these samples are higher than Samples 19 and 20, even though they were run at a temperature 10°C lower. Examination of the optical metallography of Sample 21 (Figure 4.16) and Sample 22 (Figure 4.17) compared to Sample 19 (Figure 4.14) and Sample 20 (Figure 4.15) confirm that the quantity of hydrides present is higher and that the variability in rim thickness observed correlates with the measured hydrogen concentrations.

The large increase in hydrogen adsorption was likely due to the changing of the flow settings on the furnace. Changes in hydrogen gas flow were observed to change the reaction rate in previous testing (Hanson et al. 2013) and have been shown to modify the thermal profile of the furnace significantly. The flow used on the furnace is at the low end of the rotameter used (2 scfm on a 30 scfm) so the variability in the flow rate between the two runs in question is likely very high. Because the furnace is used by several projects, it is not possible to maintain a constant flow setting for a series of tests. For that reason, work with the CAMCo furnace was discontinued after this run.

Table 4.4 Hydrogen Gas Concentration by Gas Fusion for Samples 21 and 22

Sample ID	Subsample Location	Hydrogen Concentration (wppm)		RPD (%)
		Average	Standard Deviation	
21	Middle-Top	1800	510	28%
	Middle Bot	1093	583	53%
22	Middle-Top	583	128	22%
	Middle Bot	1235	489	40%



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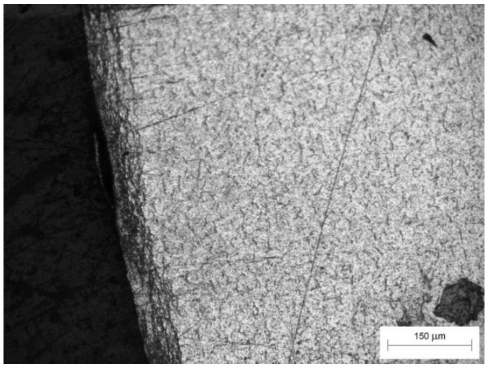

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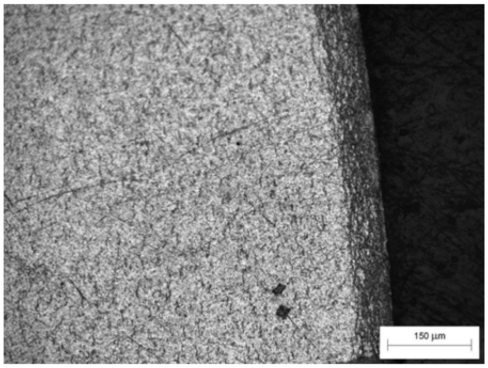

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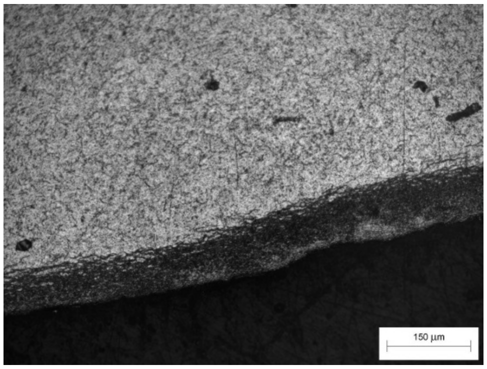
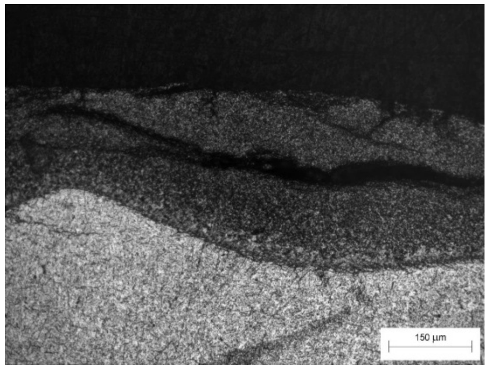


Quadrant 1

Figure 4.16 Optical Images of Sample 21 at 100x Magnification



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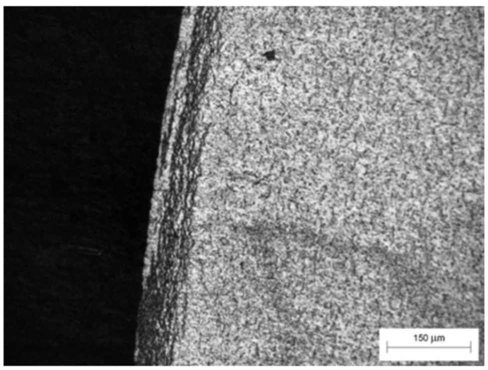

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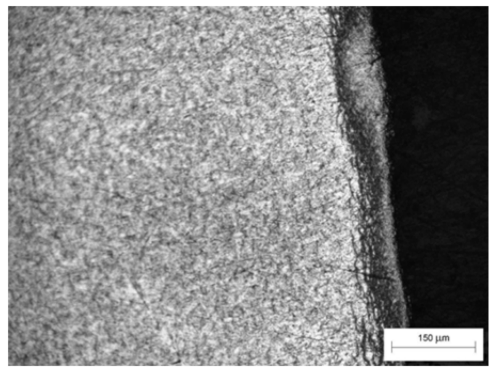

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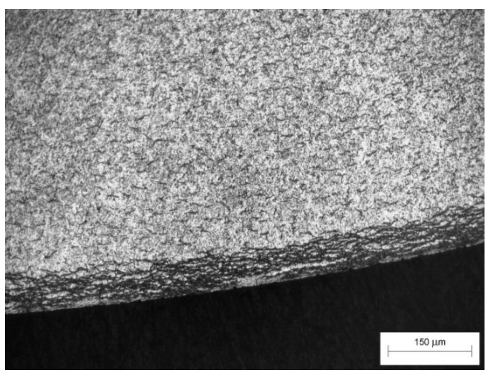


Quadrant 1

Figure 4.17 Optical Images of Sample 22 at 100x Magnification

4.2 Testing in Thermolyne Furnace

Upon completion of testing with Samples 21 and 22, another furnace was selected to complete this study. The Themolyne tube furnace described in Section 3.1 and in Figure 3.2 had many advantages that we were looking for:

- A very controlled heat zone good with flowing gas.
- System used a more appropriately sized gas flow controller.

For the series of tests performed with this furnace, the pre-hydriding heat treatment was:

1) Place samples into chamber and purge with hydrogen gas for 15 minutes with a flow rate of 600 standard cubic centimeters per minute (sccm) at room temperature.
2) Increase the temperature to the test temperature at a rate of 2°C/min while flowing hydrogen at 600 sccm.
3) Hold the samples at the test temperature for 24 hours while flowing hydrogen at 600 sccm.
4) Cool the samples at a rate of 2°C/min while flowing hydrogen until the temperature falls below 40°C.

As with the CAMCO furnace, two samples were run at a time per furnace run. One sample's outer wall was blasted 24 hours prior to pre-hydriding and the other was blasted on the outer wall within an hour of the start of the test. Samples were run at 270°C, 280°C, and 290°C with the samples standing upright in the furnace. While the furnace is a horizontal tube furnace designed for horizontal entries, there was no means of suspending the tube samples horizontally without one spot on the circumferential wall touching the bottom of the furnace. The owner of the furnace uses the system for reduction of flat samples and already had a flat surface plate available to use for the samples and the vertical clearance was available for the 50 mm long sample used for this study. The first samples run with this method (Samples 23 and Sample 24) tipped over during the test and were not analyzed for this study.

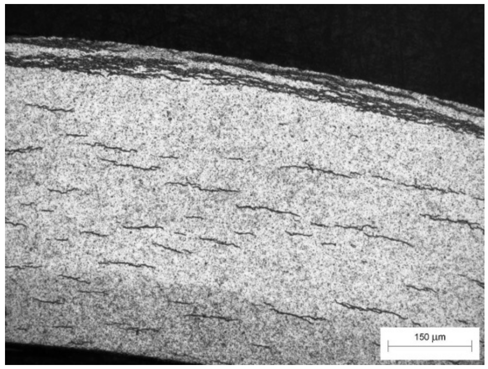
Each set of samples were again radially subsampled for hydrogen analysis by gas fusion in two vertical locations in the samples to see if variation in the axial concentration existed. Also, each ringlet was divided into four sections and measured independently to measure the variation in concentration circumferentially on the sample as well. Subsamples were also taken for optical metallography to visually examine any hydride rim formation visually in the samples.

Hydrogen gas concentration results for three furnace runs are summarized in Table 4.6. Examination of the data shows that variability is lower axially and circumferentially in this furnace versus the CAMCo furnace. Another trend is that circumferential variation (higher RPD values in the average measures) is seen in the samples that are allowed to sit in air for 24 hours after having the outside oxide layer stripped from blasting. This variation is likely do to the oxide layer growing back and doing so unevenly in just air, instead of a water bath, which is typically done in manufacturing. Another trend is that the overall hydrogen concentration appears to increase exponentially with temperature, which was an expected result and shows how sensitive the temperature parameter is for this process.

Examination of Sample 25 (Figure 4.18), Sample 26 (Figure 4.19), Sample 27 (Figure 4.20), Sample 28 (Figure 4.21), Sample 29 (Figure 4.22), and Sample 30 (Figure 4.23) by optical metallography confirms the presence of hydrides at the concentrations indicated by the gas concentration measurements. The slower cooling rate used for the Thermolyne tests (2°C/min versus the >10°C/min rate used for the recent CAMCo runs) caused the hydrides present in the interior of the cladding to elongate to more prototypic lengths seen in irradiated cladding. The optical images confirmed that the samples that were blasted the day before had more variation in the rim thickness. This was especially evident between Sample 29 (Figure 4.22) and Sample 30 (Figure 4.23). Sample 30 (blasted immediately before pre-hydriding) showed the most consistent rim thickness out of all the samples performed and had the lowest amount of variation found in its hydrogen measurements. While Sample 29 also showed a significant rim formation, its average measured rim thickness was 100 microns versus Sample 30 with an average thickness of 120 microns.

Table 4.5 Hydrogen Gas Concentration by Gas Fusion for Samples 25 through 30

Sample ID	Test Conditions	Subsample Location	Hydrogen Concentration (wppm)		RPD (%)
			Average	Standard Deviation	
25	270°C Blast-24HR	Middle-Top	200	82	41%
		Middle Bot	223	43	19%
26	270°C Blast	Middle-Top	154	71	46%
		Middle Bot	165	37	22%
27	280°C Blast-24HR	Middle-Top	363	33	9%
		Middle Bot	345	52	15%
28	280°C Blast	Middle-Top	343	29	8%
		Middle Bot	223	44	20%
29	290°C Blast-24HR	Middle-Top	1100	294	27%
		Middle Bot	1063	222	21%
30	290°C Blast	Middle-Top	1175	150	13%
		Middle Bot	1008	107	11%



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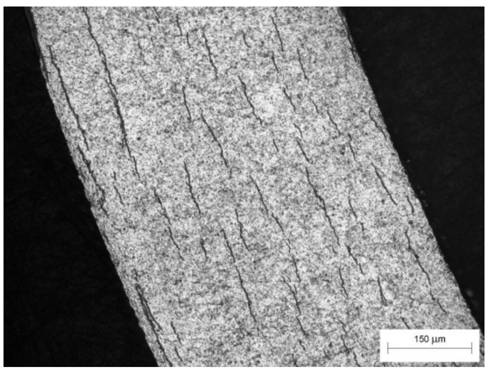

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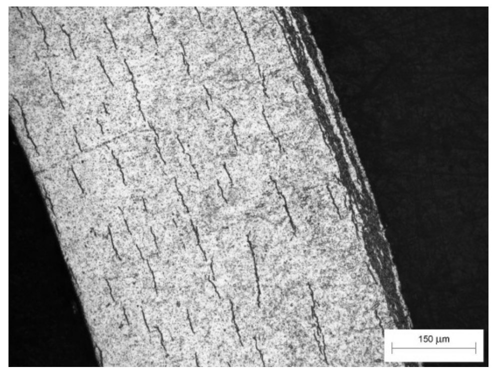
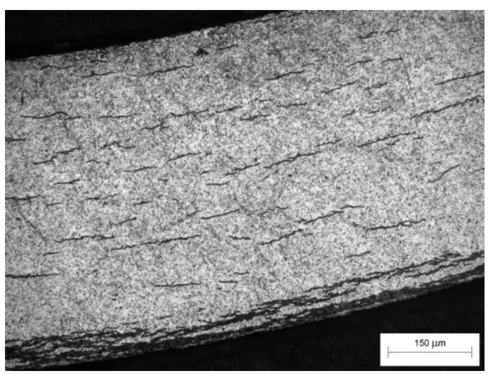
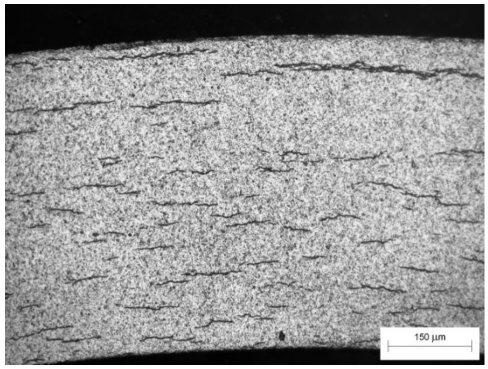


Quadrant 2

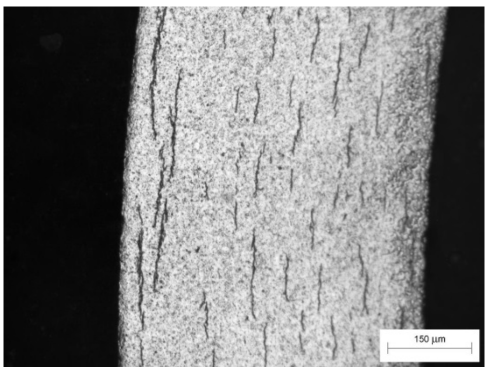


Quadrant 1

Figure 4.18 Optical Images of Sample 25 at 100x Magnification



Quadrant 3



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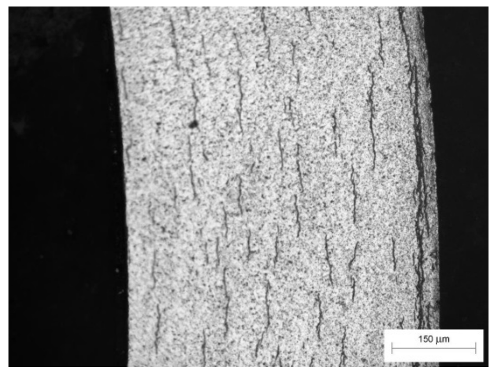

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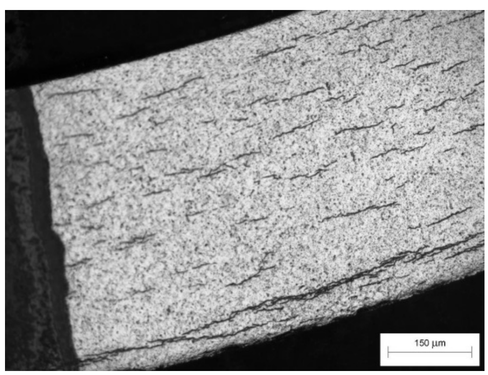
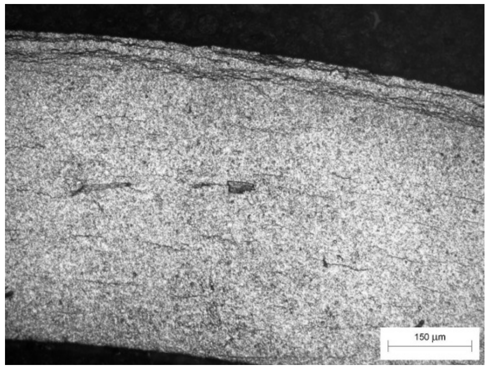


Quadrant 1

Figure 4.19 Optical Images of Sample 26 at 100x Magnification



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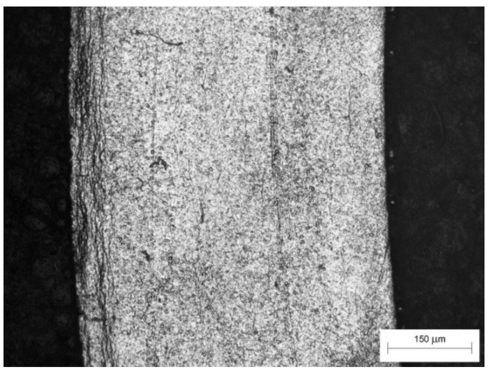

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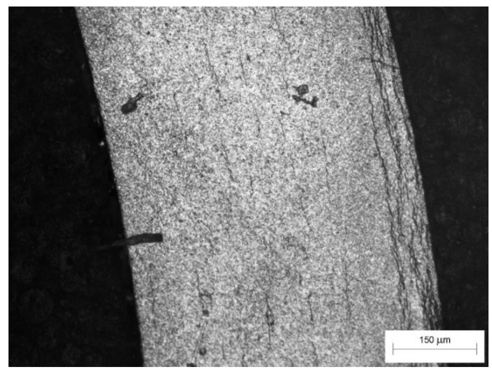

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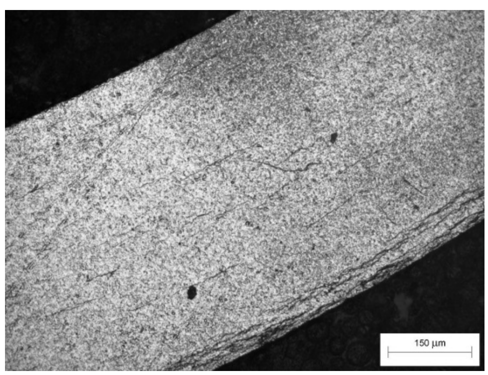
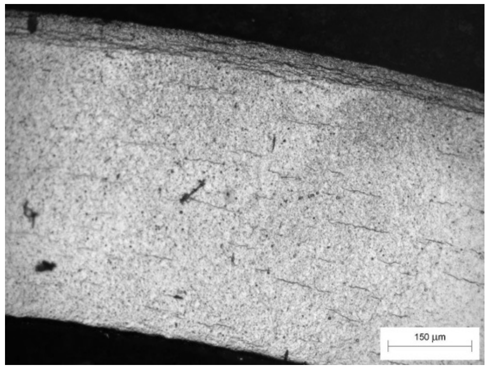


Quadrant 1

Figure 4.20 Optical Images of Sample 27 at 100x Magnification



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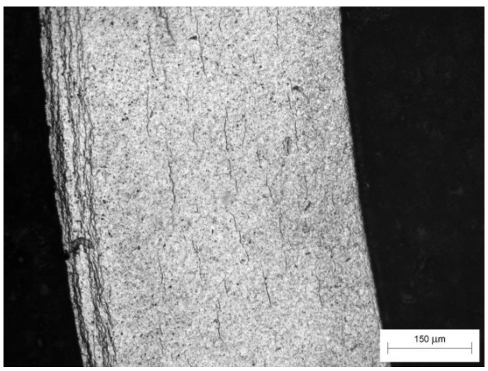

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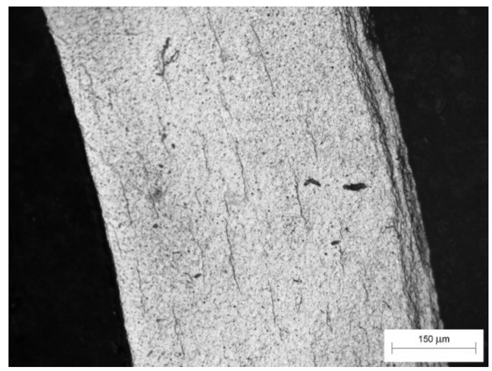

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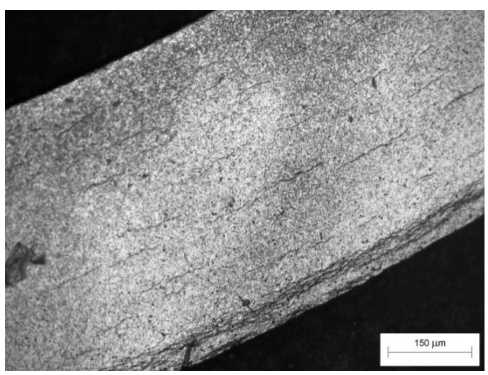
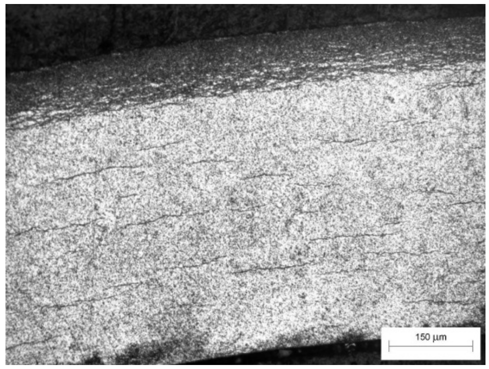


Quadrant 1

Figure 4.21 Optical Images of Sample 28 at 100x Magnification



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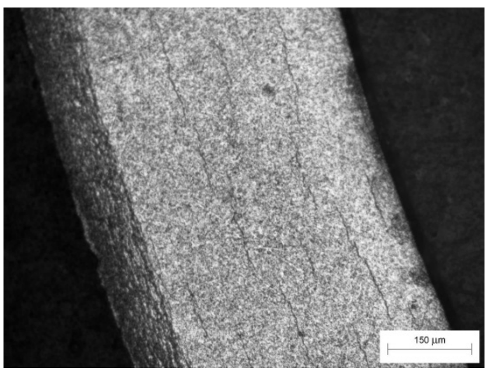

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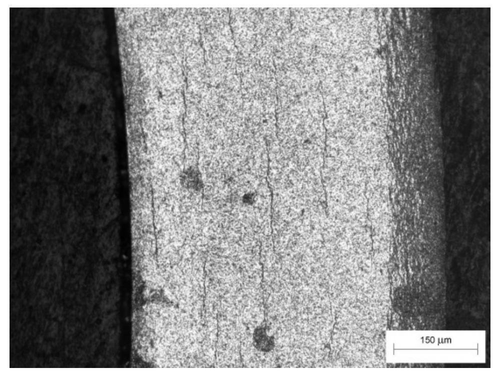
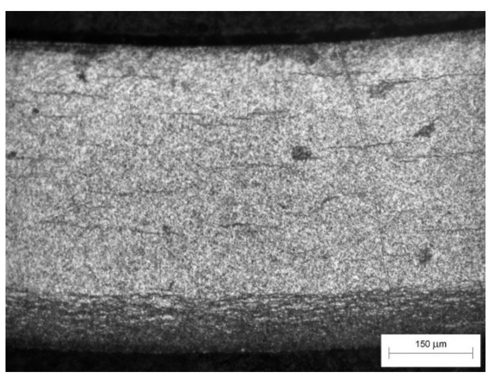
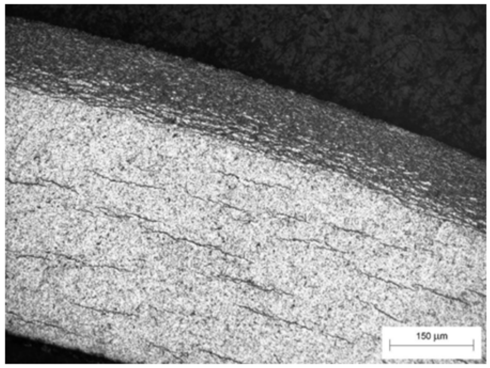


Quadrant 2



Quadrant 1

Figure 4.22 Optical Images of Sample 29 at 100x Magnification



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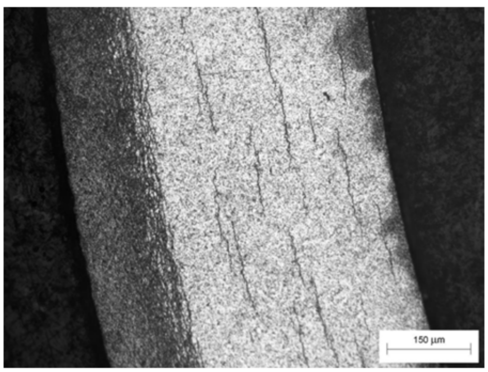

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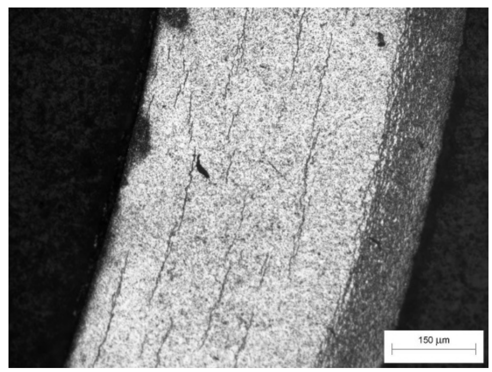

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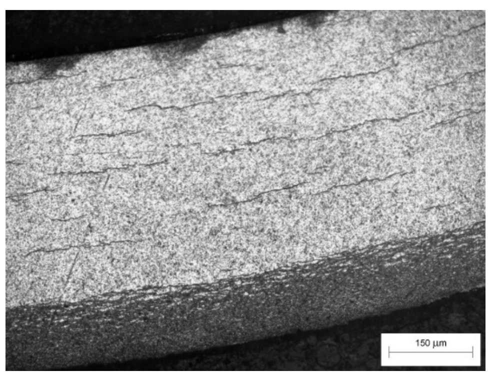


Quadrant 1

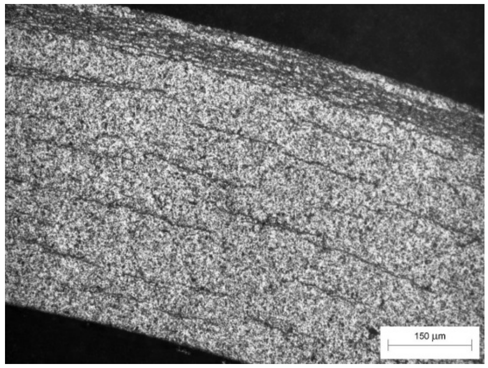
Figure 4.23 Optical Images of Sample 30 at 100x Magnification

Upon the completion of the 290°C runs, another run was performed with two samples laying horizontal on the base of the furnace holder to confirm concerns that doing so would cause variation circumferentially on the clad samples. The temperature was elevated to 300°C as well to see how increases in temperature could counter changes in gas flow.

The hydrogen analysis results in Table 4.6 show high circumferentially variation as expected with one side of the outer wall being in contact with the furnace holder surface. Examination by optical metallography for Sample 30 (Figure 4.24) and Sample 31 (Figure 4.25) confirm this showing a thick hydride rim on one side measuring close to 120 microns and decreasing in size to about 70 microns on the opposite side. In the future, design of sample holders need to consider how to minimize this impact and allow gas to flow evenly along the entire circumference of longer cladding samples that would need to be placed in such a furnace horizontally.

Table 4.6 Hydrogen Gas Concentration by Gas Fusion for Samples 31 through 32

Sample ID	Test Conditions	Subsample Location	Hydrogen Concentration (wppm)		RPD (%)
			Average	Standard Deviation	
31	300°C; Flat Blast-24HR	Middle-Top	690	141	20%
		Middle Bot	768	296	39%
32	300°C; Flat Blast	Middle-Top	750	297	40%
		Middle Bot	863	288	33%



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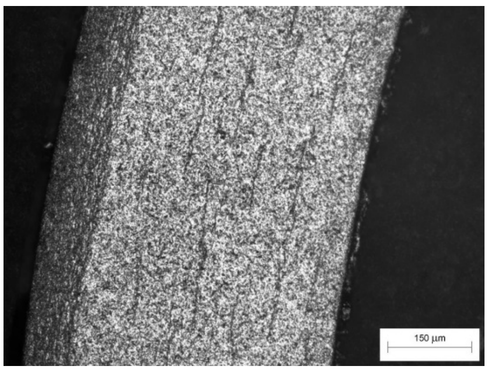

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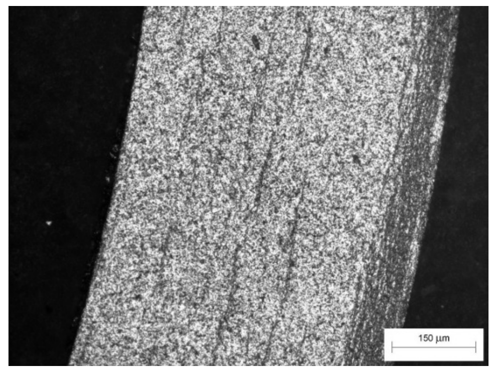

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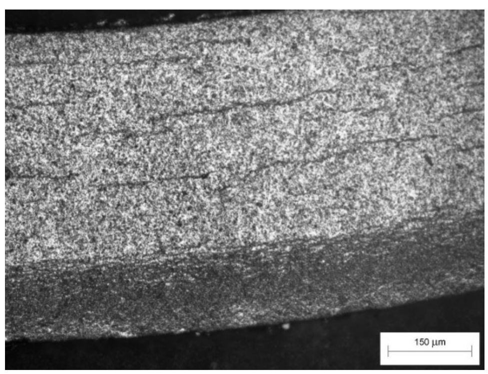
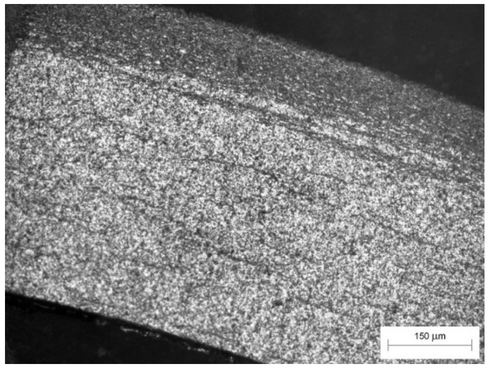


Quadrant 1

Figure 4.24 Optical Images of Sample 31 at 100x Magnification



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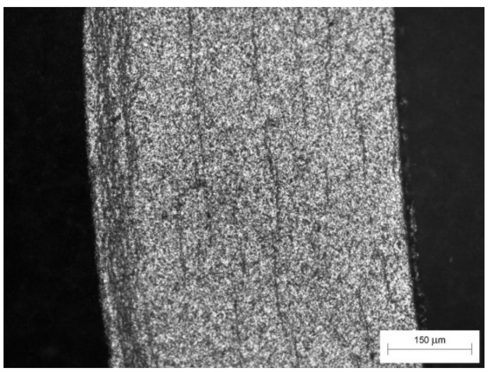

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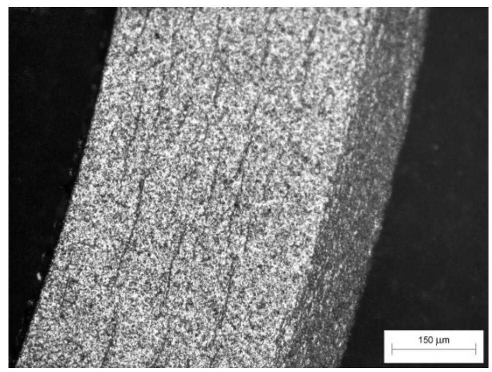

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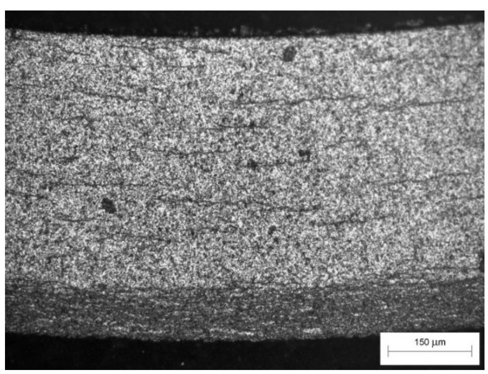


Quadrant 1

Figure 4.25 Optical Images of Sample 32 at 100x Magnification

5. LESSONS LEARNED AND FUTURE WORK

The purpose of this work was to optimize the successful techniques developed by Hanson et al. (2013) for the M2 milestone to pre-hydriding unirradiated Zircaloy cladding such that a high concentration, or rim, of hydrides is formed at the cladding outside diameter as is found in cladding for high burnup fuels. The worked confirmed that initial results to produce a significant hydride rim on unirradiated Zircaloy cladding using the methods developed could be done in a consistent manner. The results indicate the following:

- Optimal pre-hydriding occurs between 280°C-300°C in pure H_2 using the surface conditioning methods used, depending on the flow conditions.
- Temperature and gas flow controls are critical and that variations in either of these process parameters along the circumference or length of tube samples will cause variation in the rim thickness and in the total hydrogen adsorbed.
- Time of conditioning the outside diameter is not as significant as temperature and gas flow, but the length of time between conditioning of the outer wall and the time it enters the furnace appears to increase variability in the absorbance rate. However, this may be due to the finishing technique used.

Work to be pursued in the future:

- Kinetics and effects of variable H_2 gas compositions (i.e., < 100%) for pre-hydriding
- Better control of clad surface conditions either through
  - More controlled surface blasting
  - Rotary grinding either by wet or dry methods
  - Treatment in HF to thoroughly remove the oxide layer
- Testing of cladding alloys other than just Zr-4 to determine the effect of chemical composition as well as texture on the ability to form the hydride rim
- Hydrogen redistribution testing of pre-hydrided unirradiated samples
- Mechanical testing (burst, tensile, contractile strain ratio) of pre-hydrided unirradiated samples

Once a reliable and repeatable method for producing pre-hydrided cladding with the desired total hydrogen concentration and hydride rim thickness in 6”-12” samples is perfected, testing to determine how radial hydrides form and their impact on mechanical properties will be performed. The focus in FY14 will be examining the effects of rewetting and multiple drying cycles on the hydride and mechanical property behavior.

6. REFERENCES

10 CFR Part 72. Licensing Requirements for the Independent Storage of Spent Nuclear Fuel and High-Level Radioactive Waste. U.S. Nuclear Regulatory Commission, Washington, D.C.

ASTM B811-02. 2007. *Standard Specification for Wrought Zirconium Alloy Seamless Tubes for Nuclear Reactor Fuel Cladding*. ASTM International, West Conshohocken, Pennsylvania.

Billone M, Y Yan, T Burtseva, and R Daum. 2008. *Cladding Embrittlement during Postulated Loss-of-Coolant Accidents*. NUREG/CR-6967, U.S. Nuclear Regulatory Commission, Washington, D.C. ML082130389 at http://www.nrc.gov/reading-rm/adams.html.

Billone MC, TA Burtseva, and RE Einziger. 2013. "Ductile-to-brittle transition temperatures for high-burnup cladding alloys exposed to simulated drying-storage conditions." *Journal of Nuclear Materials* 433:431-448.

Hanson B, H Alsaed, C Stockman, D Enos, R Meyer, and K Sorenson. 2012a. *Gap Analysis to Support Extended Storage of Used Nuclear Fuel*. FCRD-USED-2011-000136 Rev. 0, PNNL-20509, July 31, 2012. Prepared for the U.S. Department of Energy Used Fuel Disposition Campaign, Washington, D.C.

Hanson B, H Alsaed, and C Stockman. 2012b. *Used Nuclear Fuel Storage and Transportation Data Gap Prioritization.* FCRD-USED-2012-000109 Draft, PNNL-21360, April 30, 2012. Prepared for the U.S. Department of Energy Used Fuel Disposition Campaign, Washington, D.C.

Hanson B, R Shimskey, C Lavender, P MacFarlan, P Eslinger. 2013. *Hydride Rim Formation in Unirradiated Zircaloy*. FCRD-USED-2012-000151. PNNL-22438, April 30, 2013. Prepared for the U.S. Department of Energy Used Fuel Disposition Campaign, Washington, D.C.

NRC. 2010. *Standard Review Plan for Spent Fuel Dry Storage Systems at a General License Facility*. NUREG-1536, Rev. 1, U.S. Nuclear Regulatory Commission, Washington, D.C.

Schemel J. H. 1989. *Zirconium Alloy Fuel Clad Tubing Engineering Guide*. Sandvik Special Metals Coroporation, Kennewick, WA.

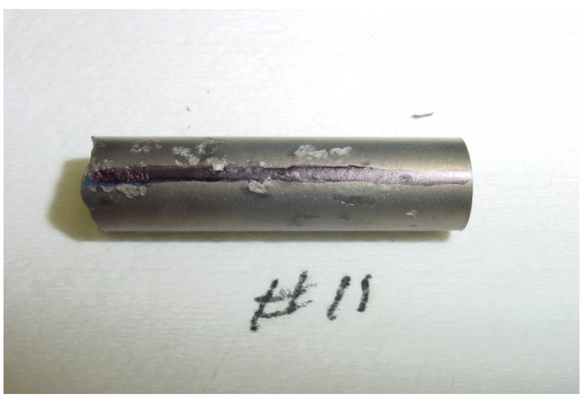
APPENDIX: SAMPLE TEST RESULTS

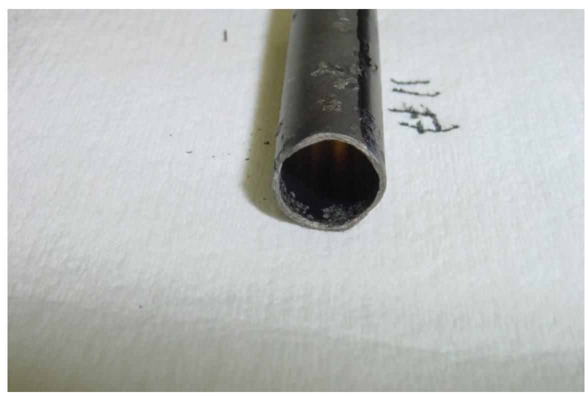
Sample ID: 11

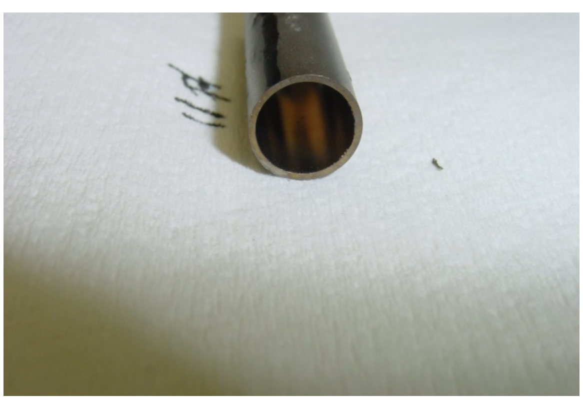
Furnace: CAMCo
Location: Bottom Tray
Temperature: 300°C Control Temperature (Actual Between 282-287°C)
OD Surface: Blasted, 24 Hr Hold in Air
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

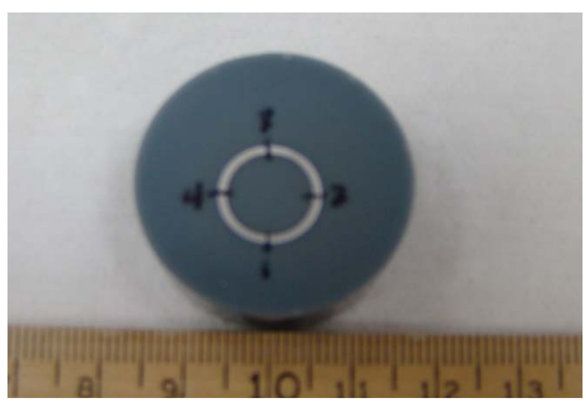
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Top	4100	4300	4400	NA	4267	153	4%
Middle-Top	2500	2100	2000	2100	2175	222	10%
Middle-Bot	2900	2800	2000	1200	2225	793	36%
Bottom	610	860	530	NA	667	172	26%

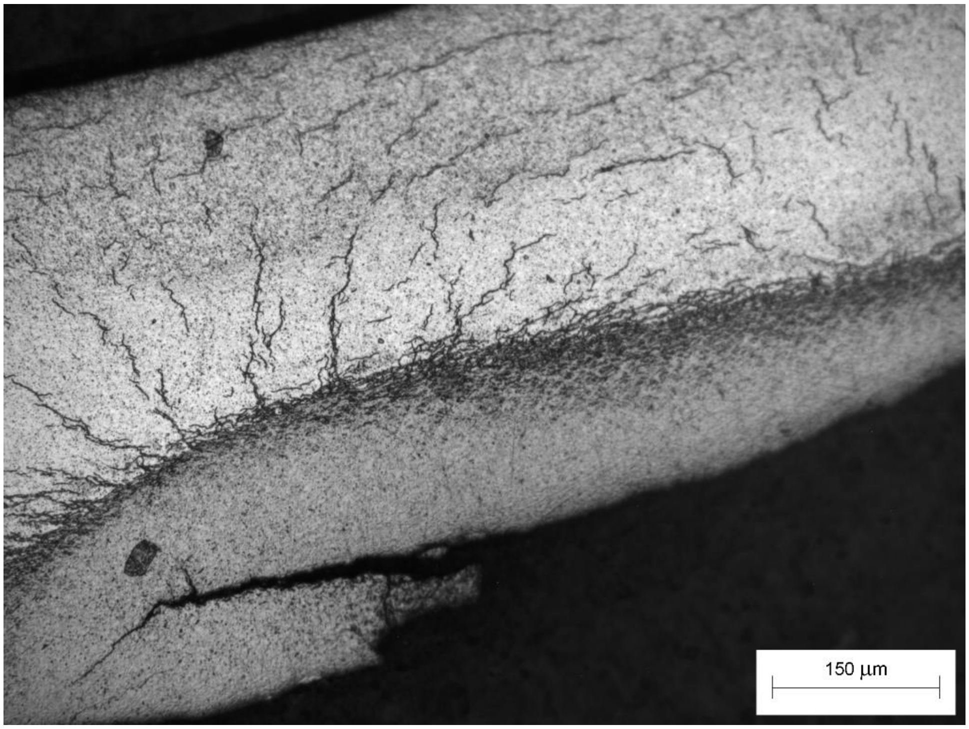




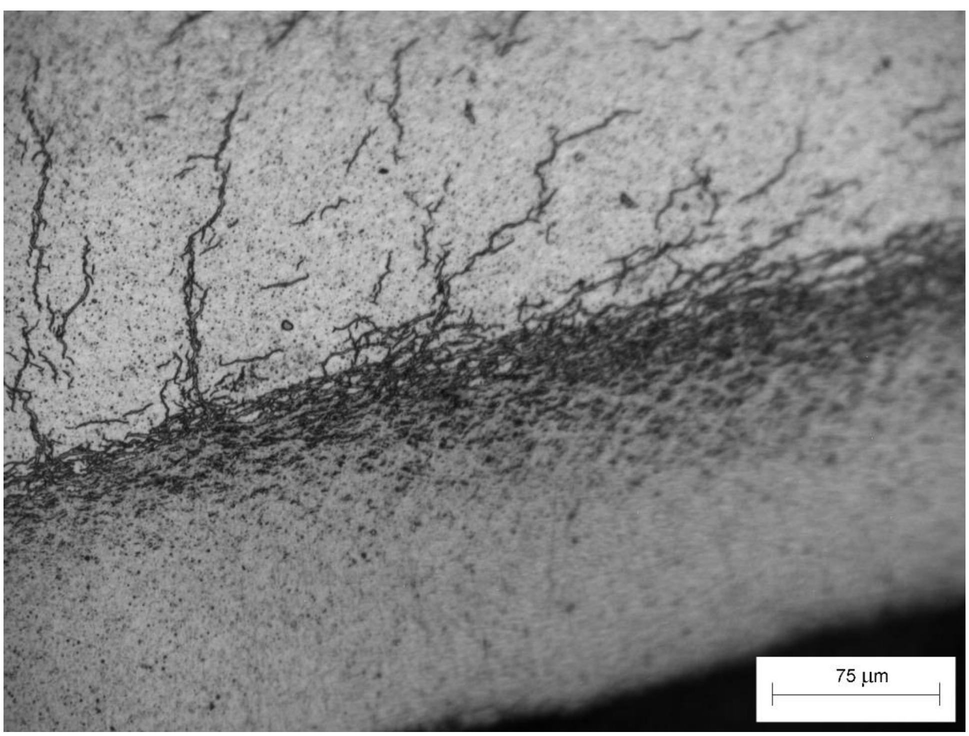




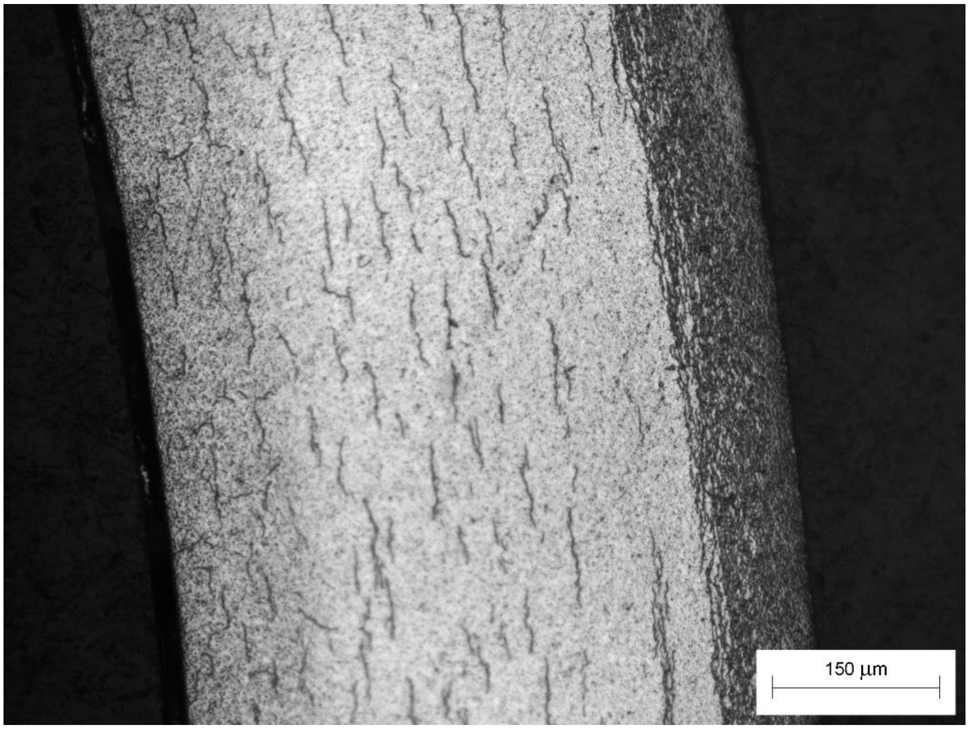
Images of Sample 11 After Hydriding and Mounted in Epoxy



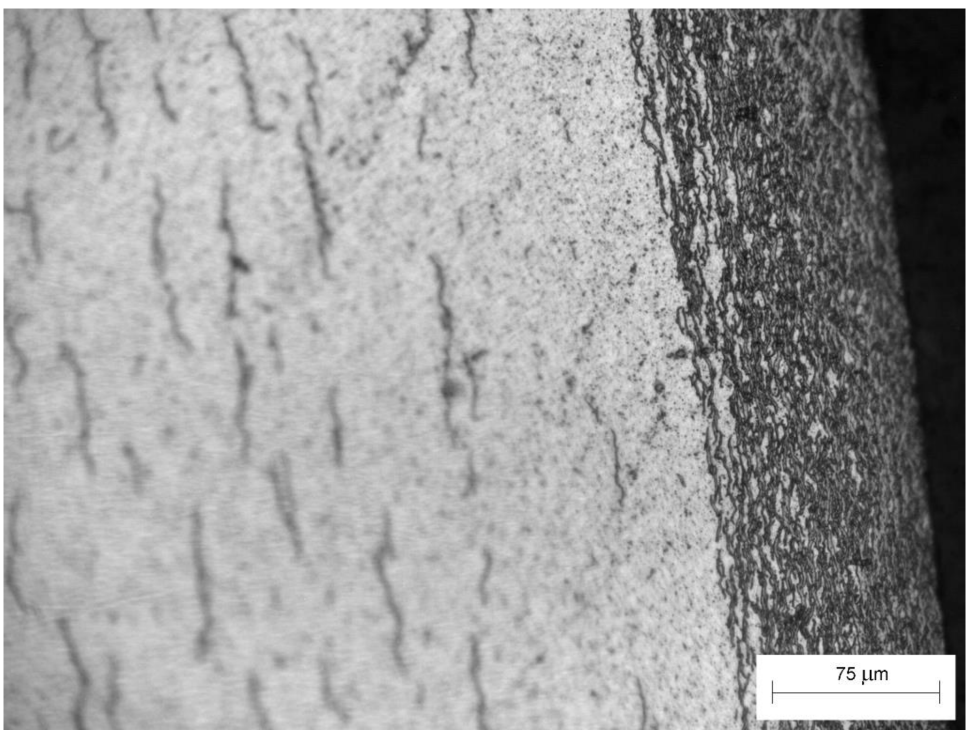
Sample 11 / Quadrant 1 / 100x Magnification



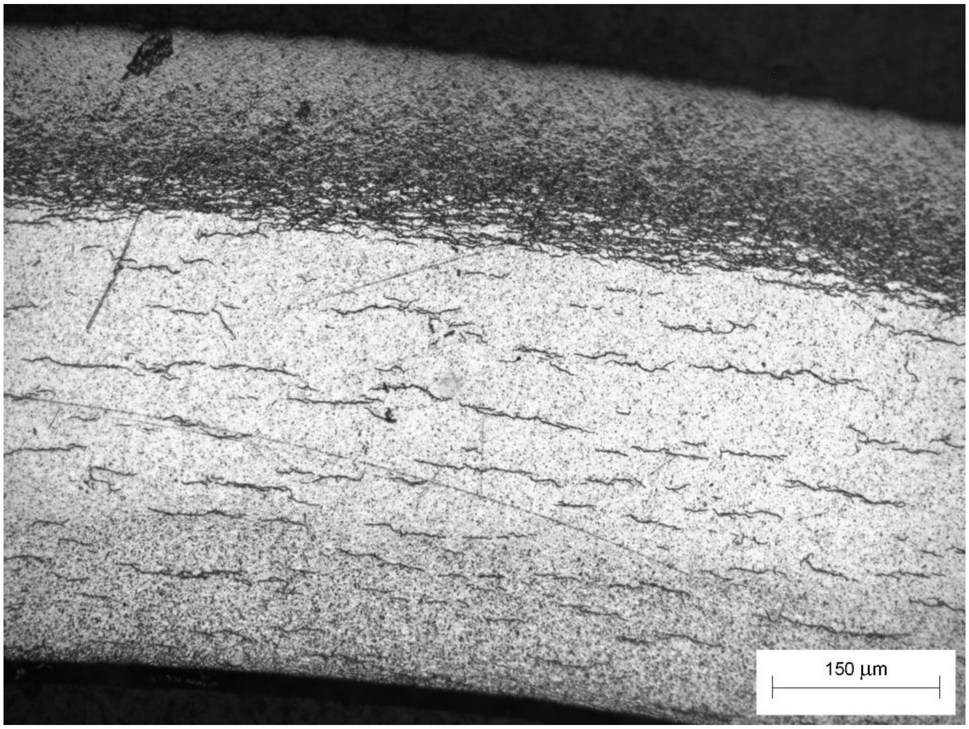
Sample 11 / Quadrant 1 / 200x Magnification



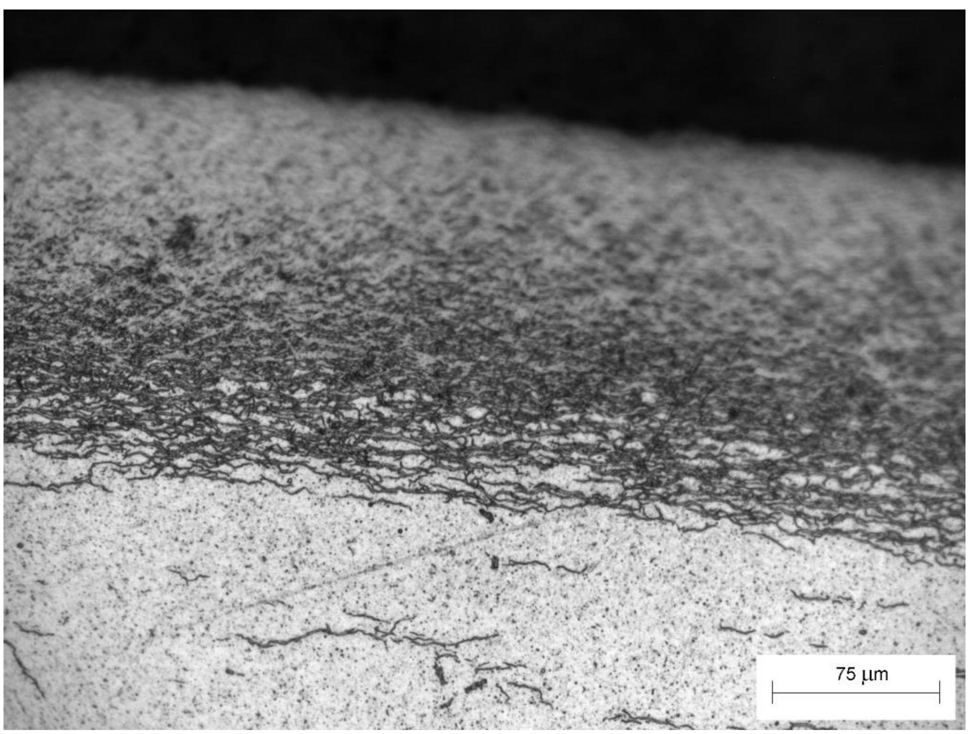
Sample 11 / Quadrant 2 / 100x Magnification



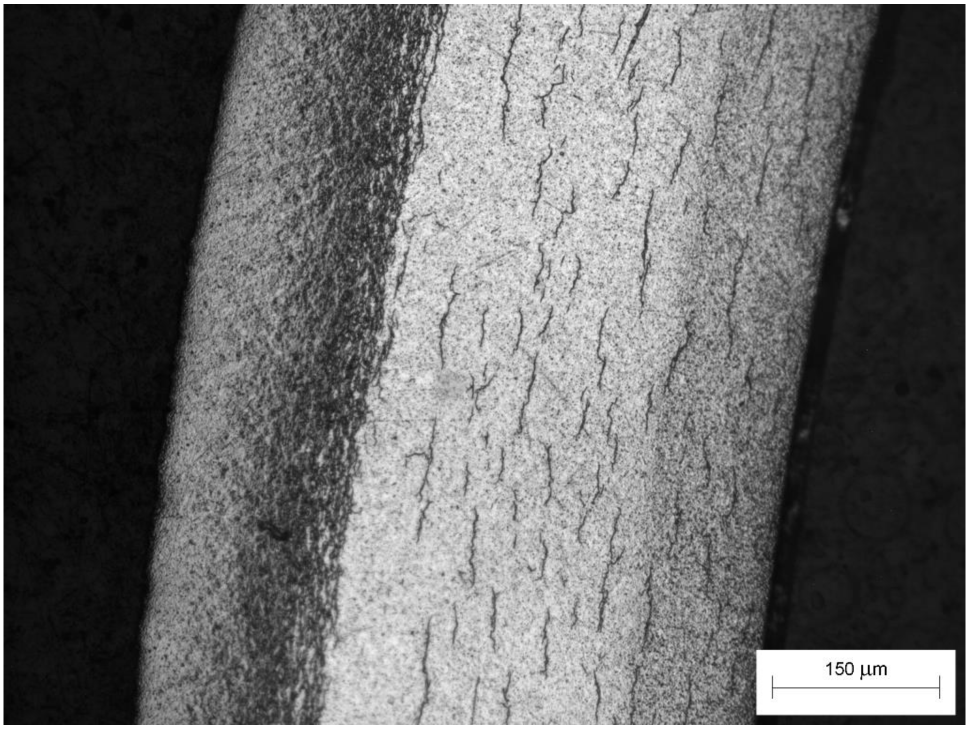
Sample 11 / Quadrant 2 / 200x Magnification



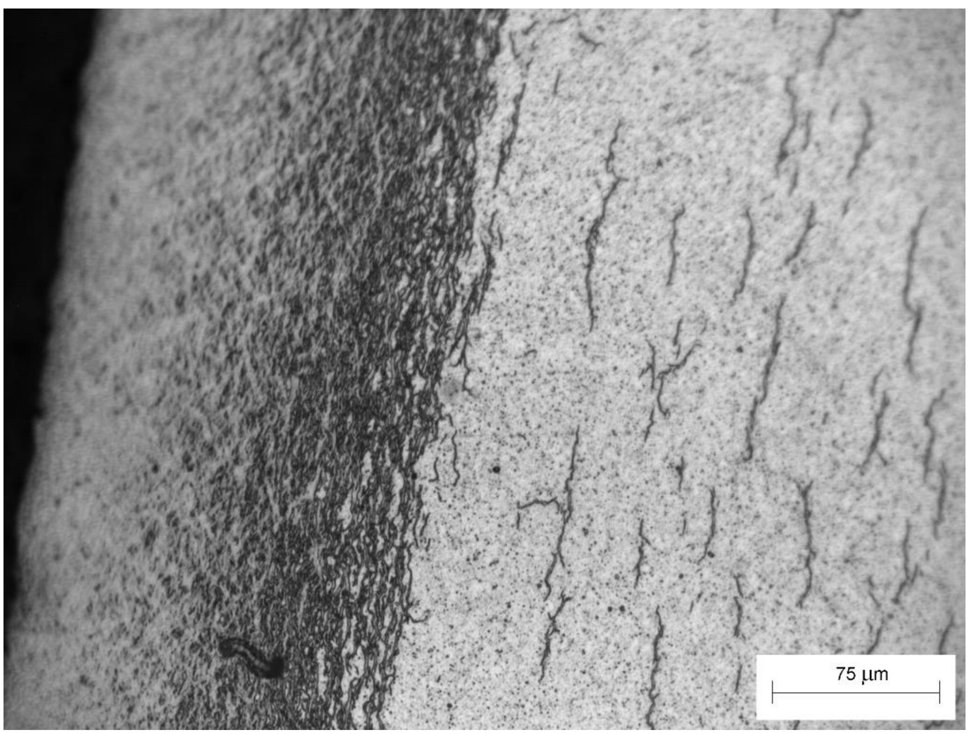
Sample 11 / Quadrant 3 / 100x Magnification



Sample 11 / Quadrant 3 / 200x Magnification



Sample 11 / Quadrant 4 / 100x Magnification



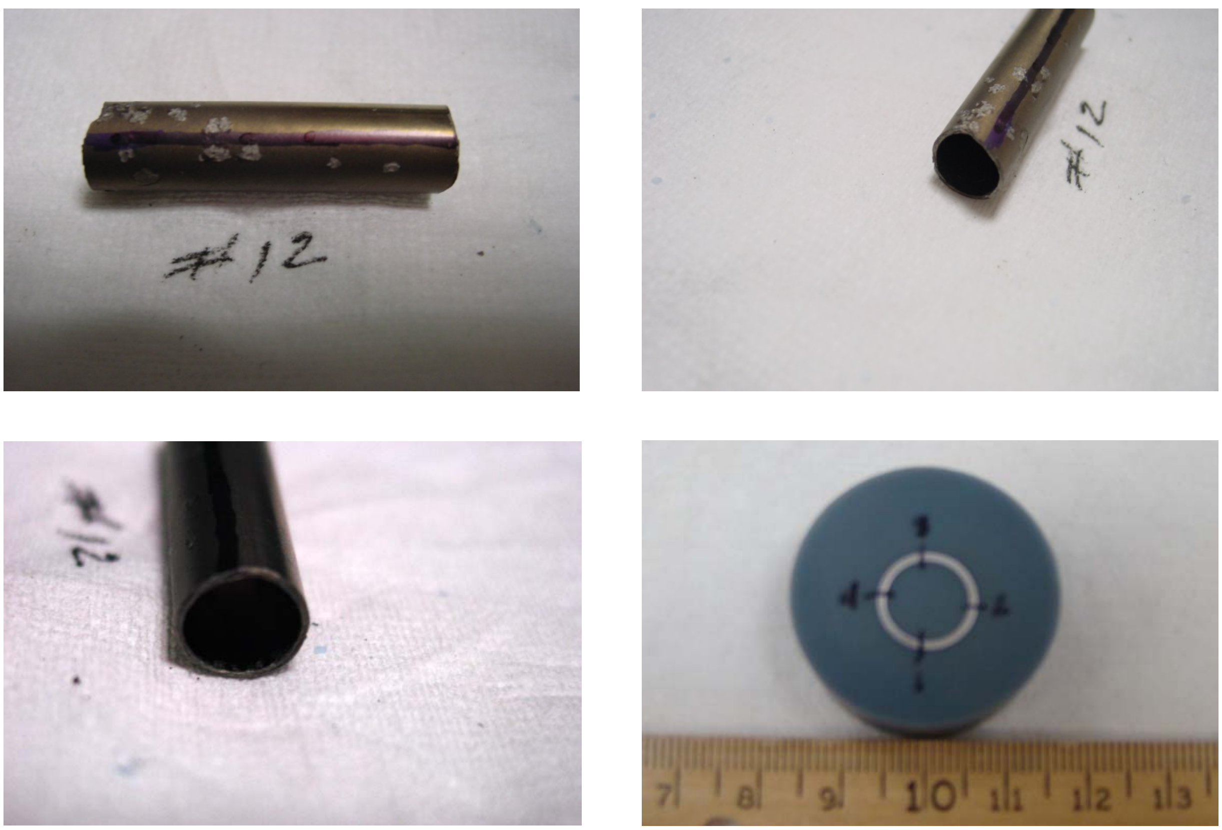
Sample 11 / Quadrant 4 / 200x Magnification

Sample ID: 12

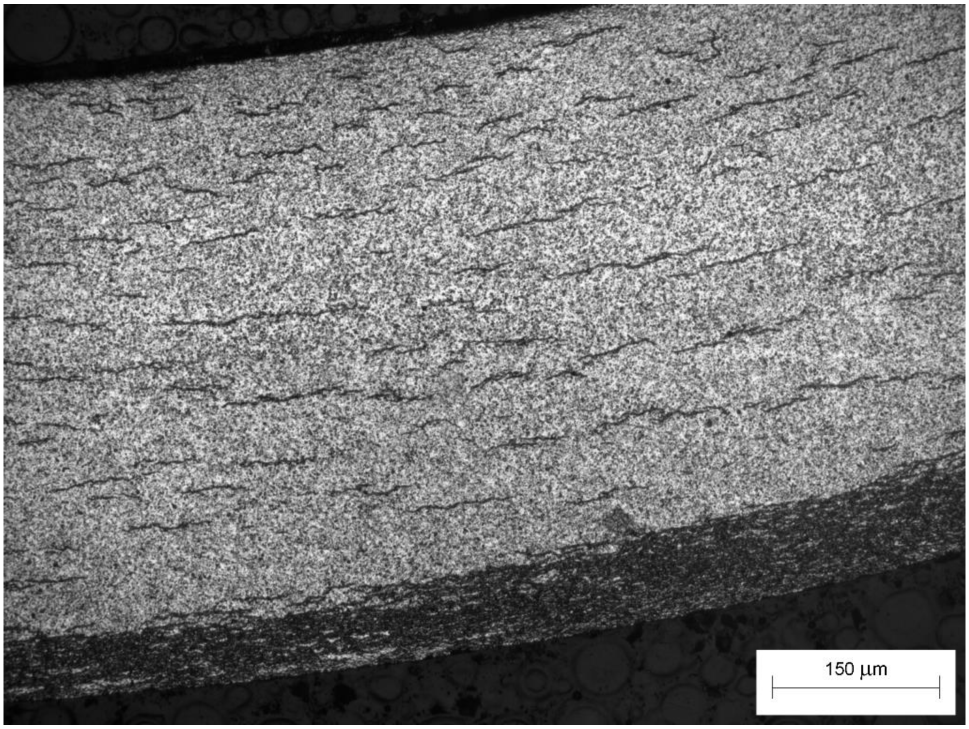
Furnace:	CAMCo
Location:	Bottom Tray
Temperature:	300°C Control Temperature (Actual Between 282-287°C)
OD Surface:	Blasted
Outside Diameter:	0.372 in (9.5 mm)
Wall Thickness:	0.022 in (0.56 mm)

Hydrogen Concentration Results

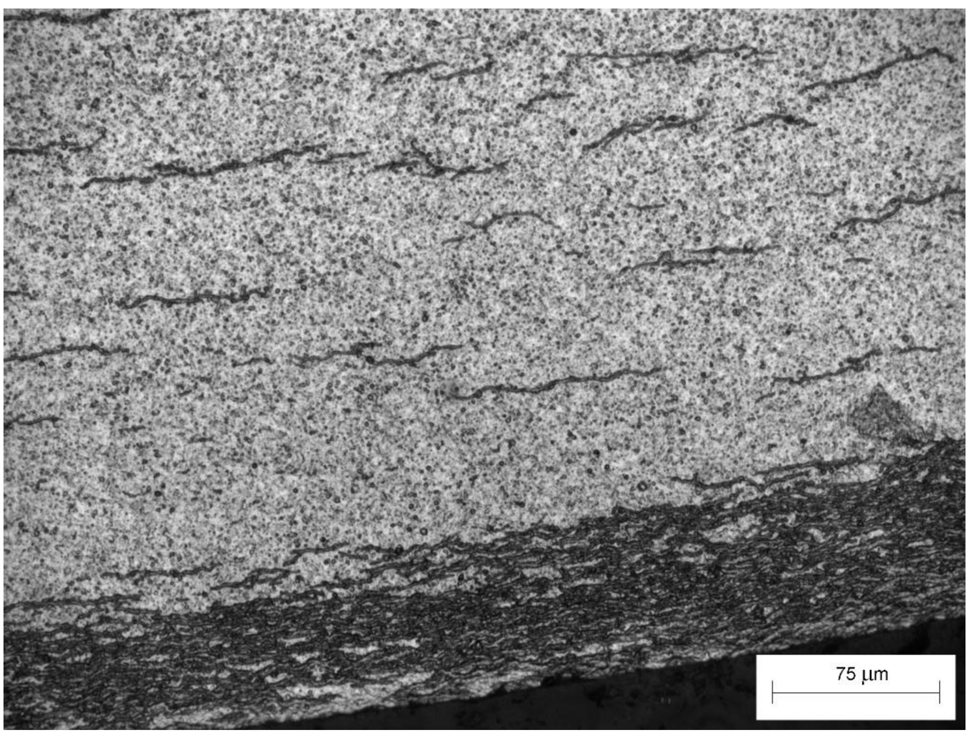
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Top	1900	2700	3100	NA	2567	611	24%
Middle-Top	1600	2900	920	500	1480	1050	71%
Middle-Bot	290	2100	1200	310	975	862	88%
Bottom	1900	2100	1500	NA	1833	306	17%



Images of Sample 12 After Hydriding and Mounted in Epoxy



Sample 12 / Quadrant 1 / 100x Magnification



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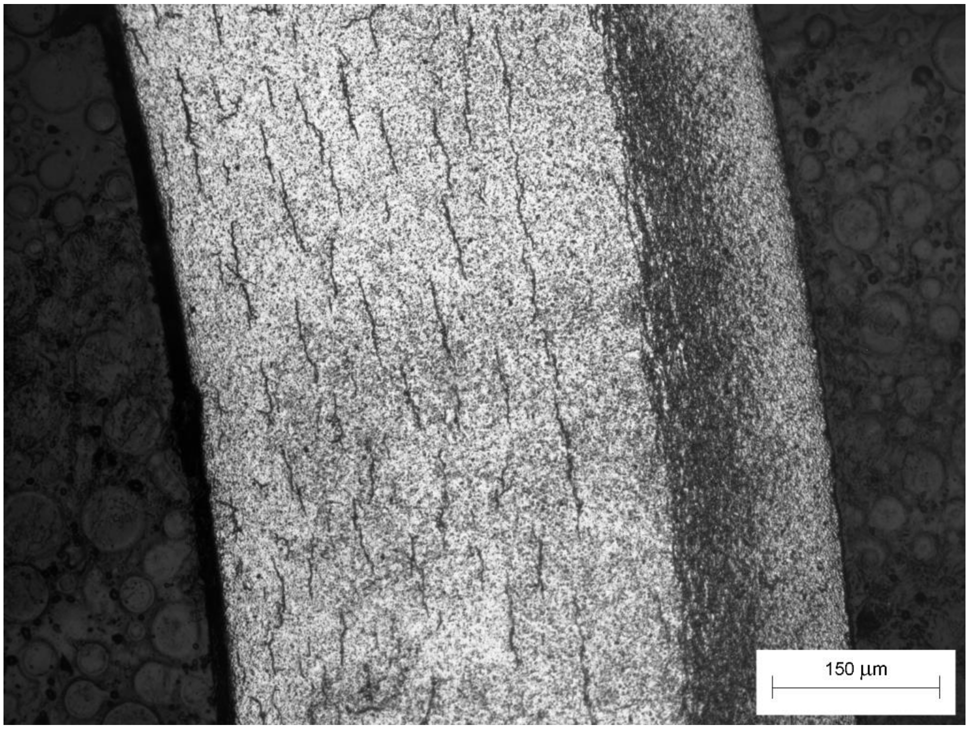
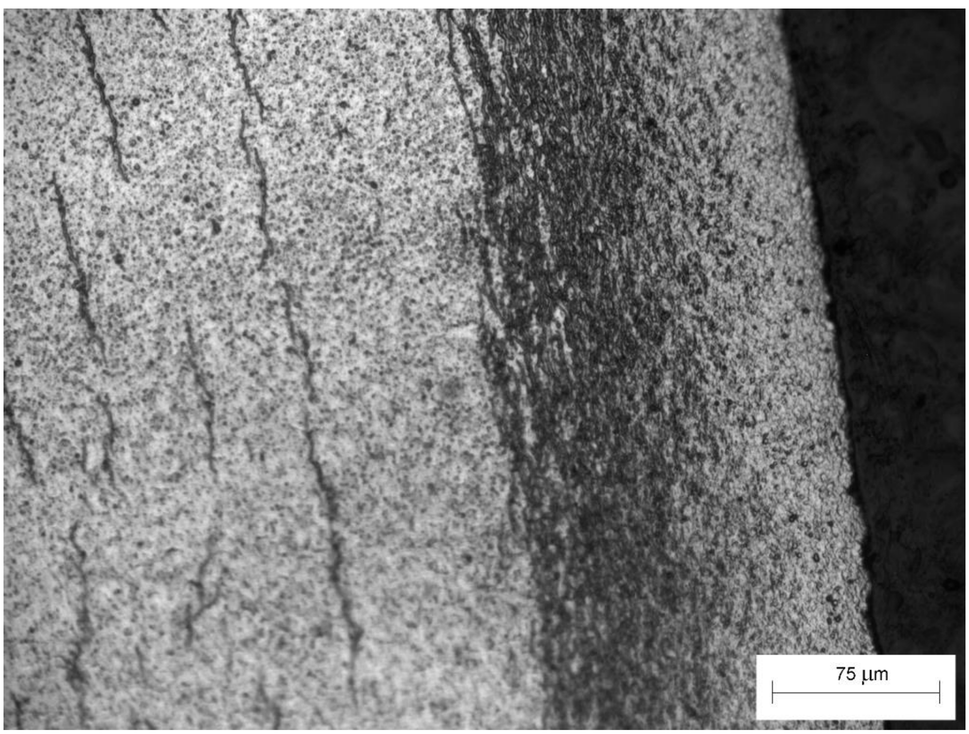


Sample 12 / Quadrant 2 / 100x Magnification



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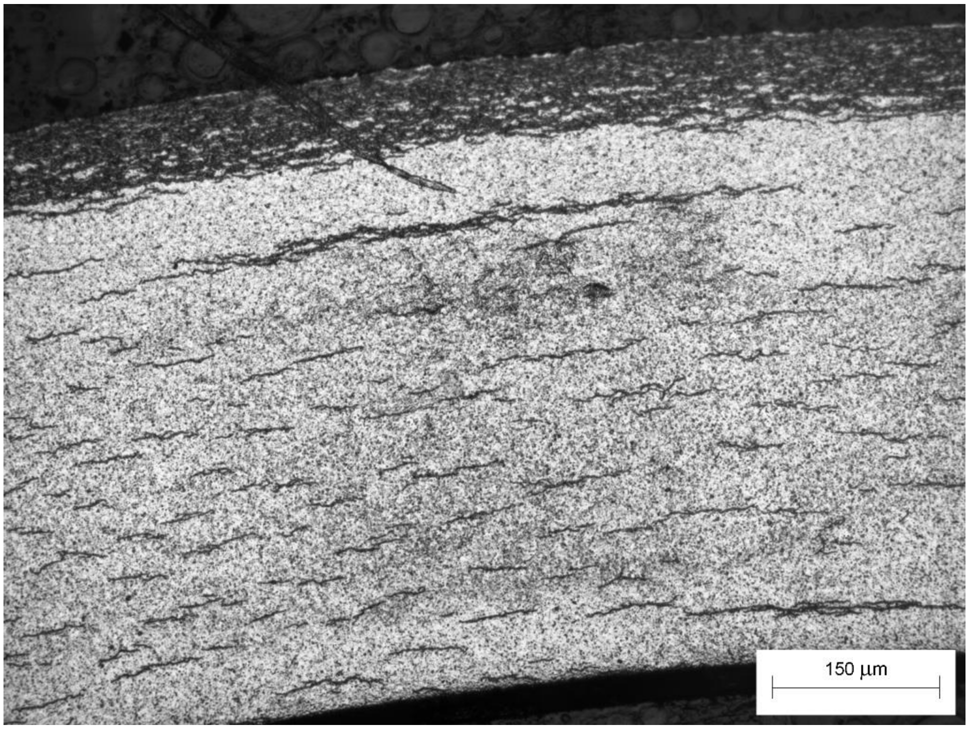
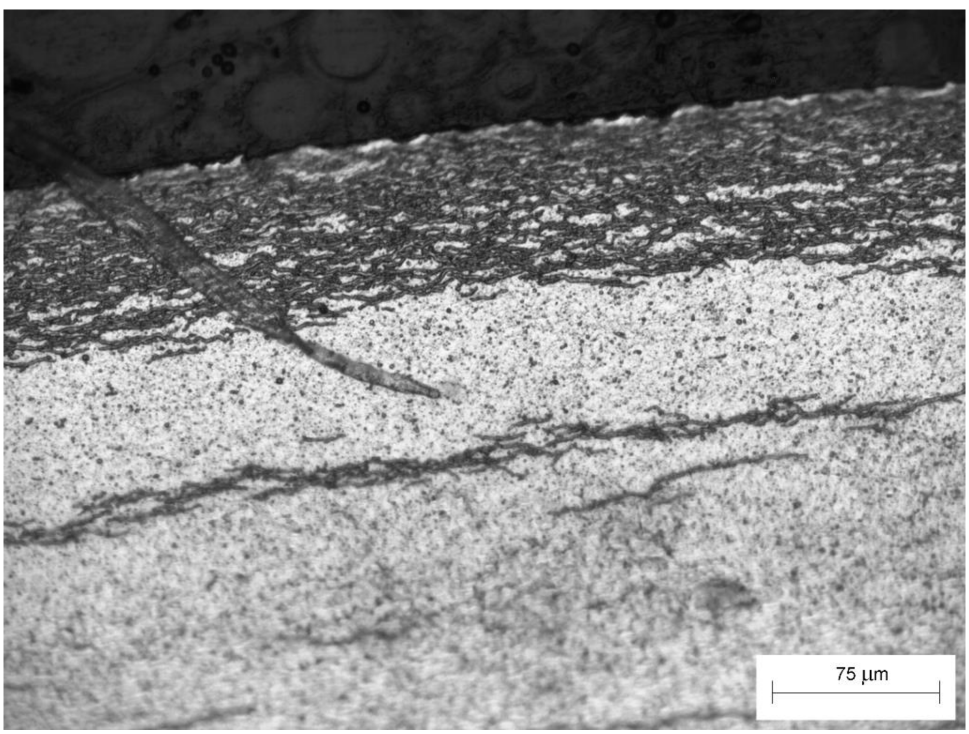


Sample 12 / Quadrant 3 / 100x Magnification



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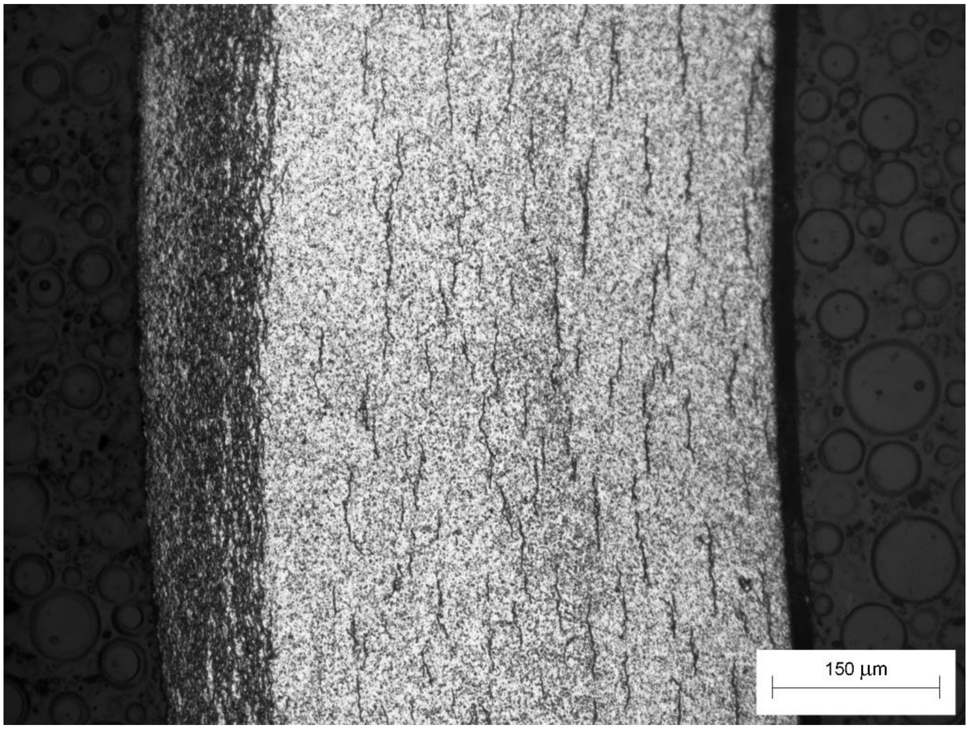
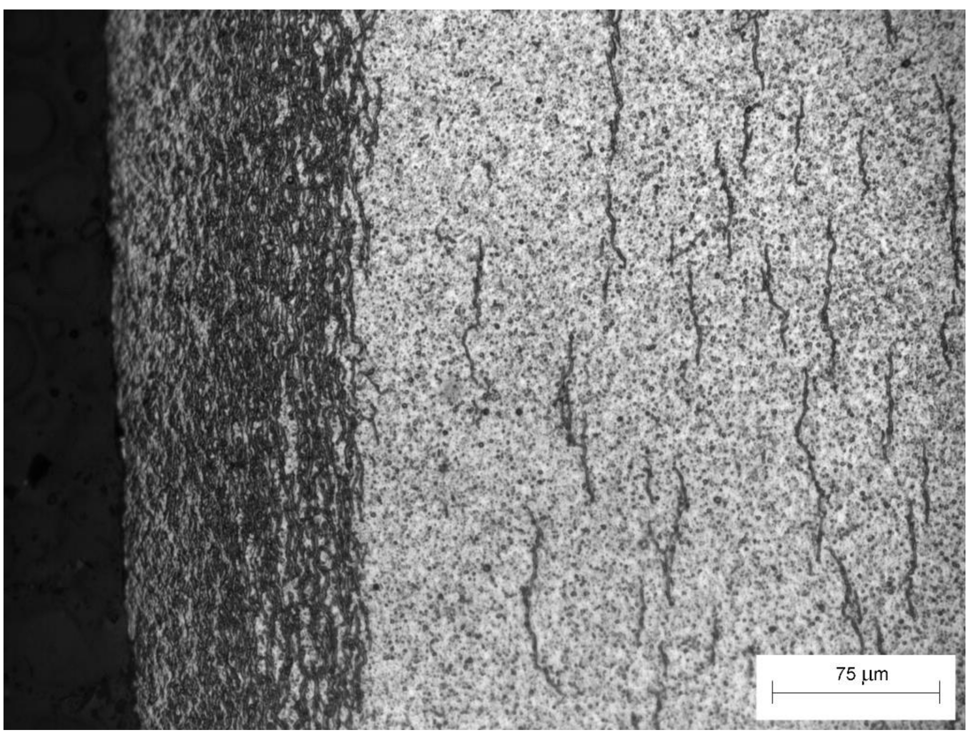


Sample 12 / Quadrant 4 / 100x Magnification



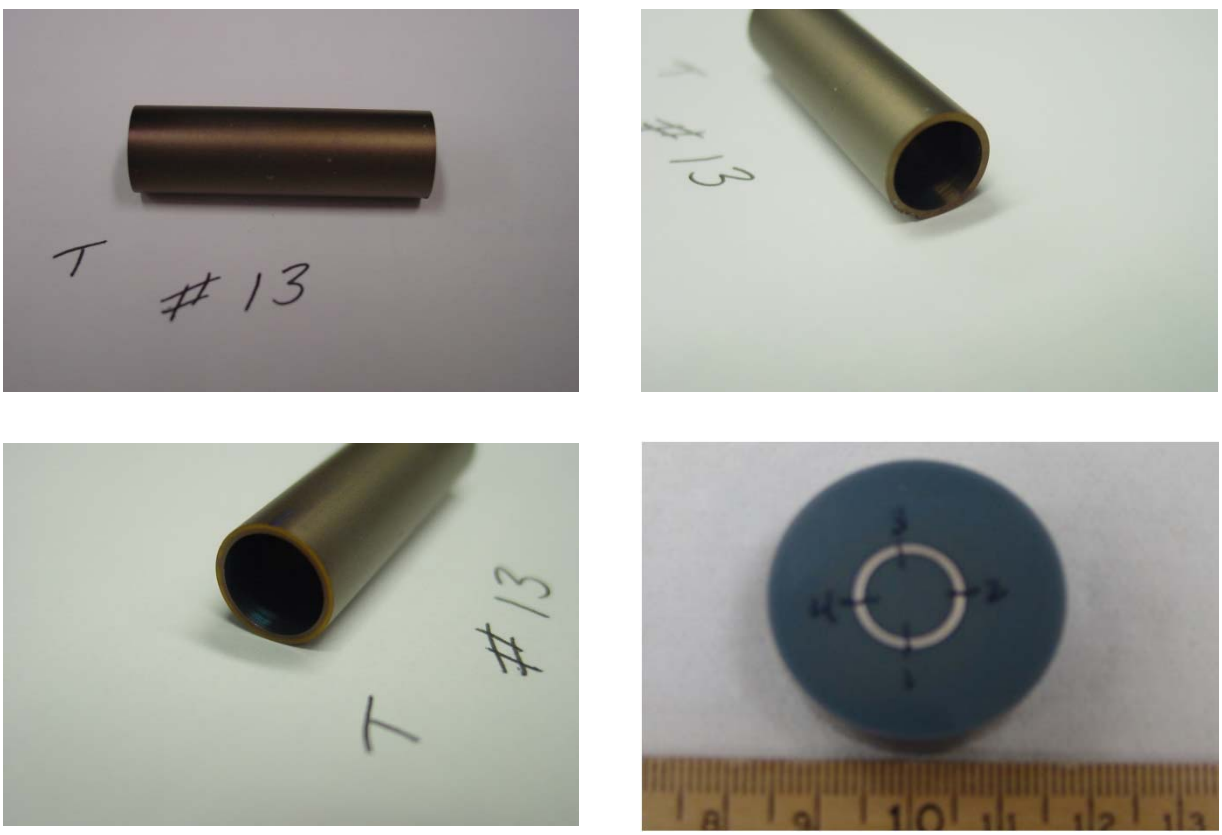
Sample 12 / Quadrant 4 / 200x Magnification

Sample ID: 13

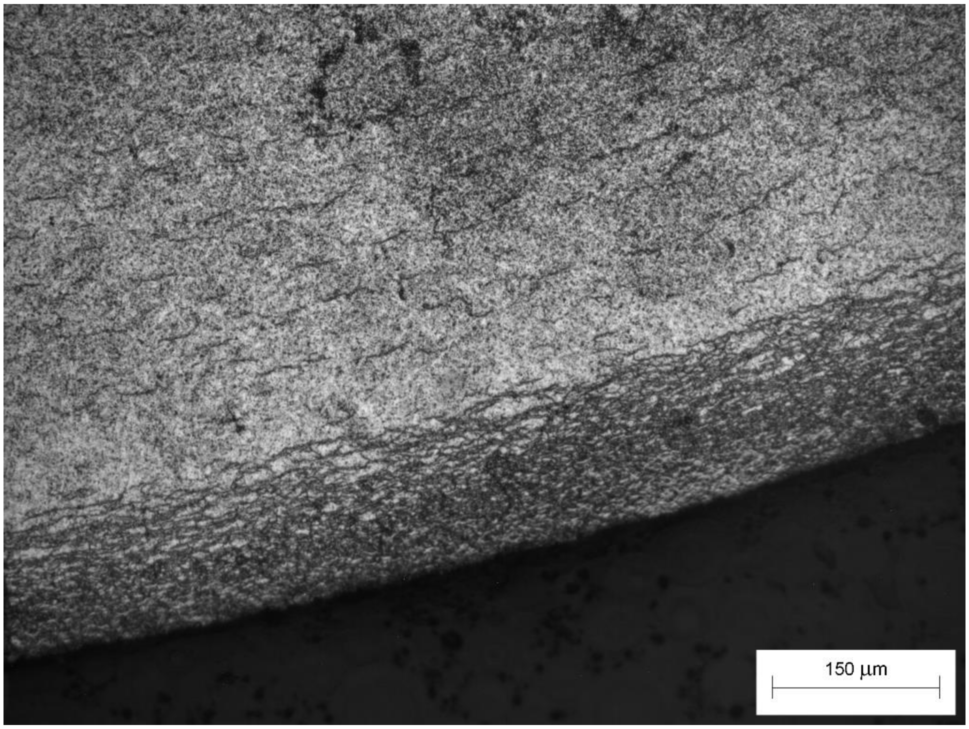
Furnace: CAMCo
Location: Bottom Tray
Temperature: 300°C Control Temperature (Actual Between 282-287°C)
OD Surface: Blasted, 24 Hr Hold in Air
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

Hydrogen Concentration Results

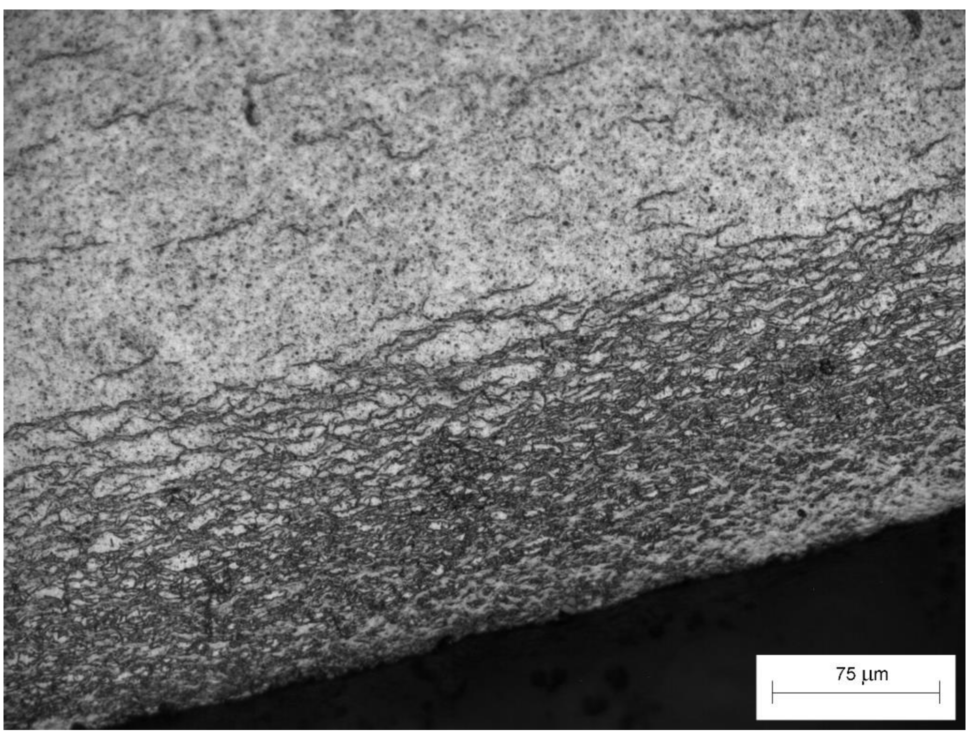
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	780	880	800	800	815	44	5%
Middle-Bot	320	540	470	500	458	96	21%



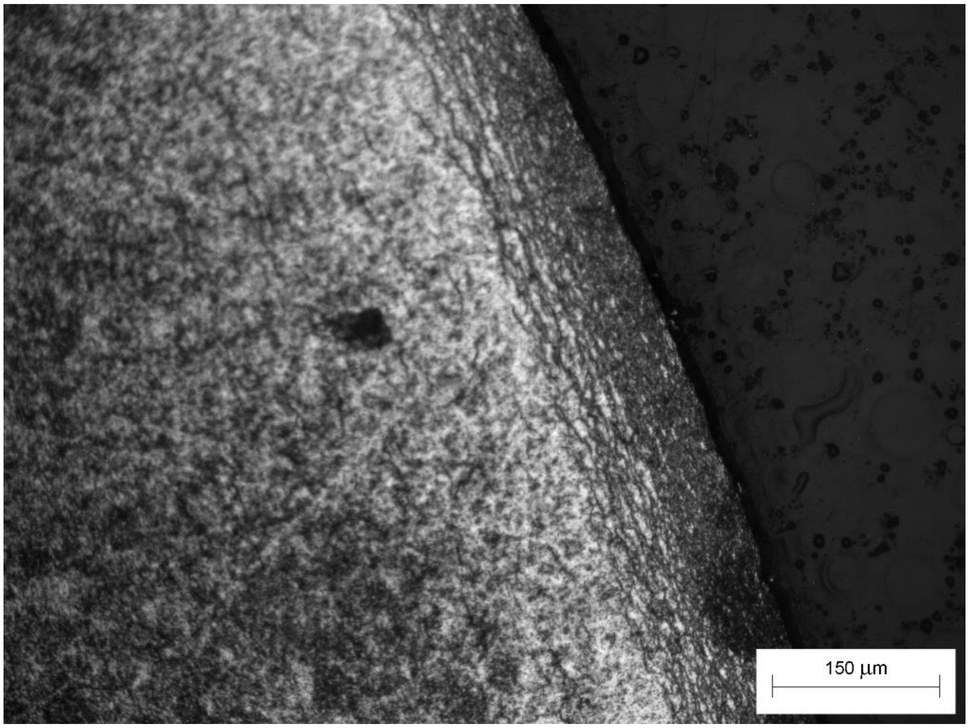
Images of Sample 13 After Hydriding and Mounted in Epoxy



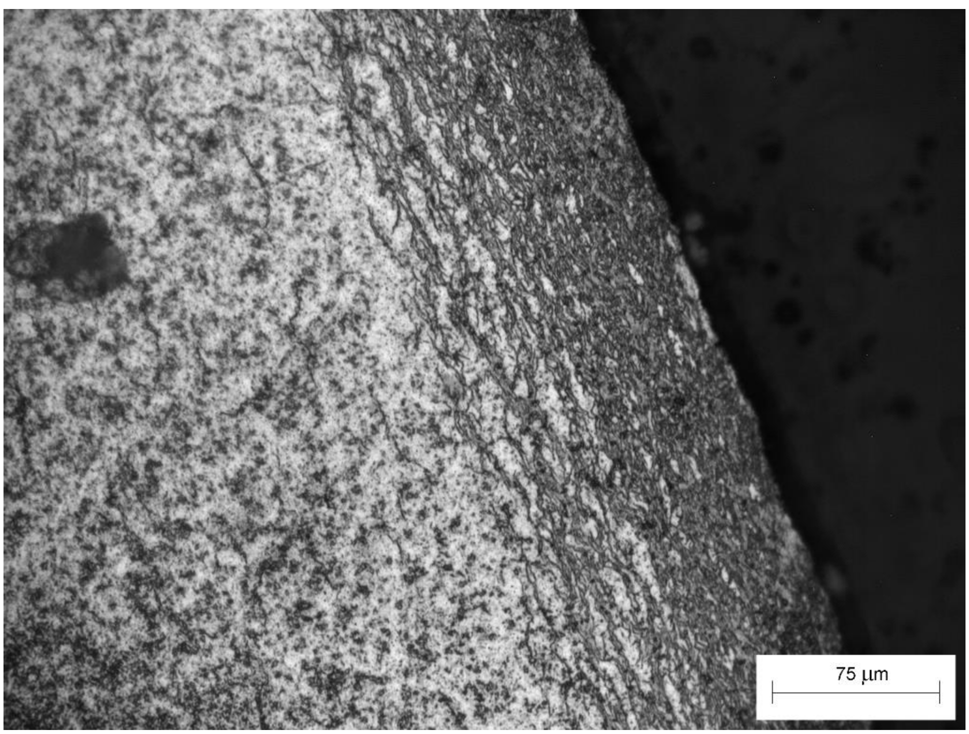
Sample 13 / Quadrant 1 / 100x Magnification



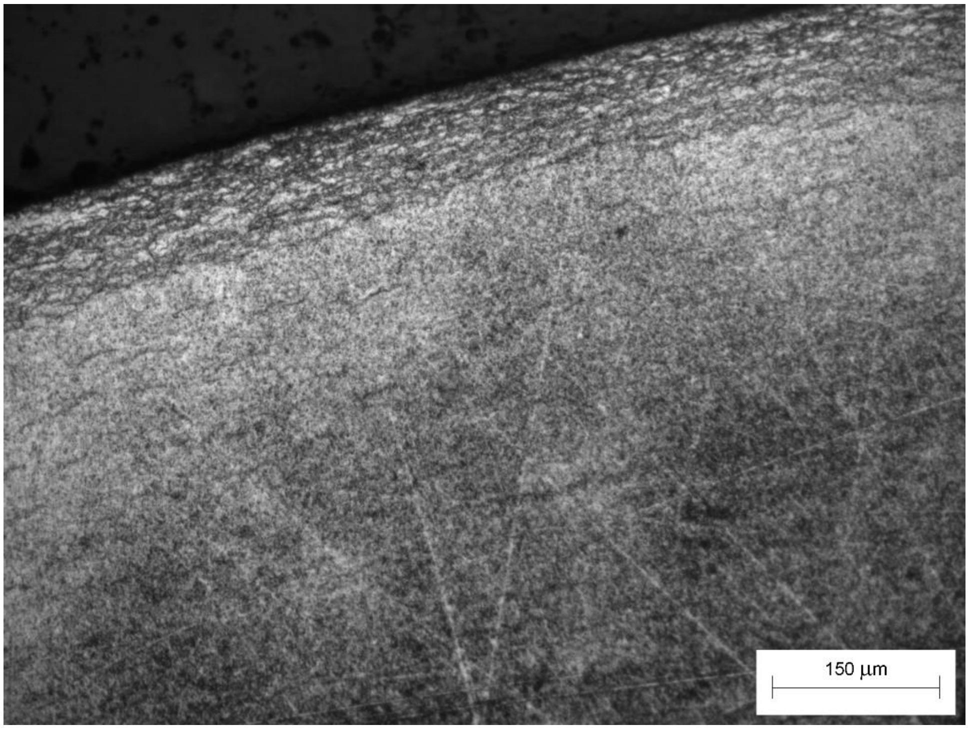
Sample 13 / Quadrant 1 / 200x Magnification



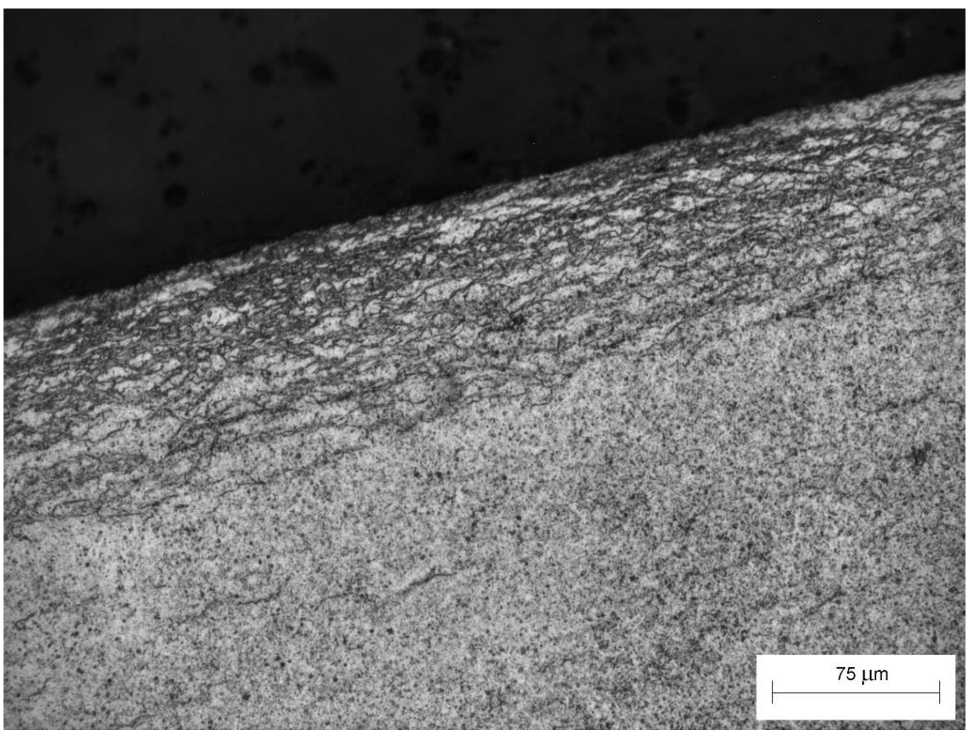
Sample 13 / Quadrant 2 / 100x Magnification



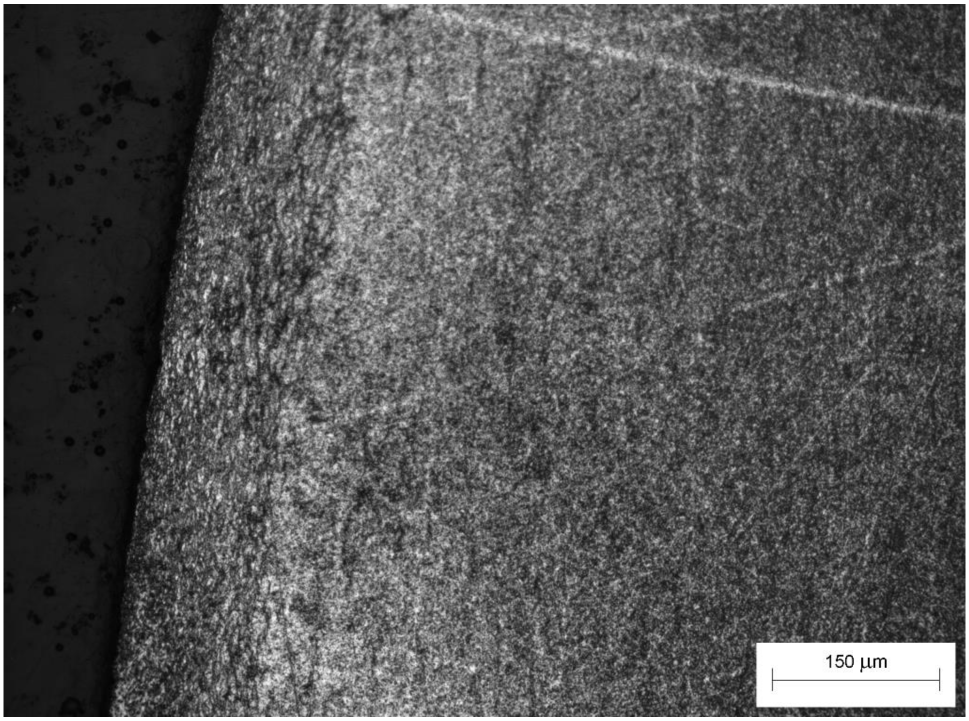
Sample 13 / Quadrant 2 / 200x Magnification



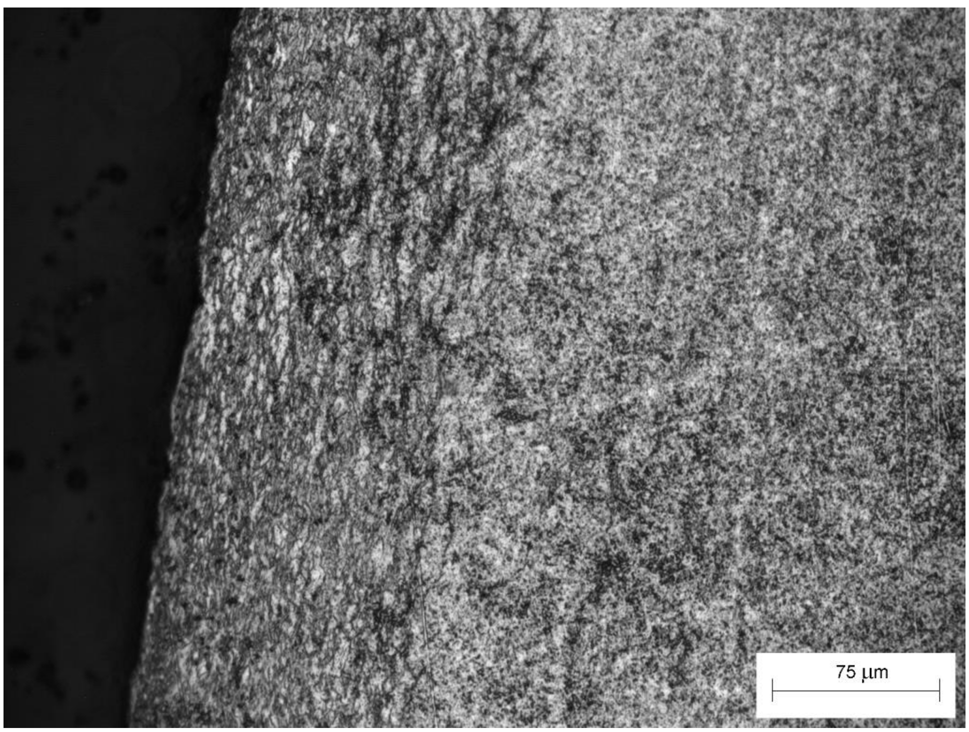
Sample 13 / Quadrant 3 / 100x Magnification



Sample 13 / Quadrant 3 / 200x Magnification



Sample 13 / Quadrant 4 / 100x Magnification



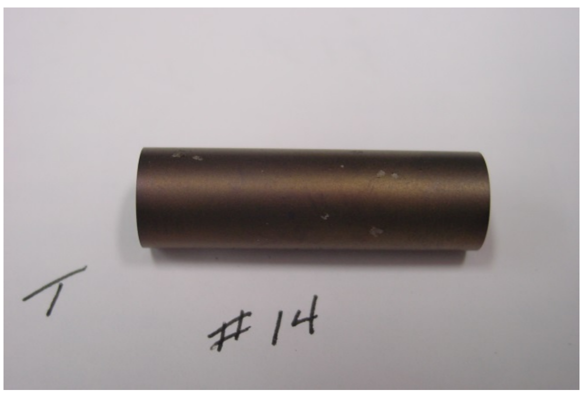
Sample 13 / Quadrant 4 / 200x Magnification

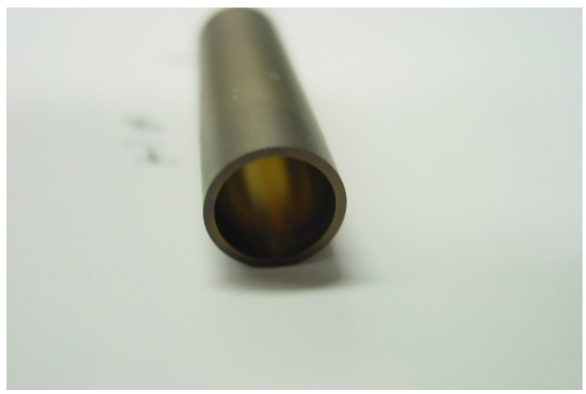
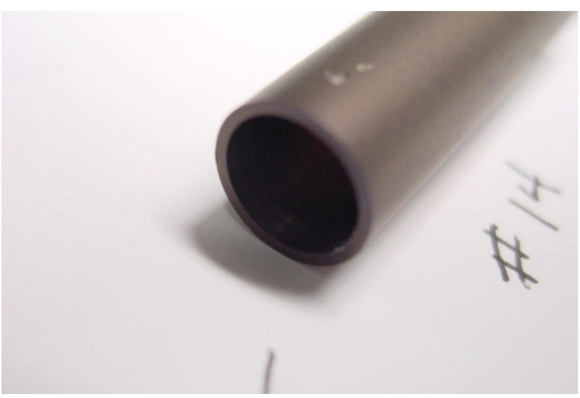
Sample ID: 14

Furnace: CAMCo
Location: Bottom Tray
Temperature: 300°C Control Temperature (Actual Between 282-287°C)
OD Surface: Blasted
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

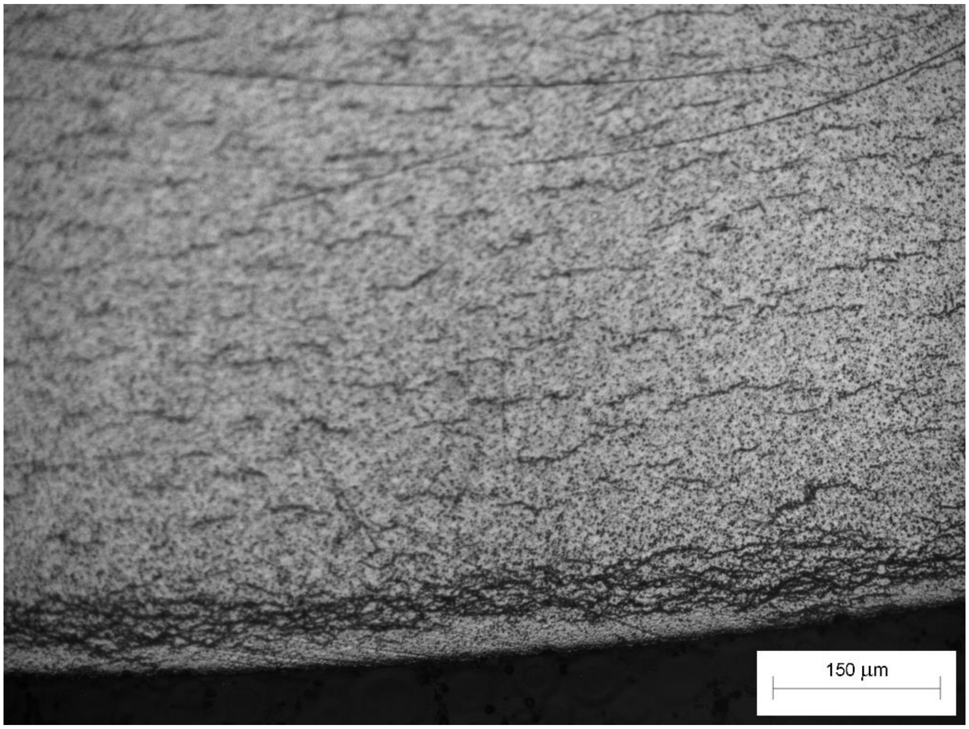
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	190	210	320	420	285	107	37%
Middle-Bot	210	370	240	270	273	69	25%

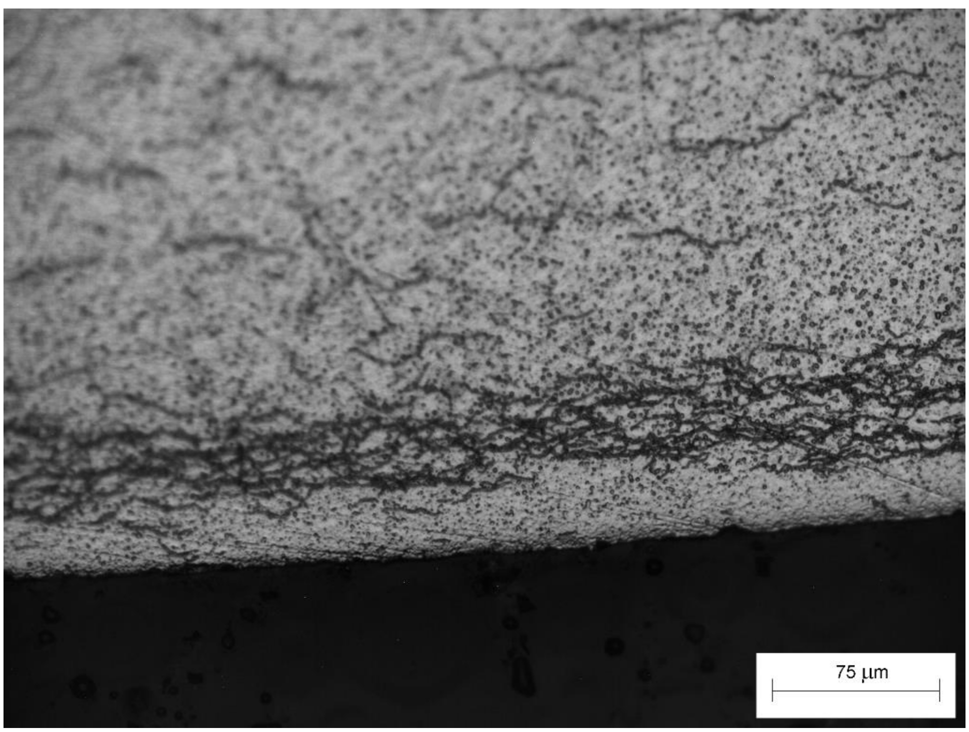


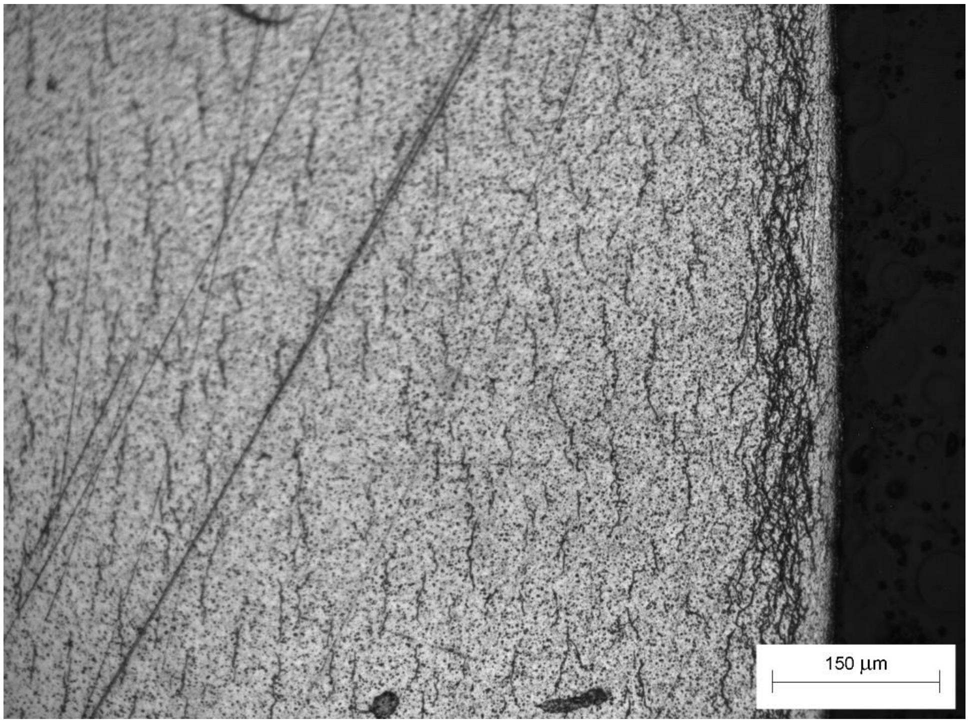
Images of Sample 14 After Hydriding and Mounted in Epoxy



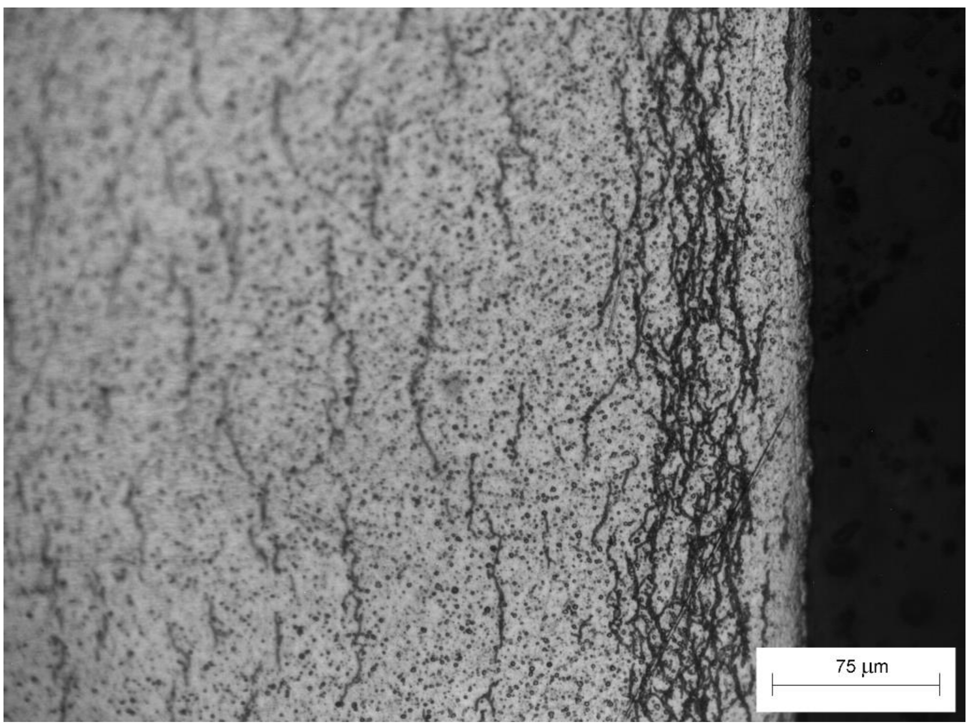
Sample 14 / Quadrant 1 / 100x Magnification



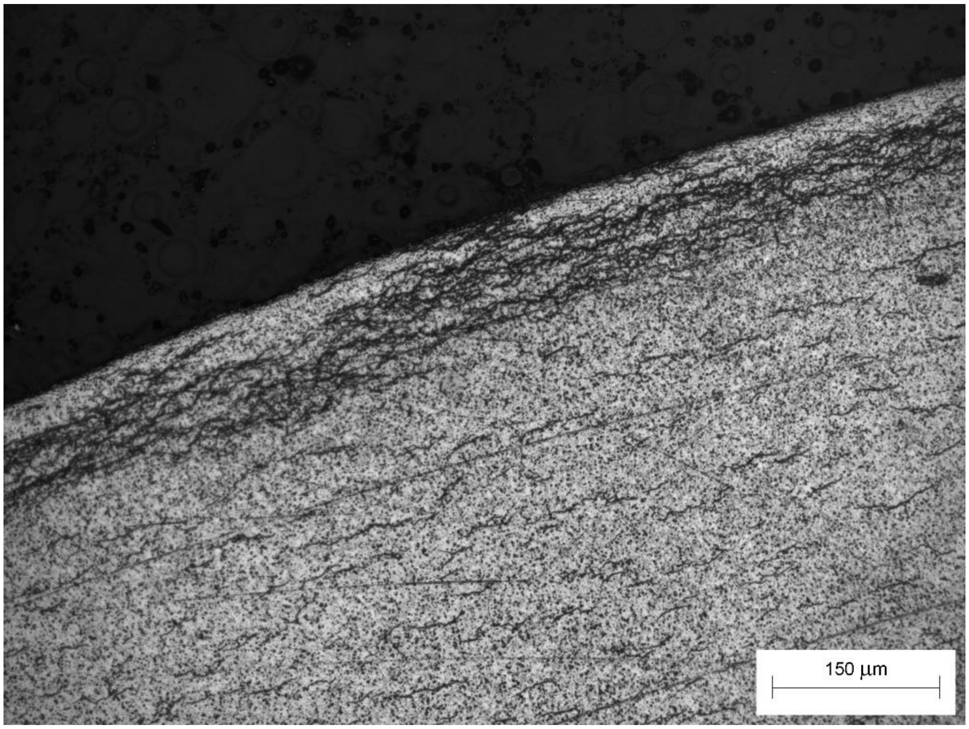
Sample 14 / Quadrant 1 / 200x Magnification



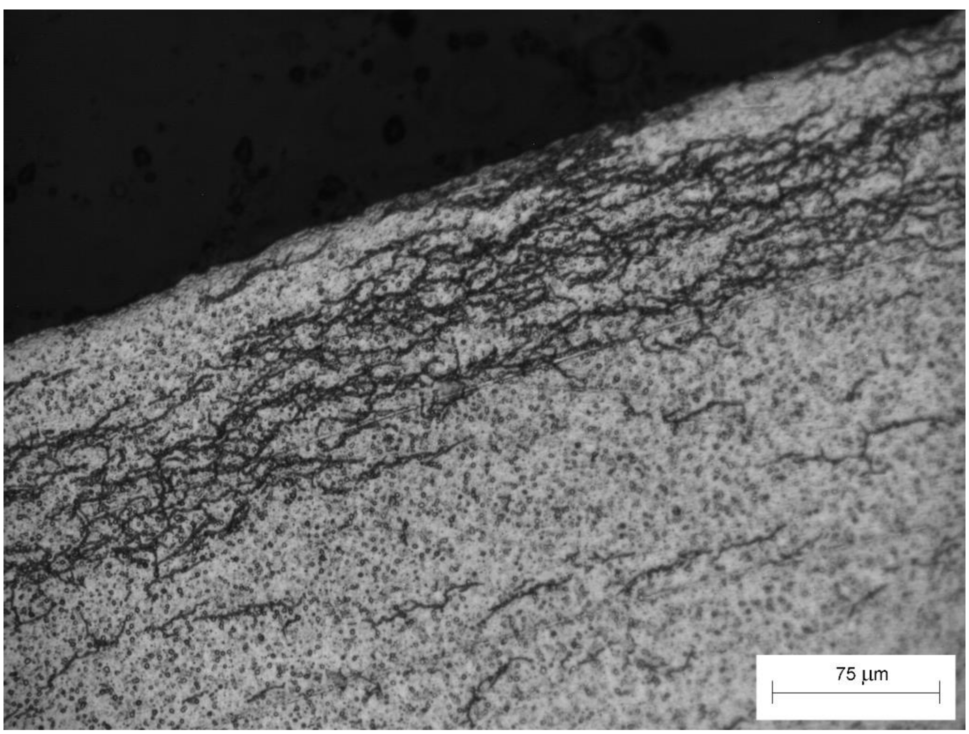
Sample 14 / Quadrant 2 / 100x Magnification



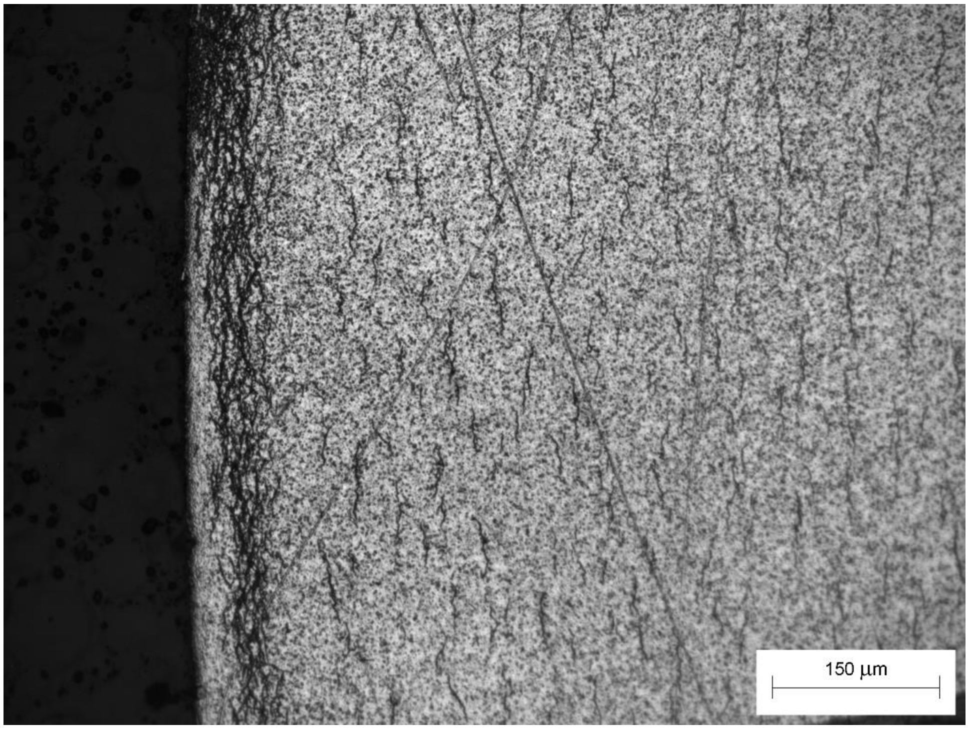
Sample 14 / Quadrant 2 / 200x Magnification



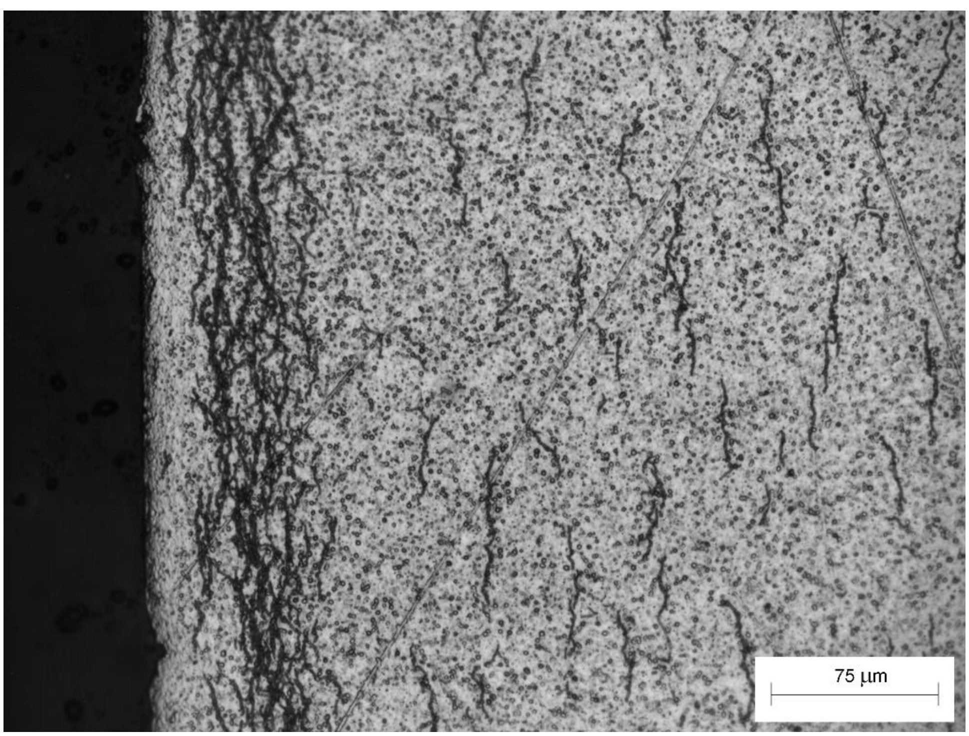
Sample 14 / Quadrant 3 / 100x Magnification



Sample 14 / Quadrant 3 / 200x Magnification



Sample 14 / Quadrant 4 / 100x Magnification



Sample 14 / Quadrant 4 / 200x Magnification

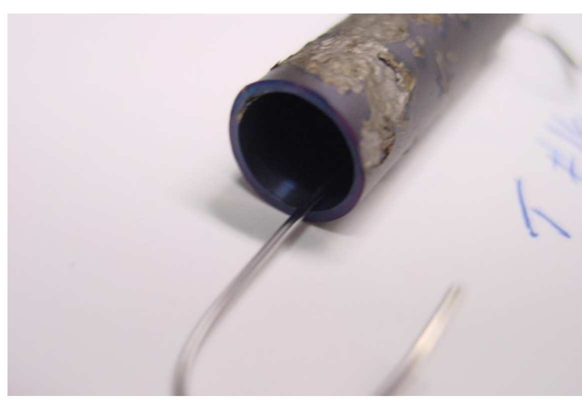
Sample ID: 15

Furnace:	CAMCo
Location:	Hung
Temperature:	300°C
OD Surface:	Blasted, 24 Hr Hold in Air
Outside Diameter:	0.451 in (11.5 mm)
Wall Thickness:	0.036 in (0.90mm)

Images of Sample 15 After Hydriding; No metallography or hydrogen analysis performed.

Sample ID: 16

Furnace:	CAMCo
Location:	Hung
Temperature:	300°C
OD Surface:	Blasted
Outside Diameter:	0.451 in (11.5 mm)
Wall Thickness:	0.036 in (0.90mm)



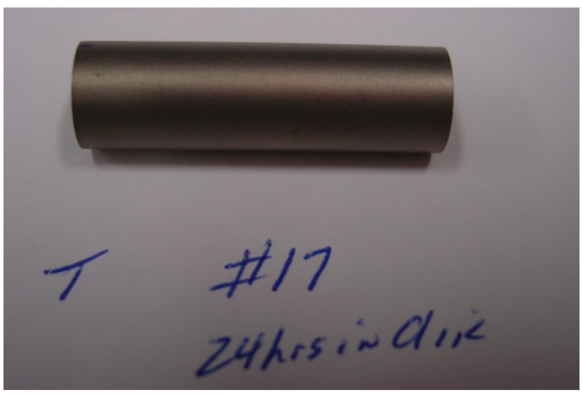
Images of Sample 15 After Hydriding; No metallography or hydrogen analysis performed.

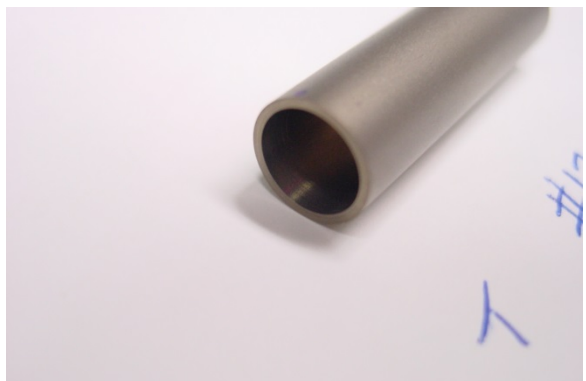
Sample ID: 17

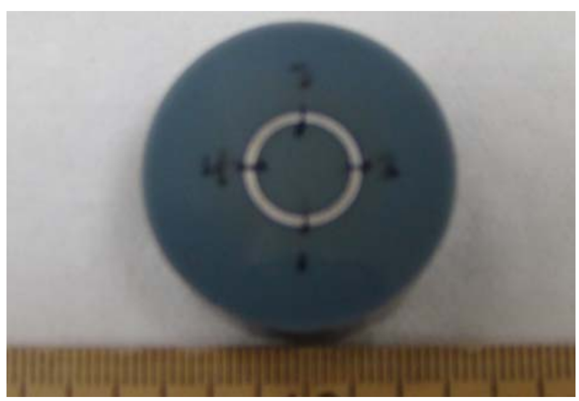
Furnace: CAMCo
Location: Hung
Temperature: 250°C
OD Surface: Blasted, 24 Hr Hold in Air
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

Hydrogen Concentration Results

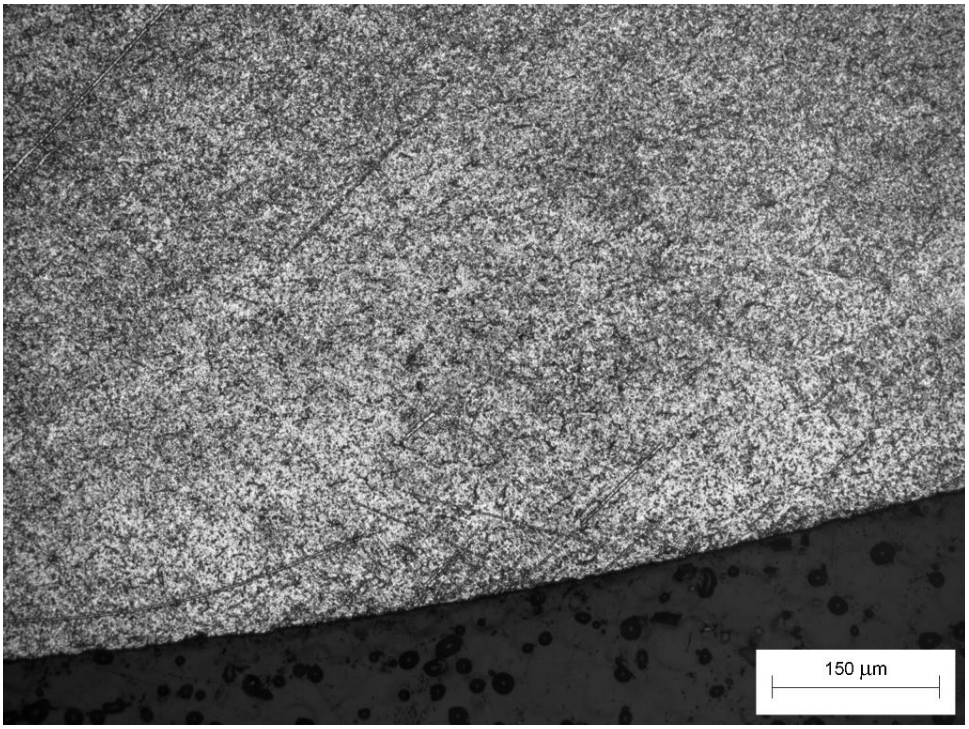
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	58	77	74	60	67	10	14%
Middle-Bot	71	61	57	64	63	6	9%



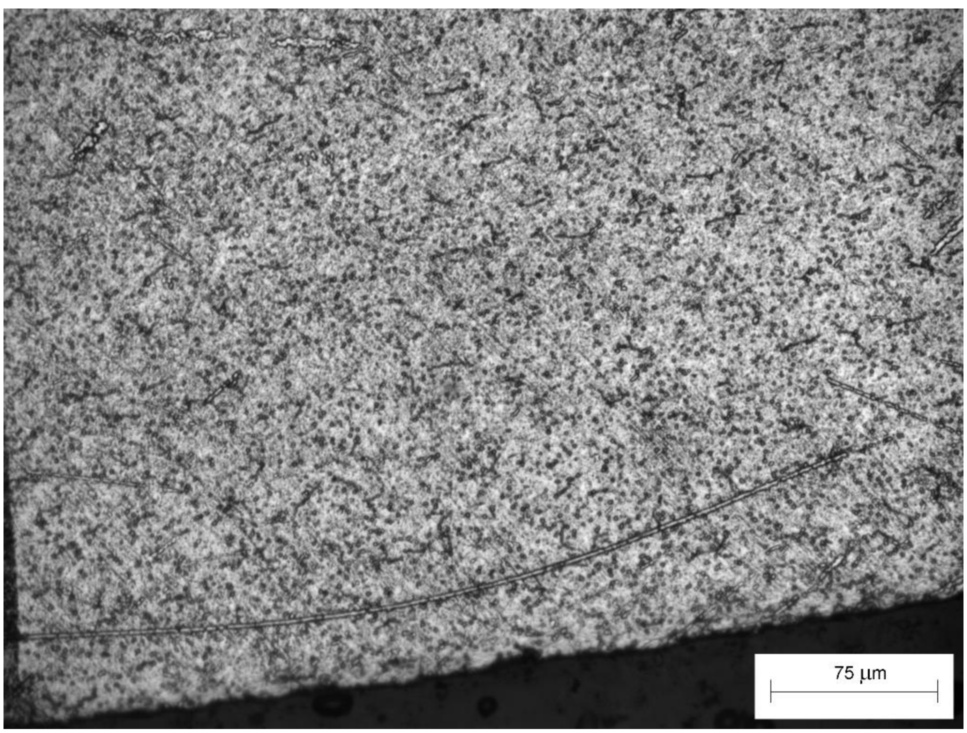




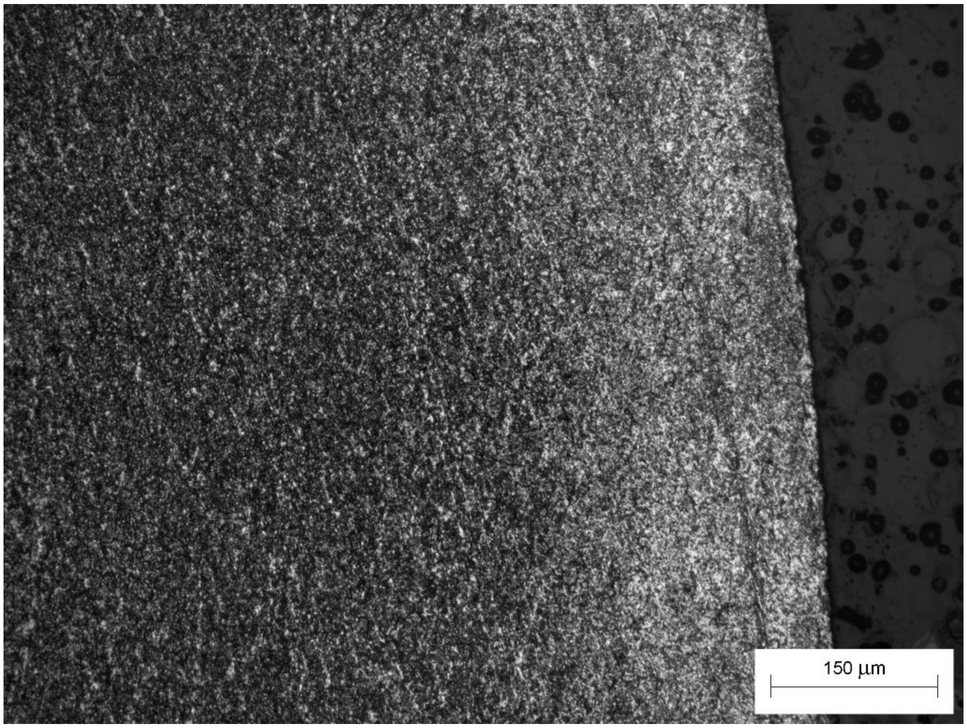
Images of Sample 17 After Hydriding and Mounted in Epoxy



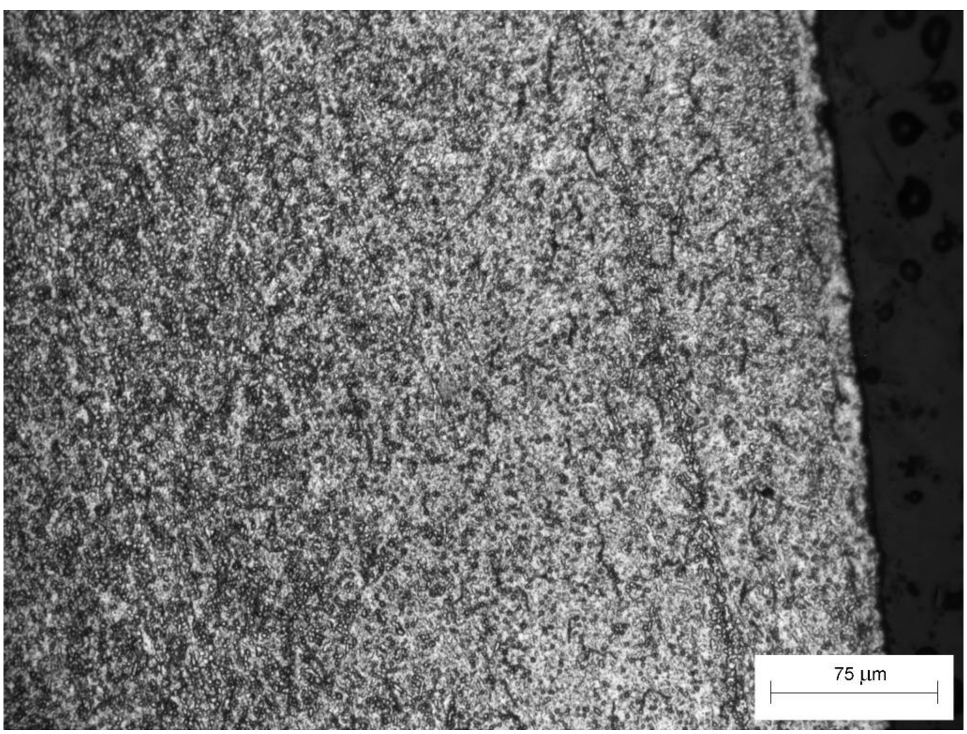
Sample 17 / Quadrant 1 / 100x Magnification



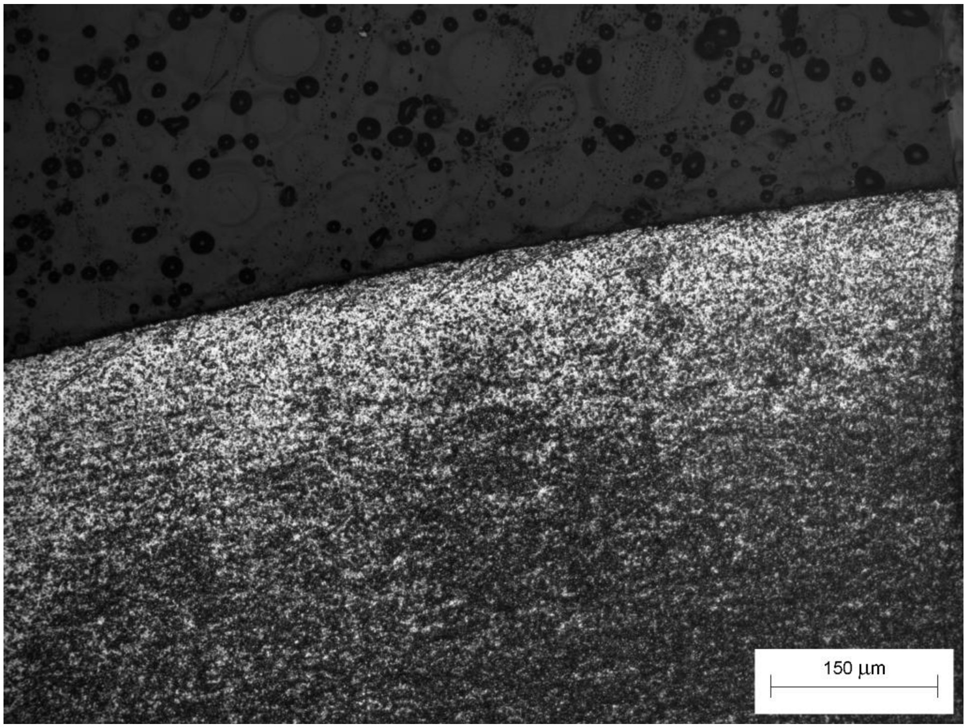
Sample 17 / Quadrant 1 / 200x Magnification



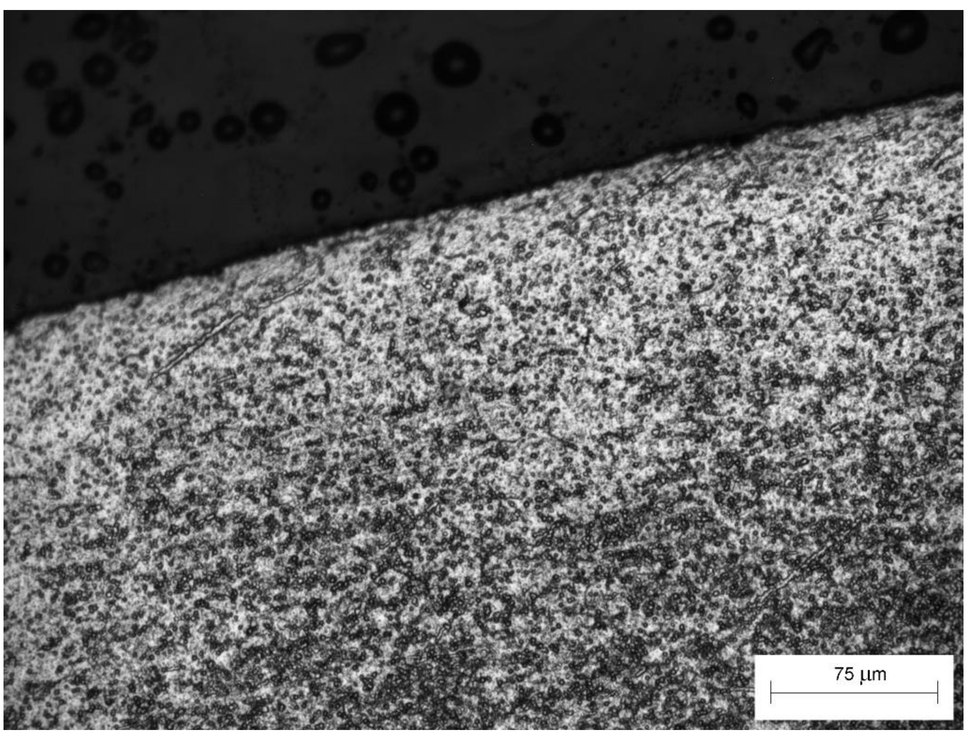
Sample 17 / Quadrant 2 / 100x Magnification



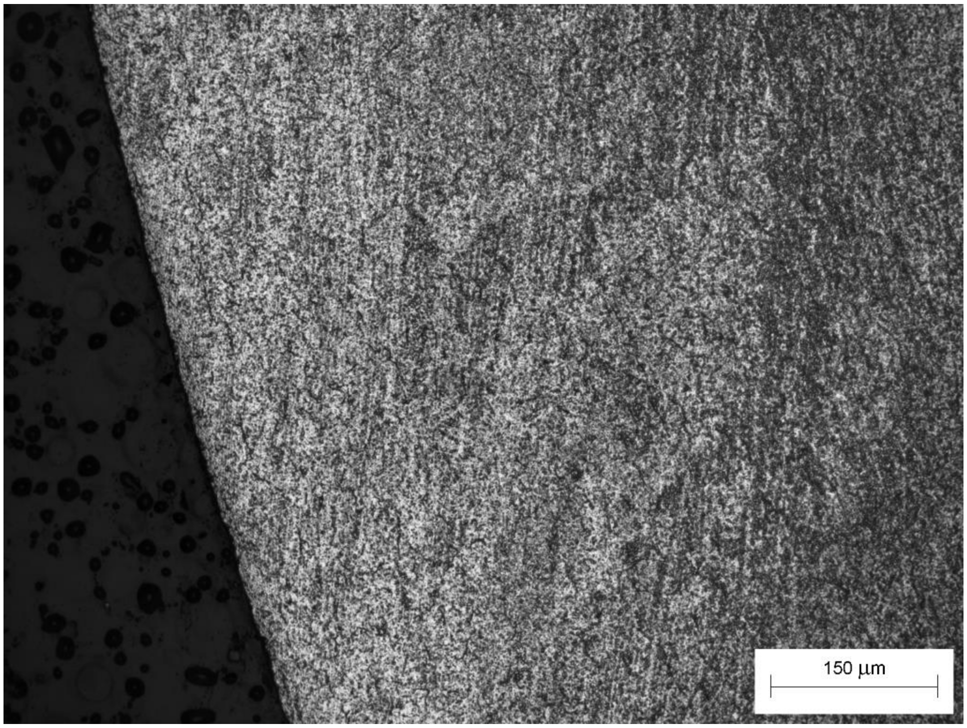
Sample 17 / Quadrant 2 / 200x Magnification



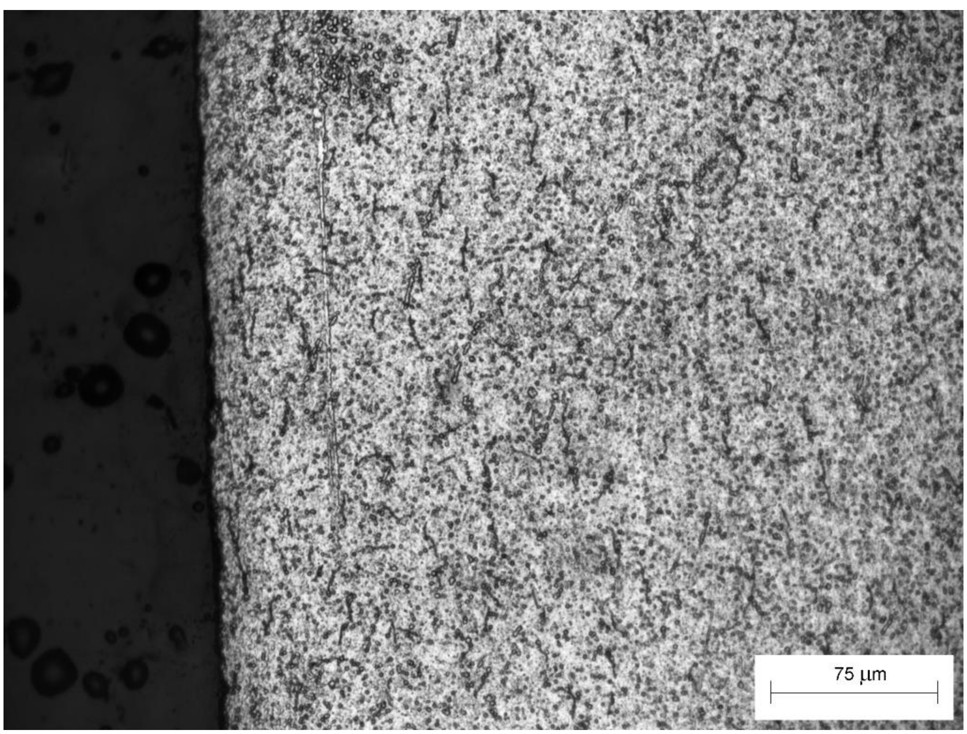
Sample 17 / Quadrant 3 / 100x Magnification



Sample 17 / Quadrant 3 / 200x Magnification



Sample 17 / Quadrant 4 / 100x Magnification



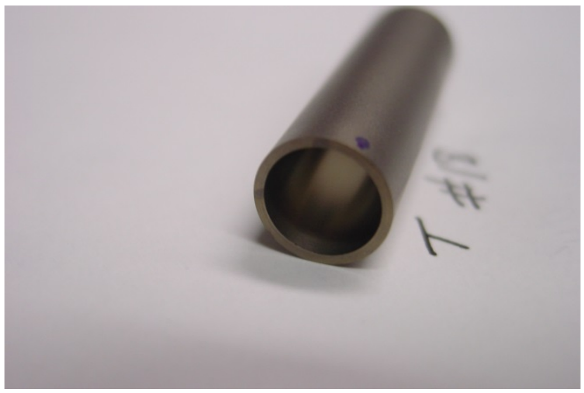
Sample 17 / Quadrant 1 / 200x Magnification

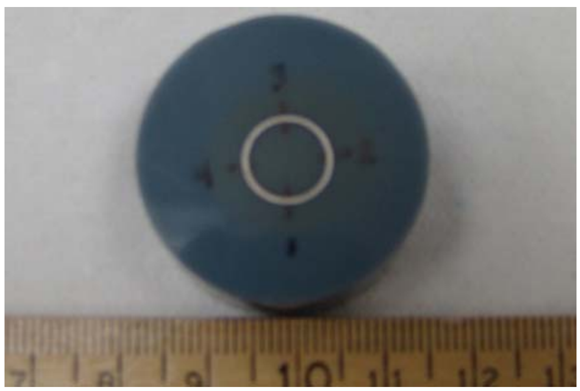
Sample ID: 18

Furnace: CAMCo
Location: Hung
Temperature: 250°C
OD Surface: Blasted
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

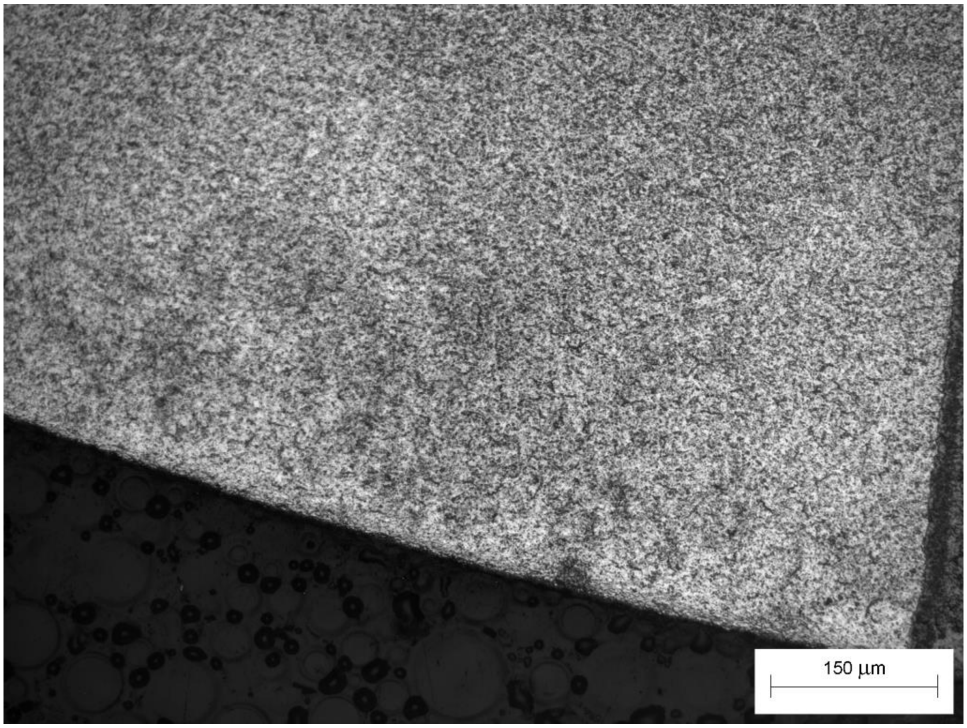
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	71	55	72	66	66	8	12%
Middle-Bot	61	63	62	49	59	7	11%

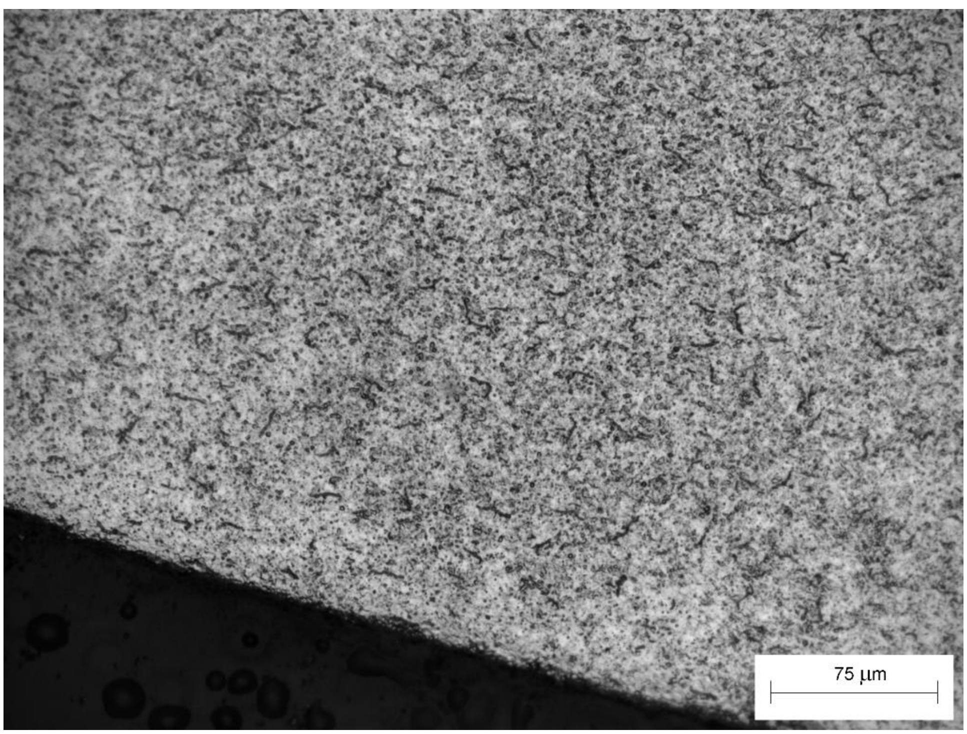




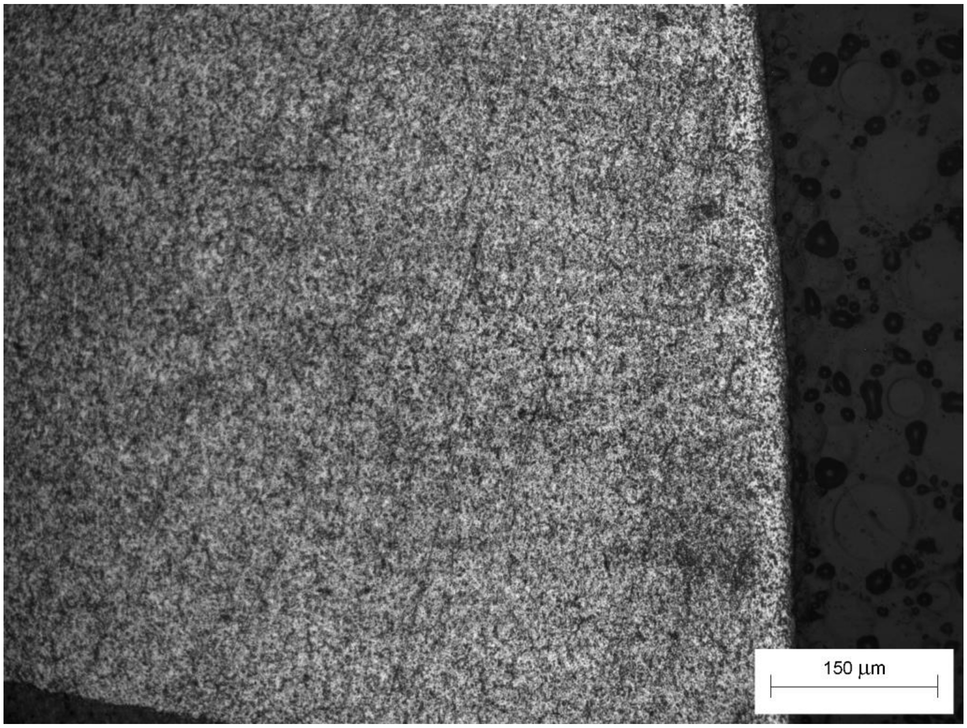
Images of Sample 18 After Hydriding and Mounted in Epoxy



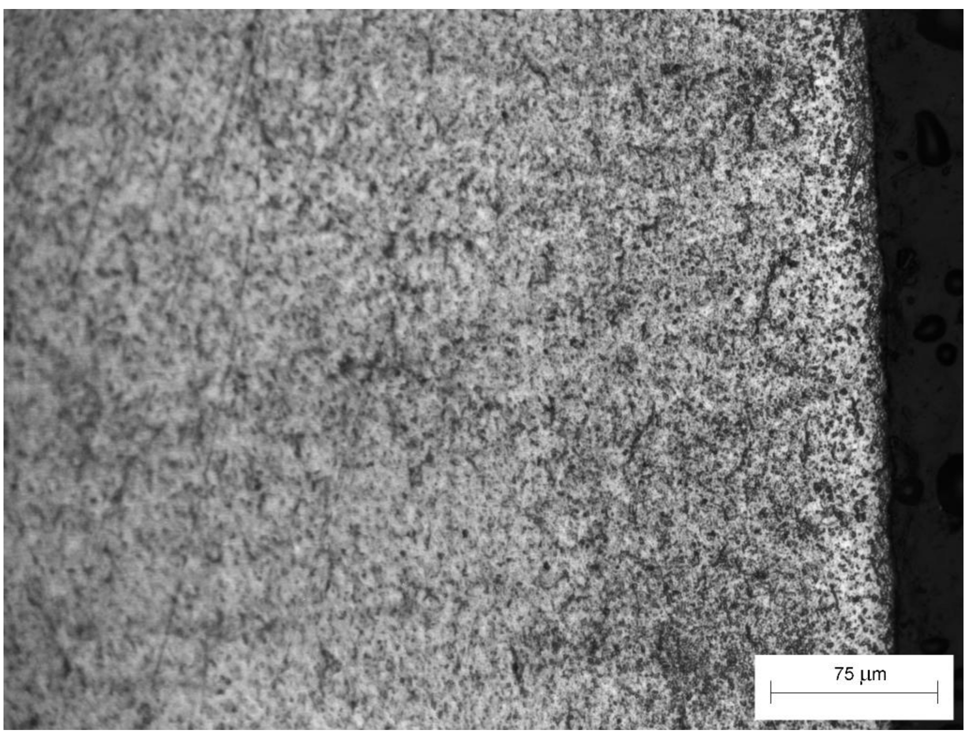
Sample 18 / Quadrant 1 / 100x Magnification



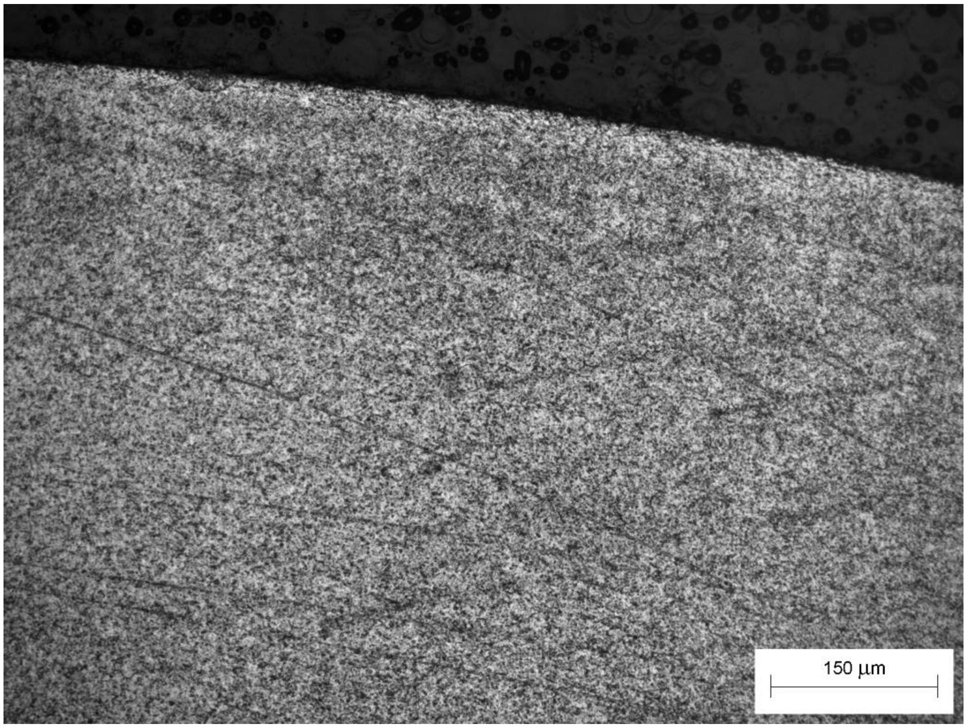
Sample 18 / Quadrant 1 / 200x Magnification



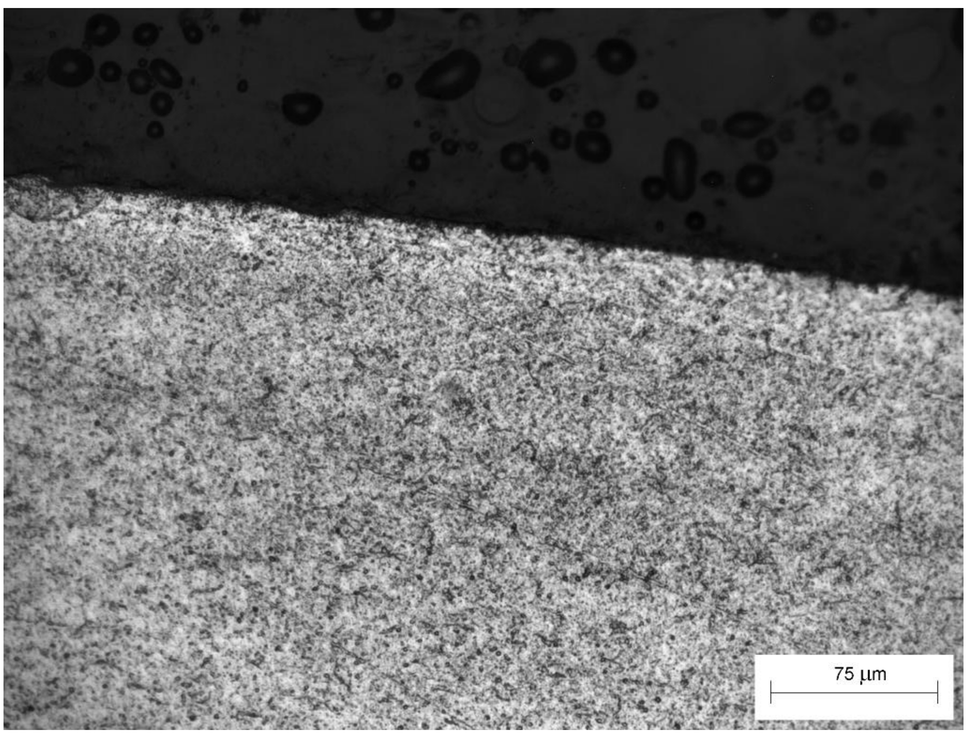
Sample 18 / Quadrant 2 / 100x Magnification



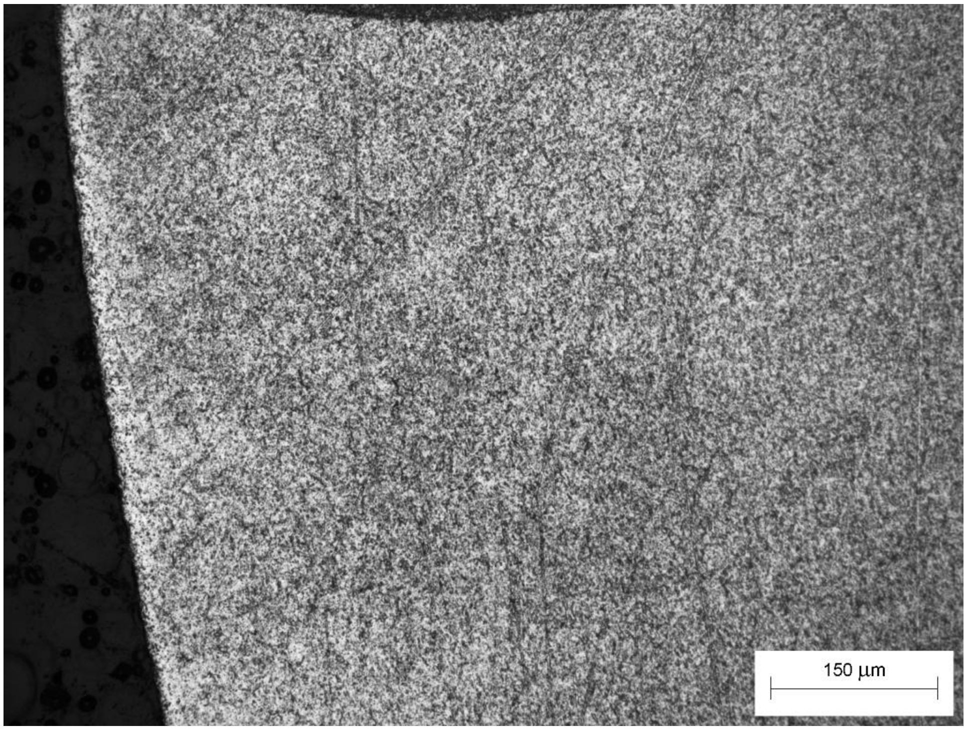
Sample 18 / Quadrant 2 / 200x Magnification



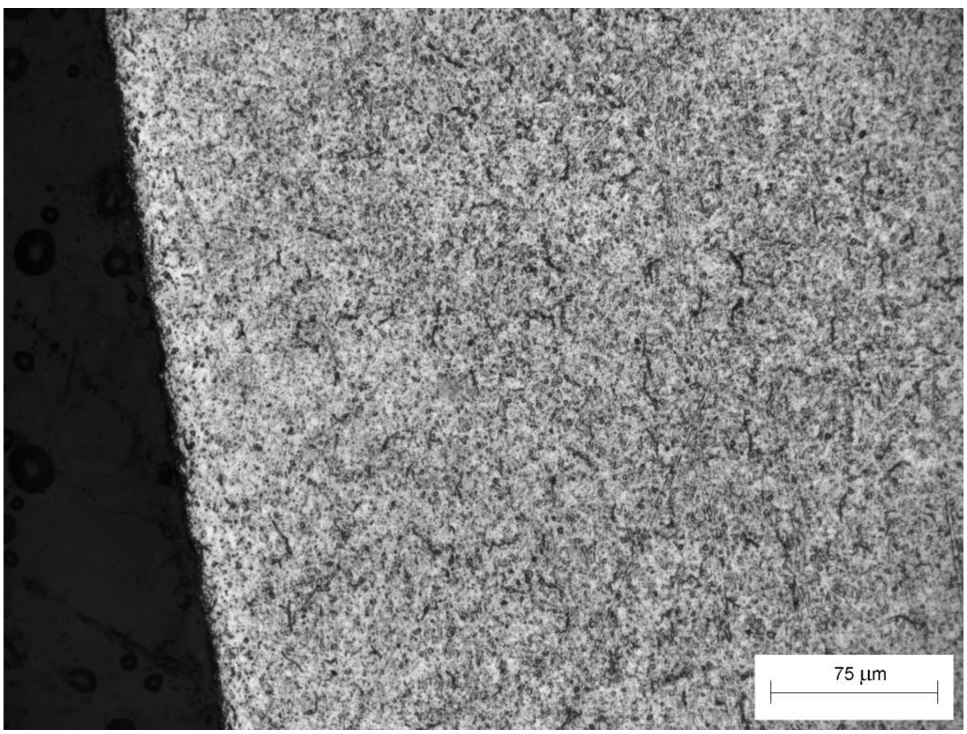
Sample 18 / Quadrant 3 / 100x Magnification



Sample 18 / Quadrant 3 / 200x Magnification



Sample 18 / Quadrant 4 / 100x Magnification



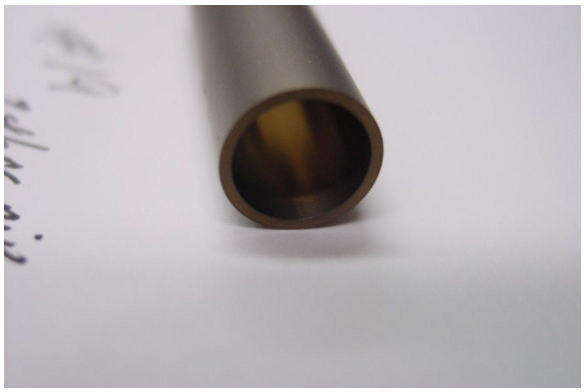
Sample 18 / Quadrant 4 / 200x Magnification

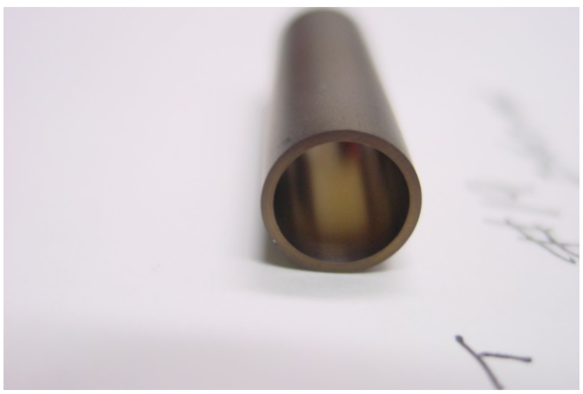
Sample ID: 19

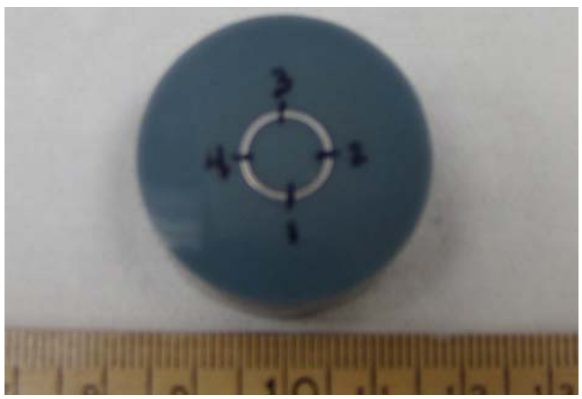
Furnace: CAMCo
Location: Hung
Temperature: 280°C
OD Surface: Blasted, 24 Hr Hold in Air
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

Hydrogen Concentration Results

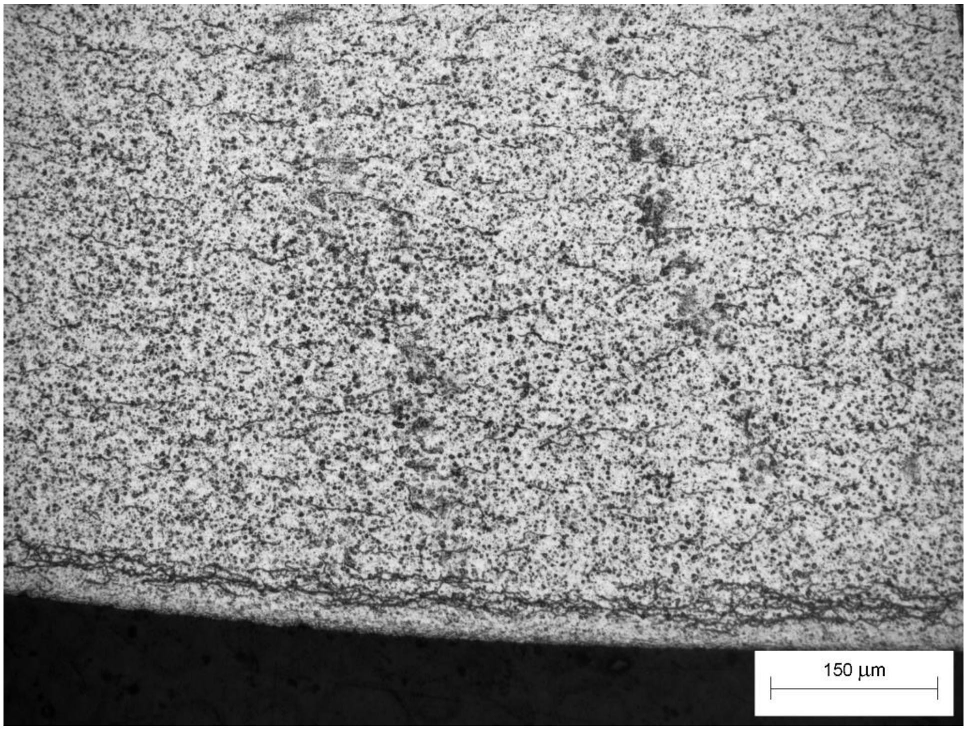
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	450	400	300	230	345	99	29%
Middle-Bot	540	290	490	390	428	111	26%



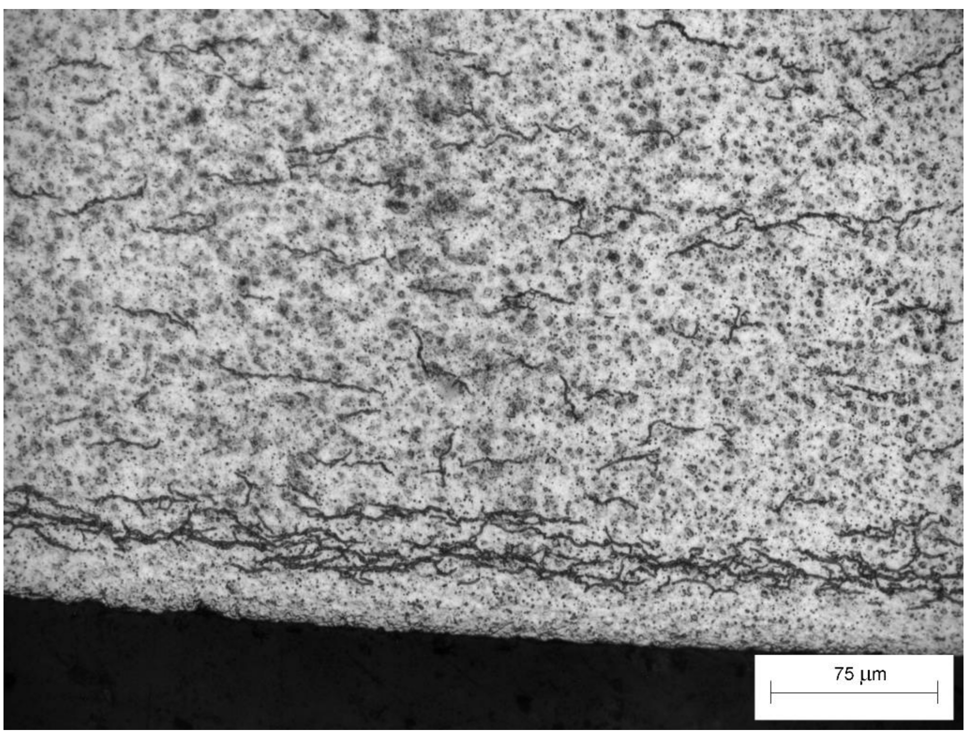




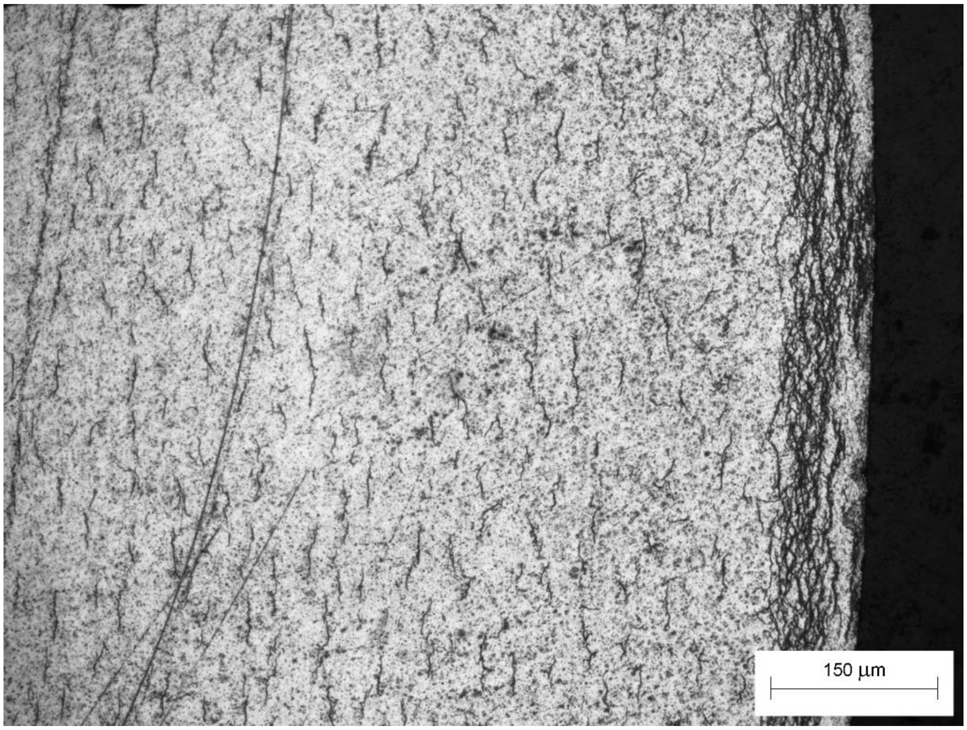
Images of Sample 19 After Hydriding and Mounted in Epoxy



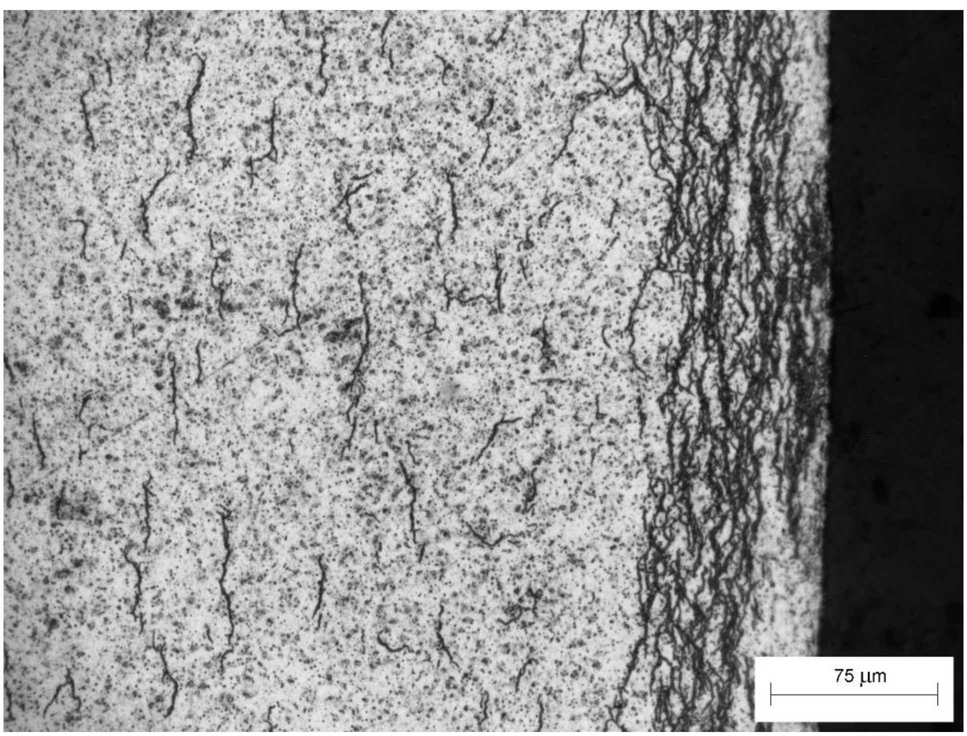
Sample 19 / Quadrant 1 / 100x Magnification



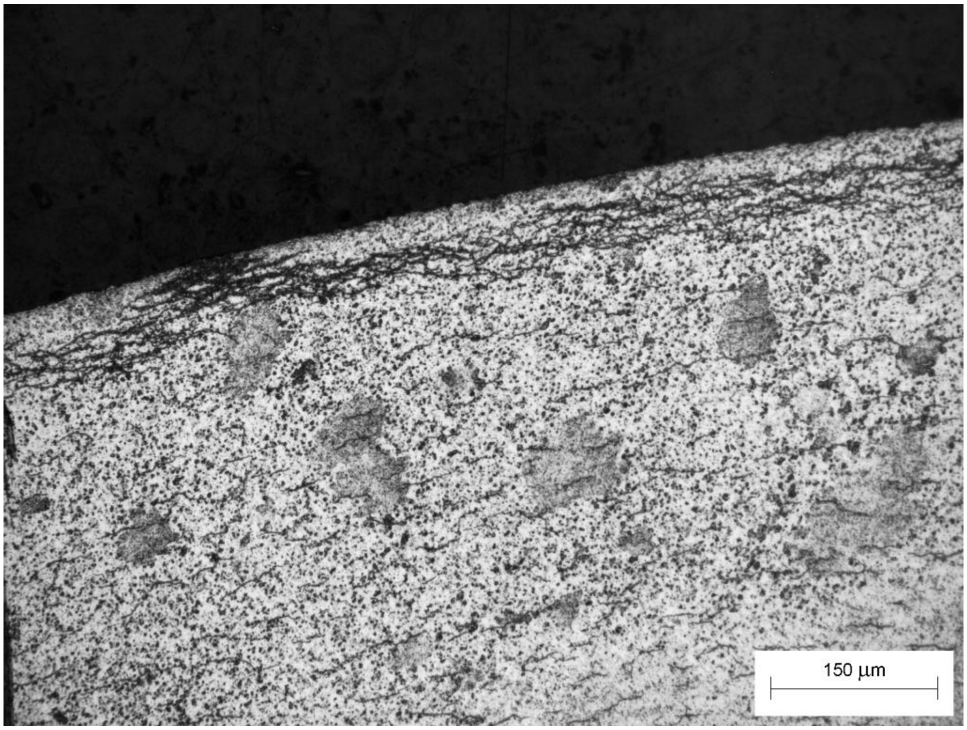
Sample 19 / Quadrant 1 / 200x Magnification



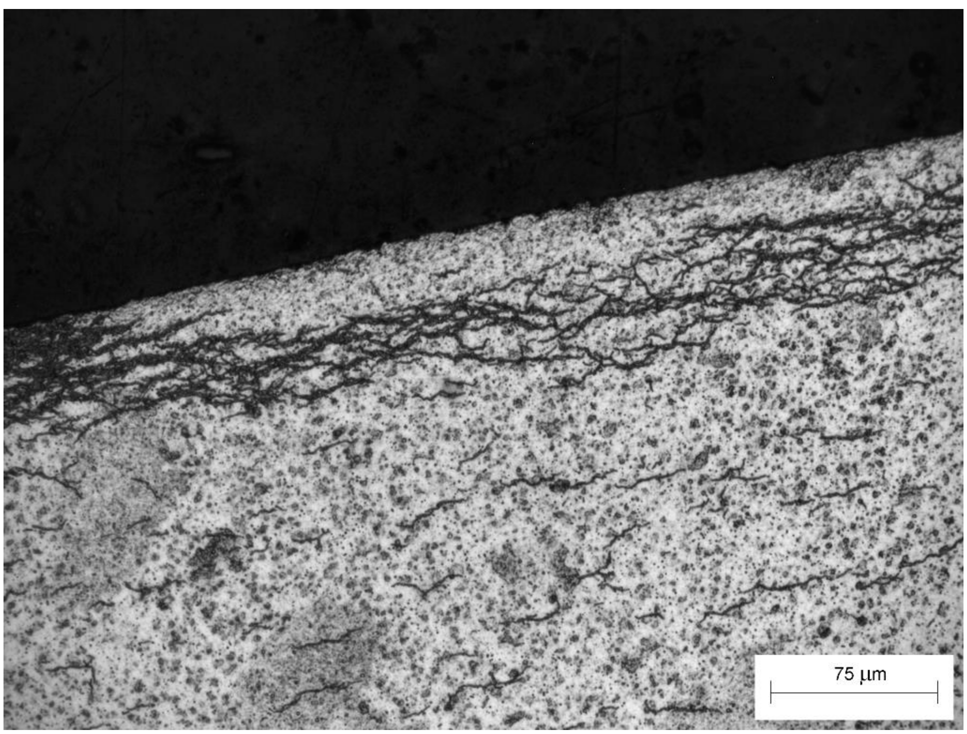
Sample 19 / Quadrant 2 / 100x Magnification



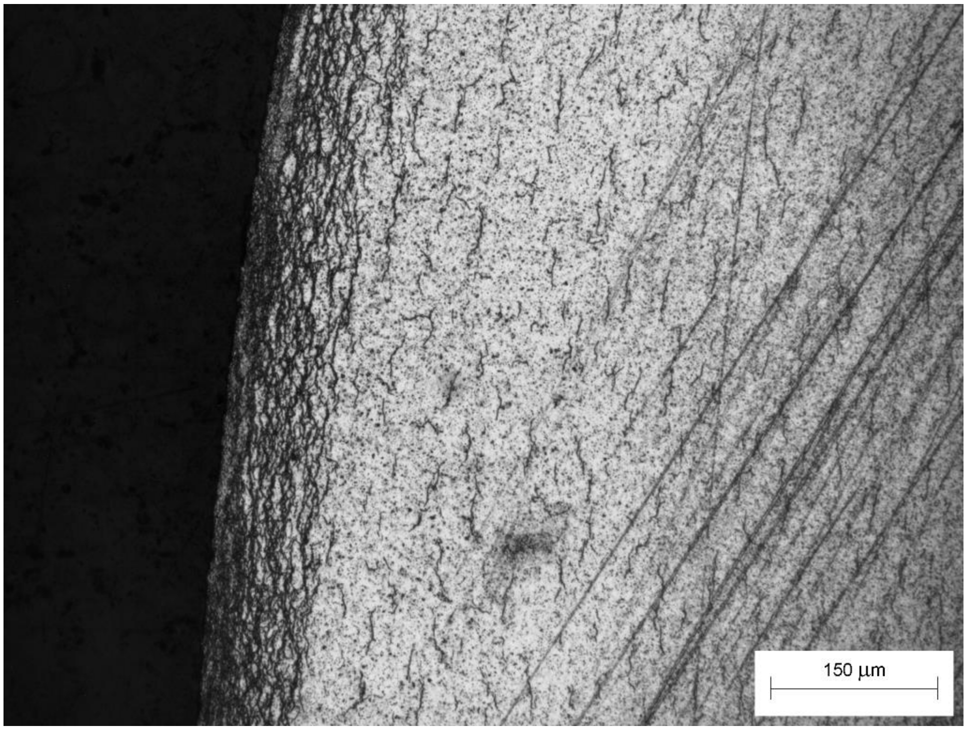
Sample 19 / Quadrant 2 / 200x Magnification



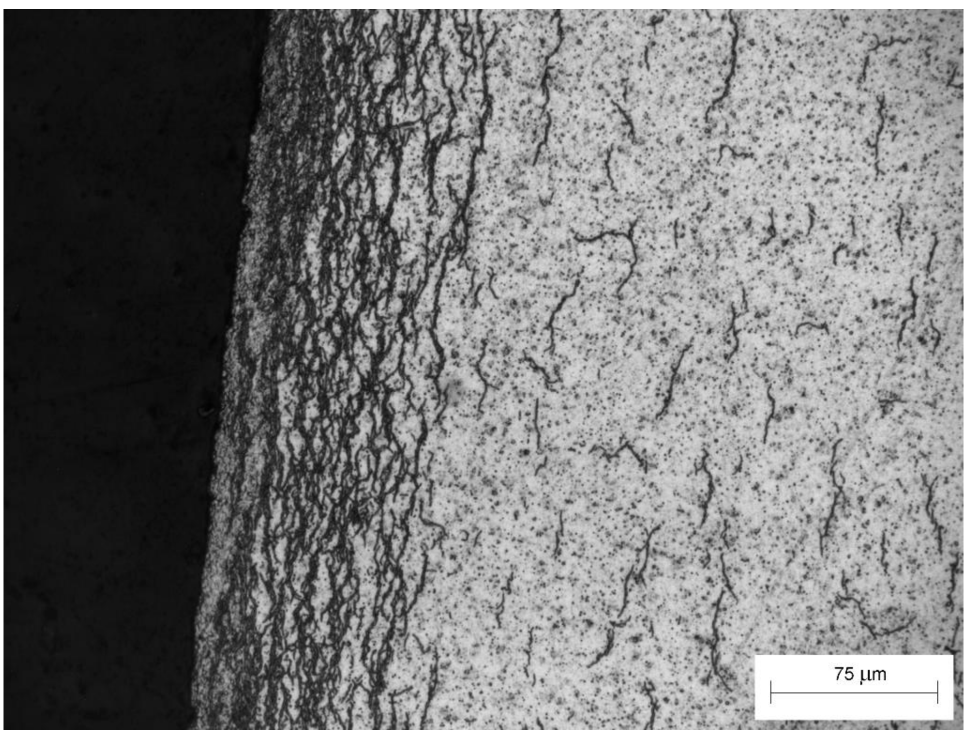
Sample 19 / Quadrant 3 / 100x Magnification



Sample 19 / Quadrant 3 / 200x Magnification



Sample 19 / Quadrant 4 / 100x Magnification



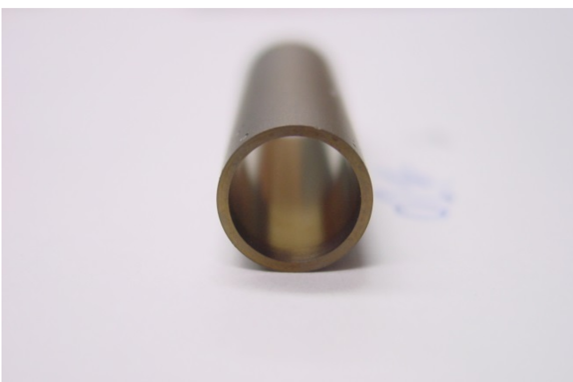
Sample 19 / Quadrant 4 / 200x Magnification

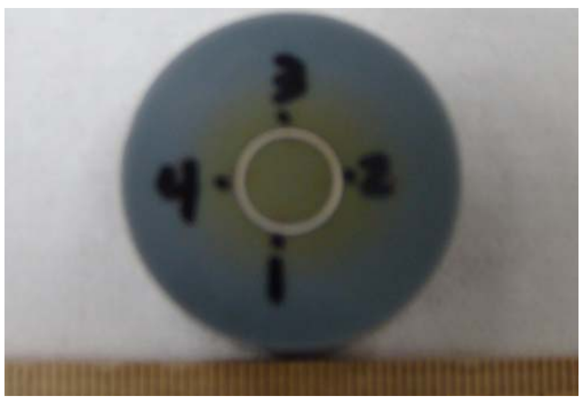
Sample ID: 20

Furnace: CAMCo
Location: Hung
Temperature: 280°C
OD Surface: Blasted
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

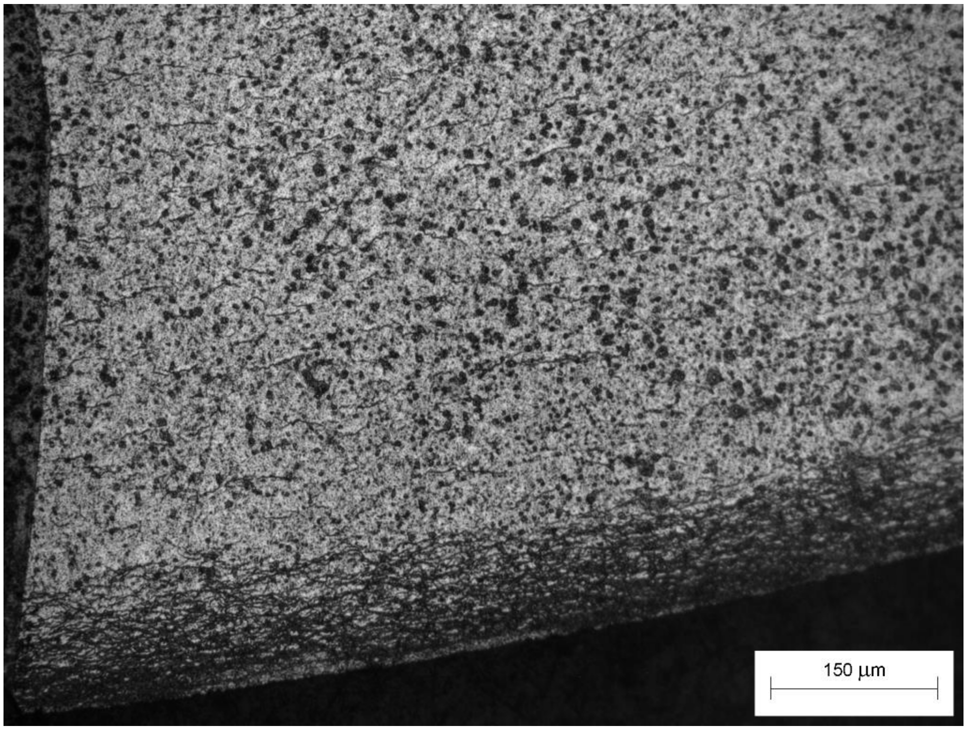
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	250	230	480	650	403	200	50%
Middle-Bot	390	380	250	400	355	70	20%

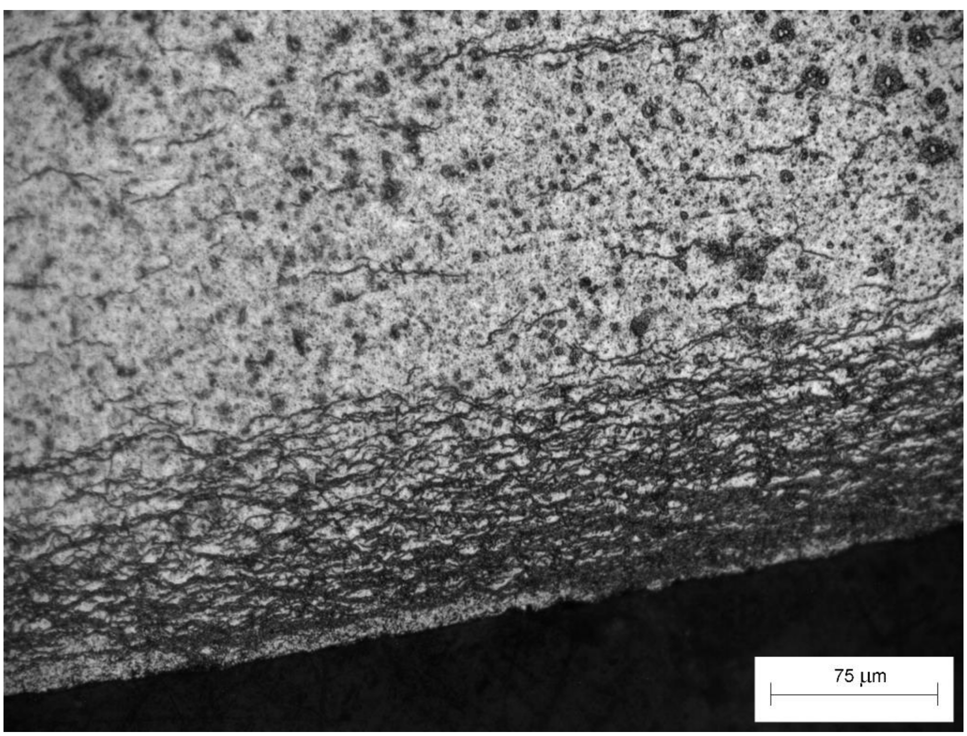




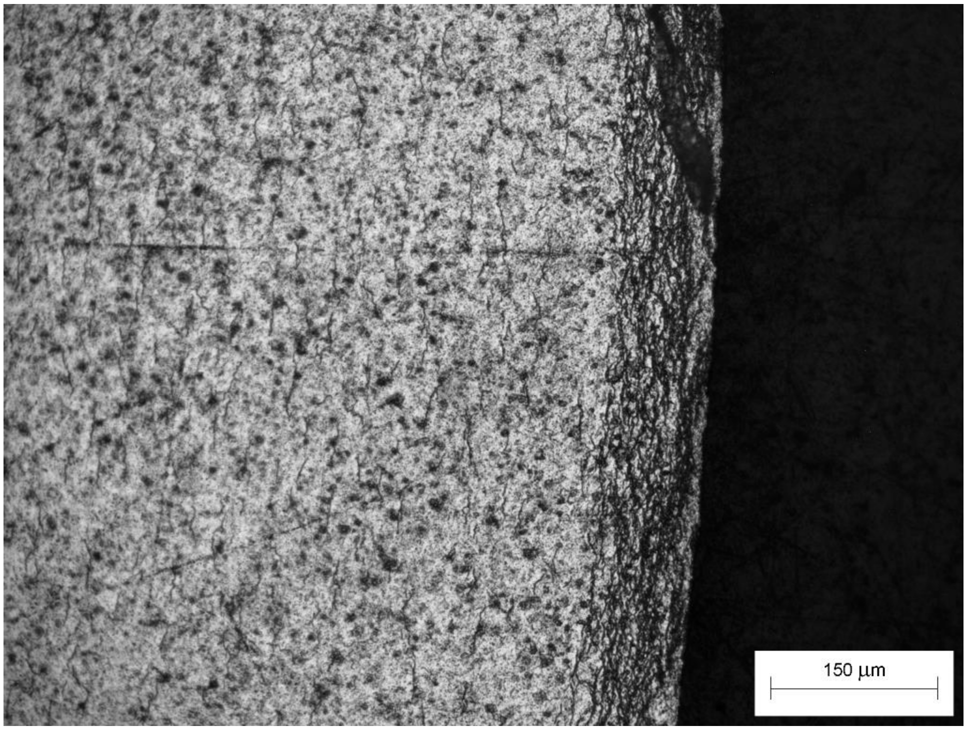
Images of Sample 20 After Hydriding and Mounted in Epoxy



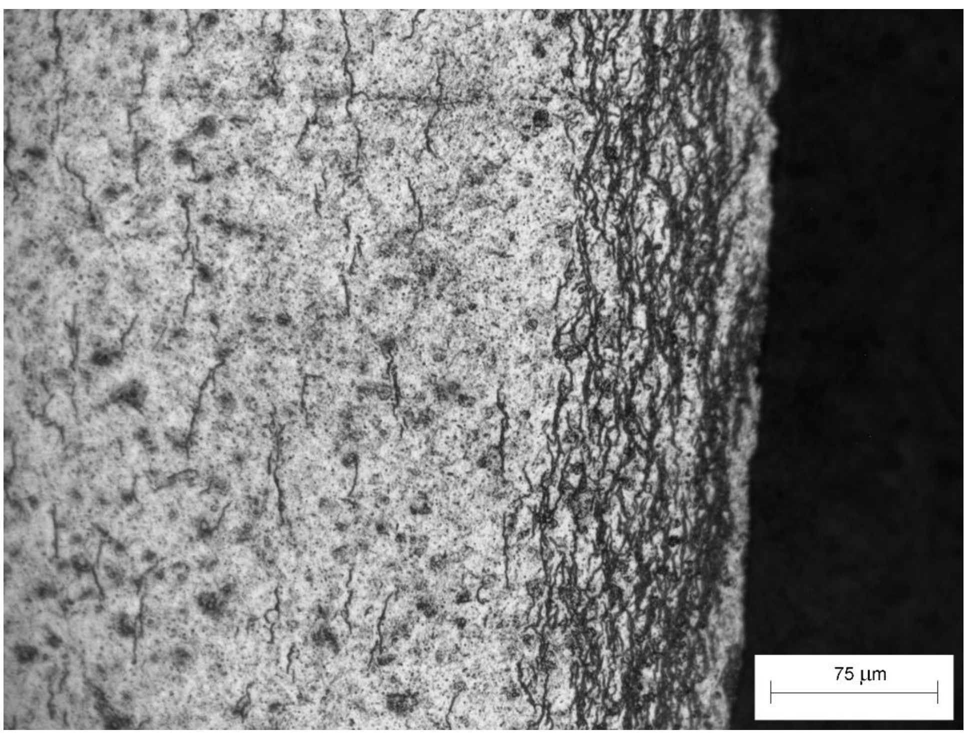
Sample 20 / Quadrant 1 / 100x Magnification



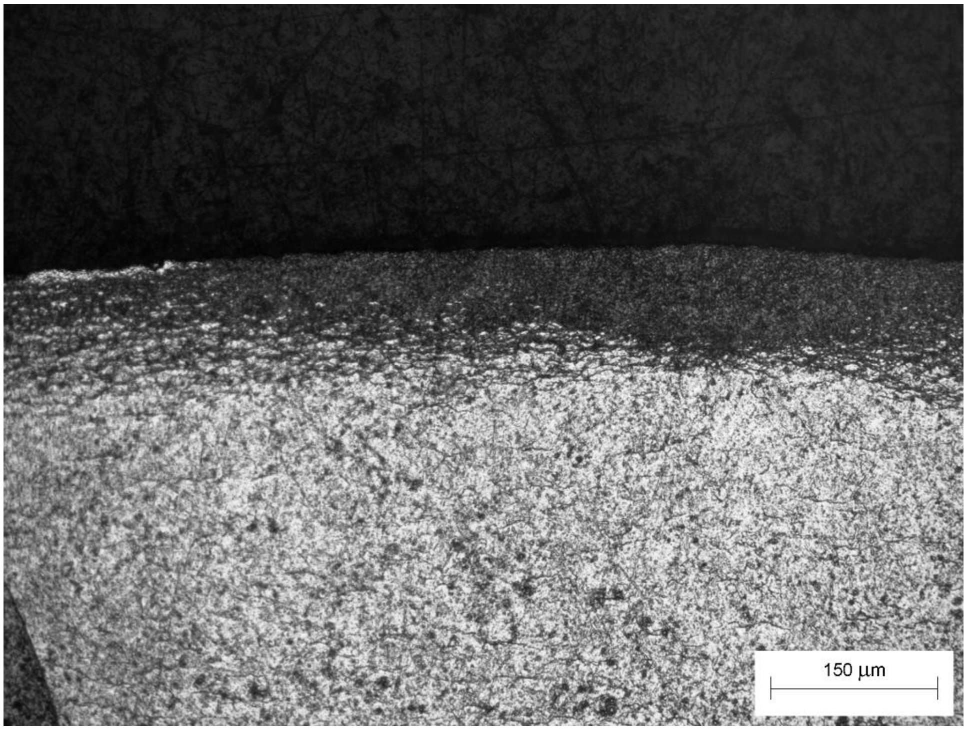
Sample 20 / Quadrant 1 / 200x Magnification



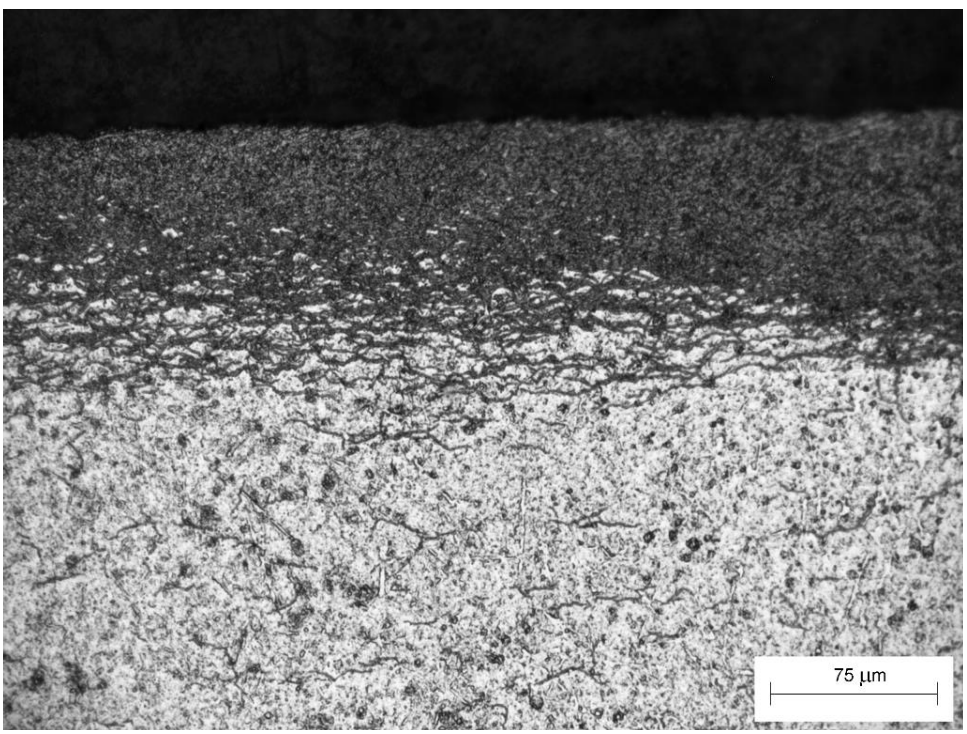
Sample 20 / Quadrant 2 / 100x Magnification



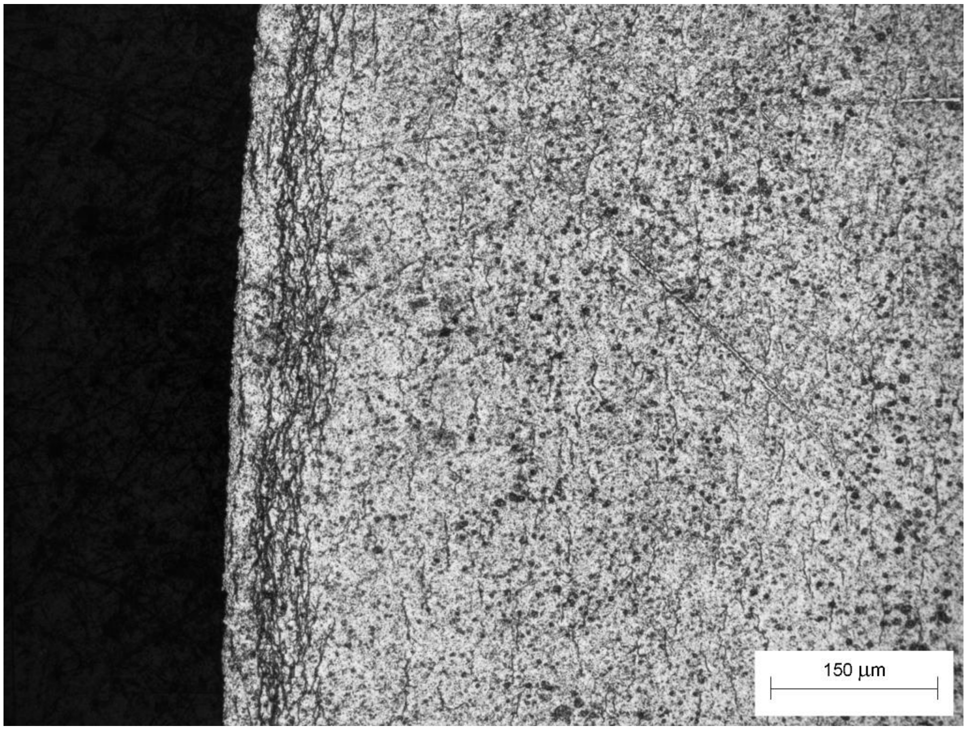
Sample 20 / Quadrant 2 / 200x Magnification



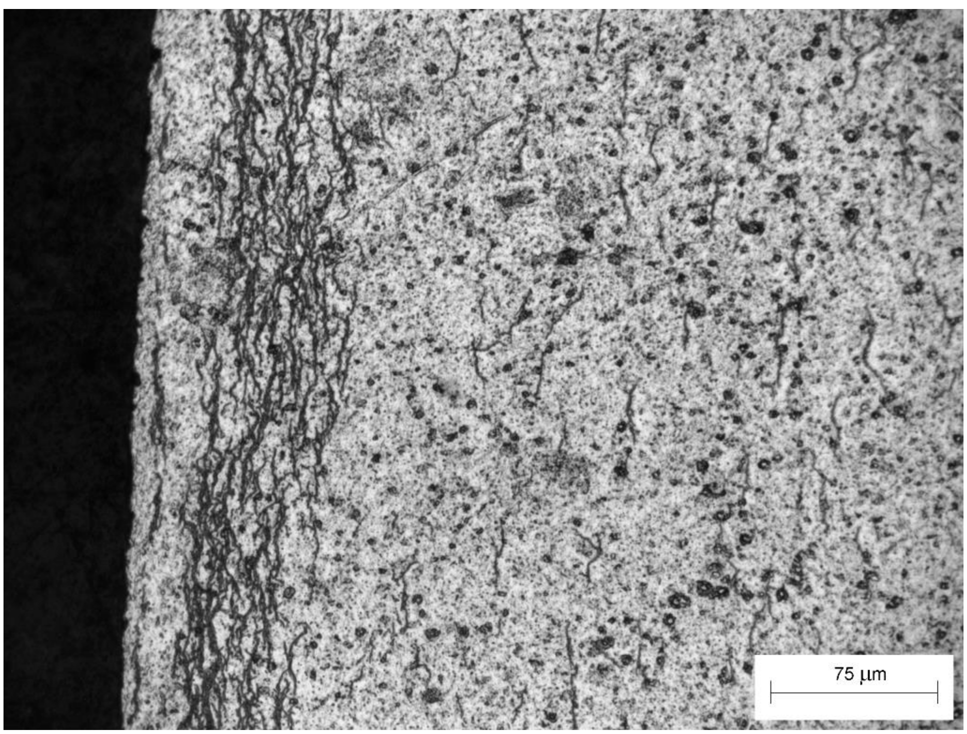
Sample 20 / Quadrant 3 / 100x Magnification



Sample 20 / Quadrant 3 / 200x Magnification



Sample 20 / Quadrant 4 / 100x Magnification



Sample 20 / Quadrant 4 / 200x Magnification

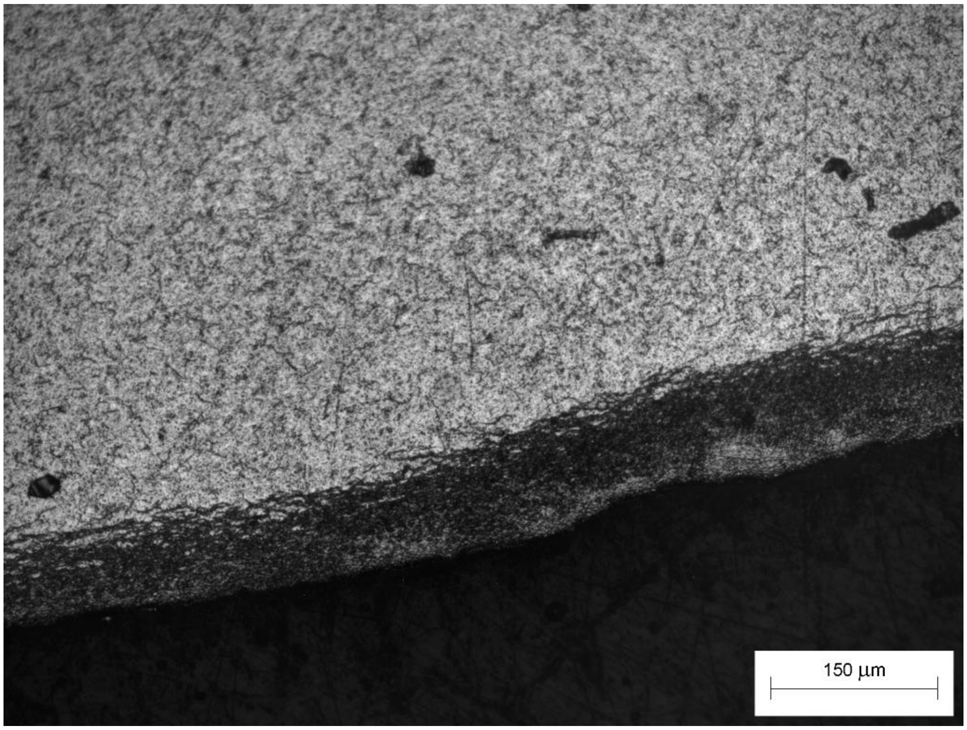
Sample ID: 21

Furnace:	CAMCo
Location:	Hung
Temperature:	270°C
OD Surface:	Blasted, 24 Hr Hold in Air
Outside Diameter:	0.451 in (11.5 mm)
Wall Thickness:	0.036 in (0.90mm)

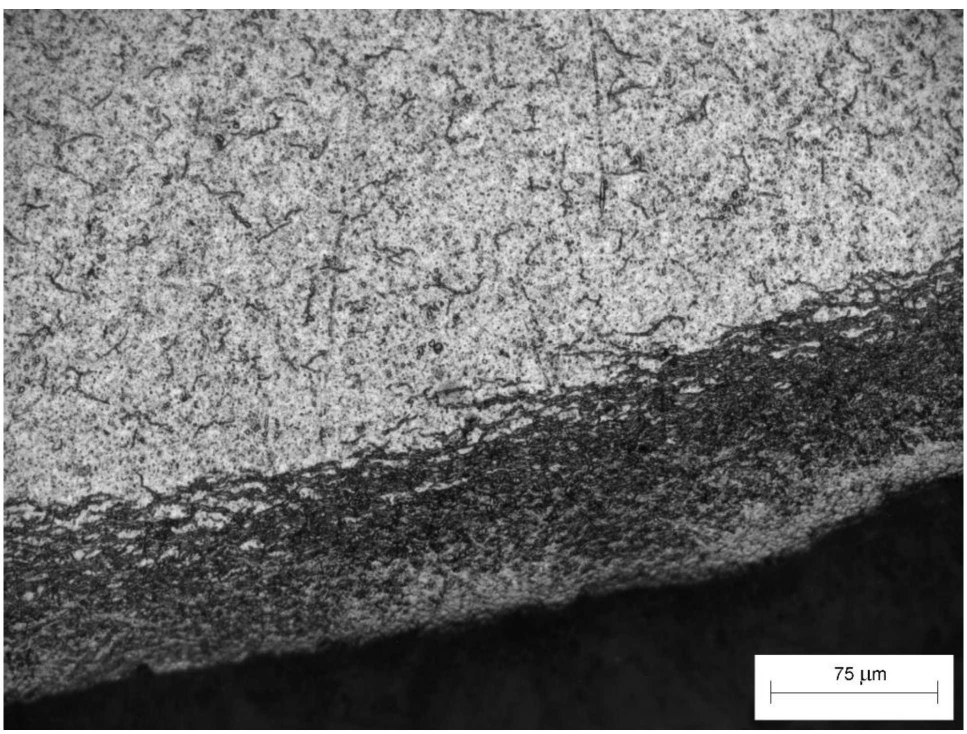
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	2300	1100	1800	2000	1800	510	28%
Middle-Bot	1200	1800	390	980	1093	583	53%

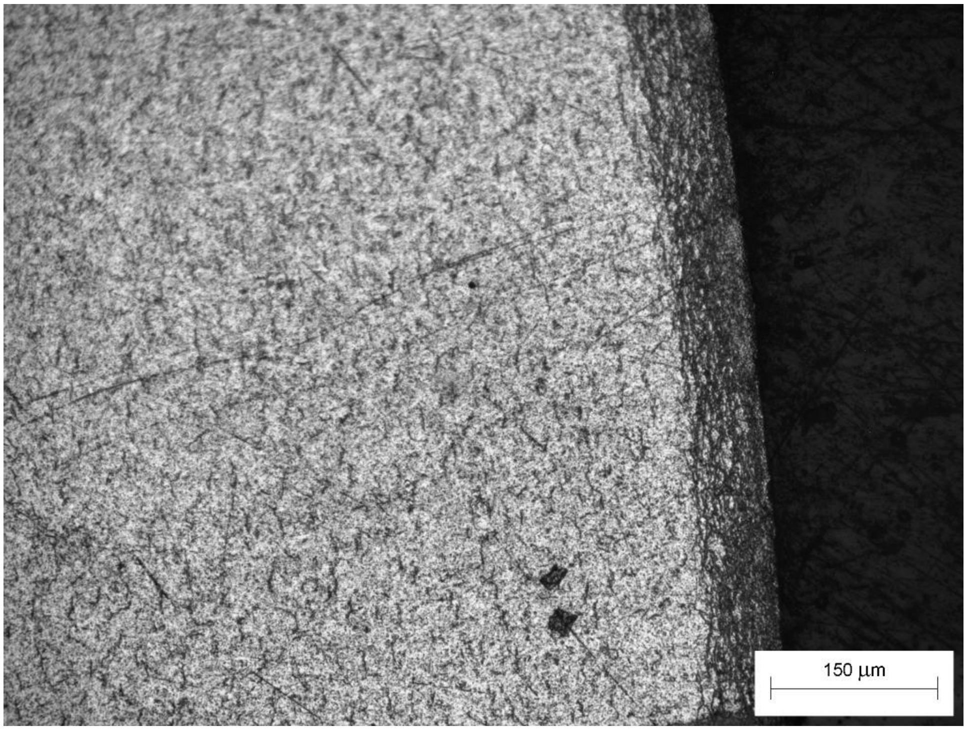
Images of Sample 21 After Hydriding and Mounted in Epoxy



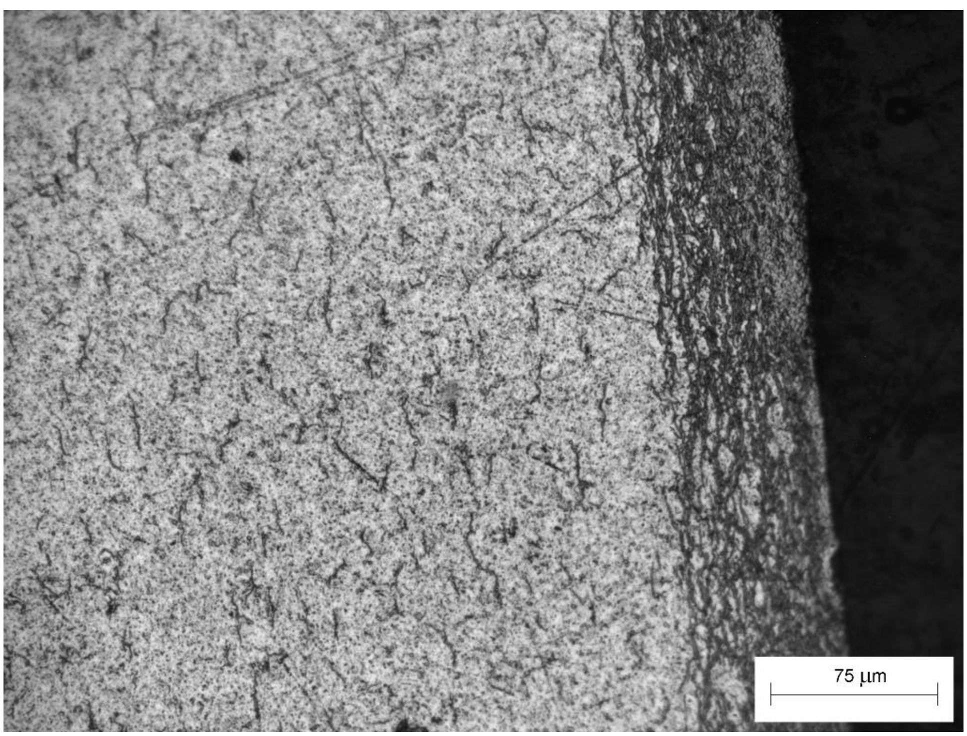
Sample 21 / Quadrant 1 / 100x Magnification



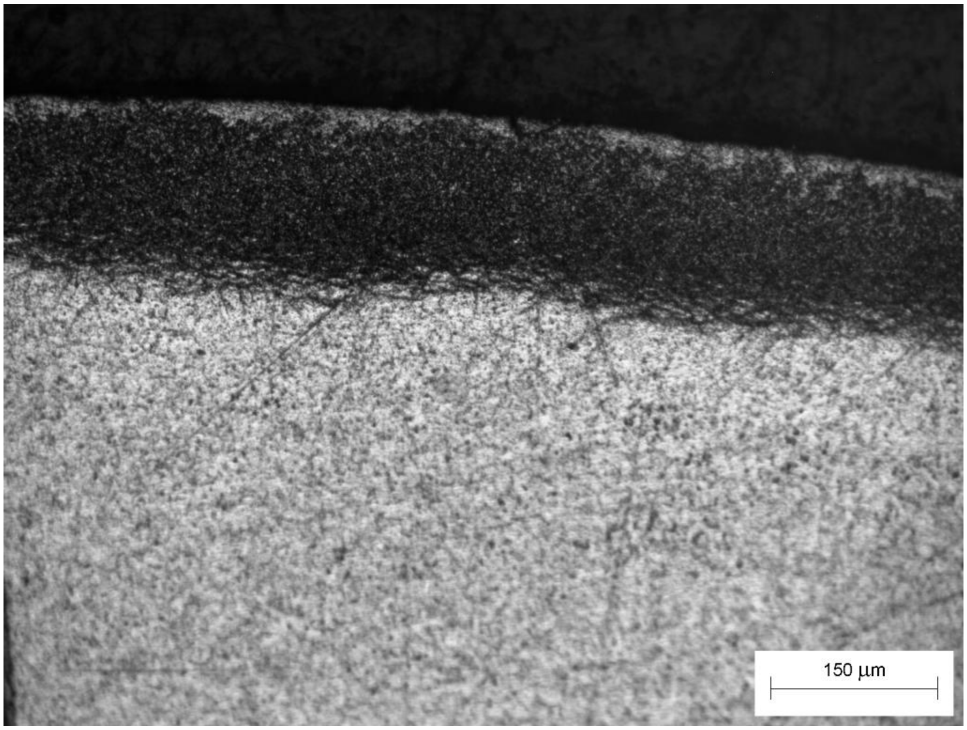
Sample 21 / Quadrant 1 / 200x Magnification



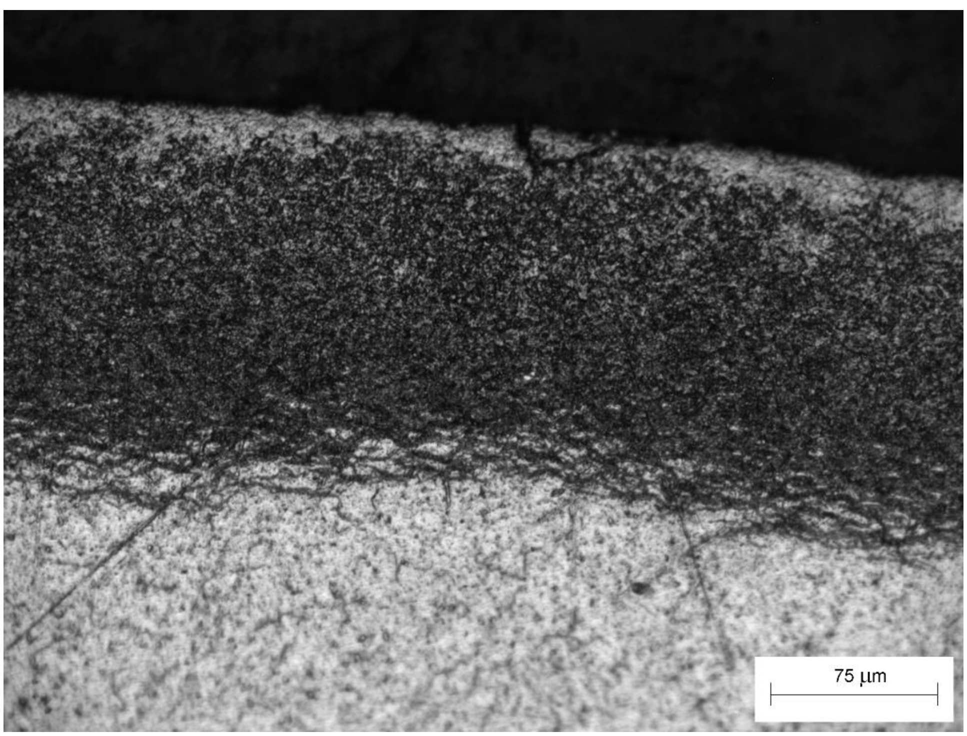
Sample 21 / Quadrant 2 / 100x Magnification



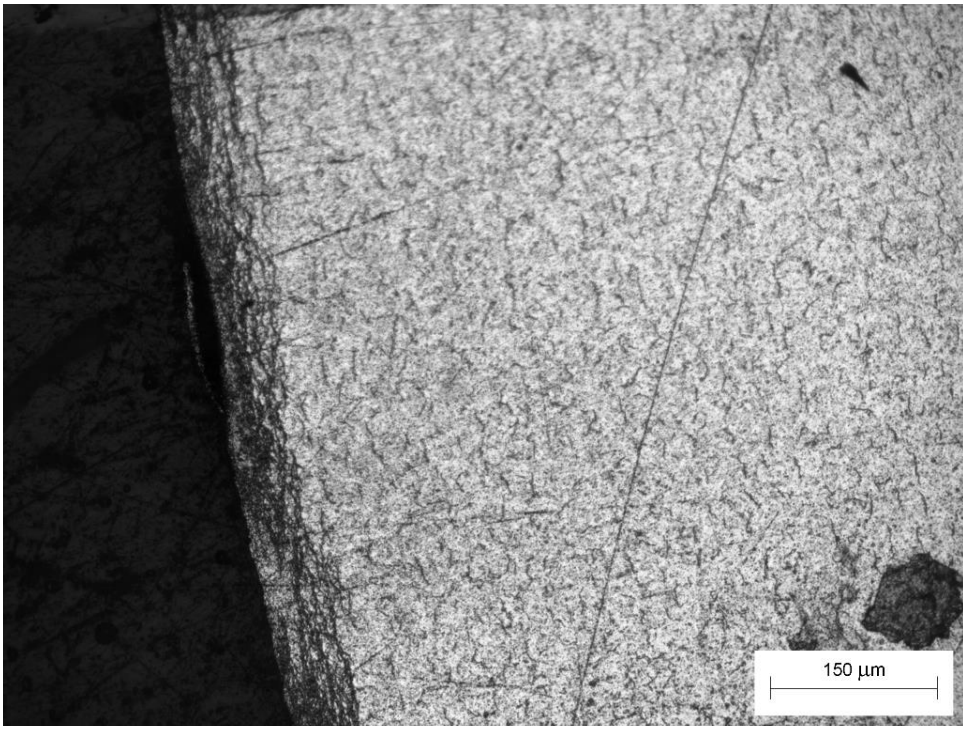
Sample 21 / Quadrant 2 / 200x Magnification



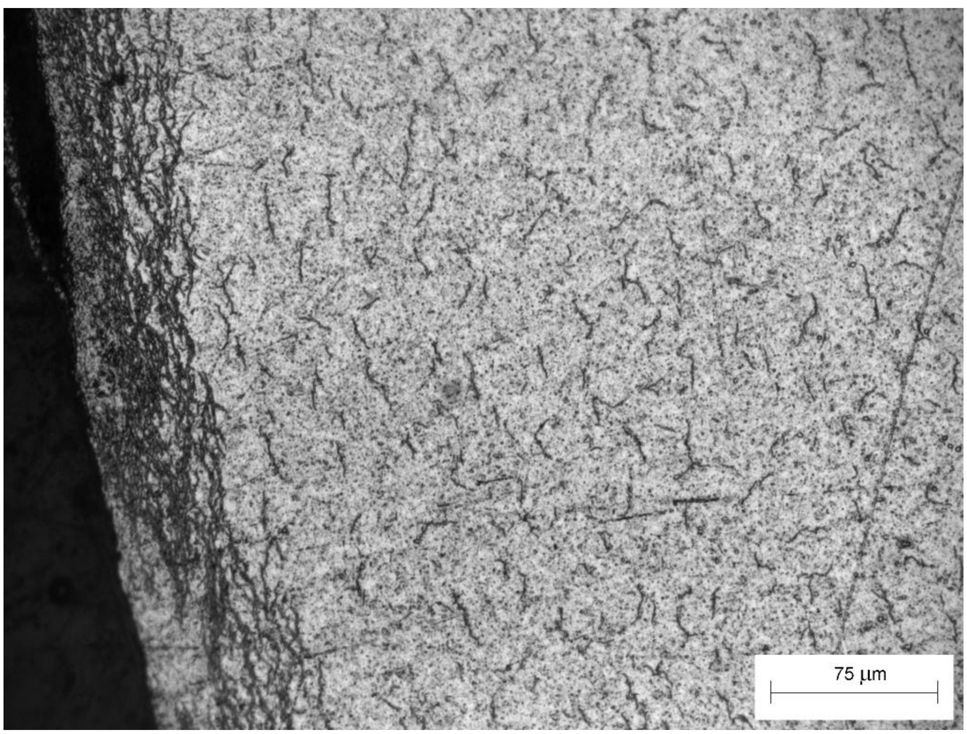
Sample 21 / Quadrant 3 / 100x Magnification



Sample 21 / Quadrant 3 / 200x Magnification



Sample 21 / Quadrant 4 / 100x Magnification



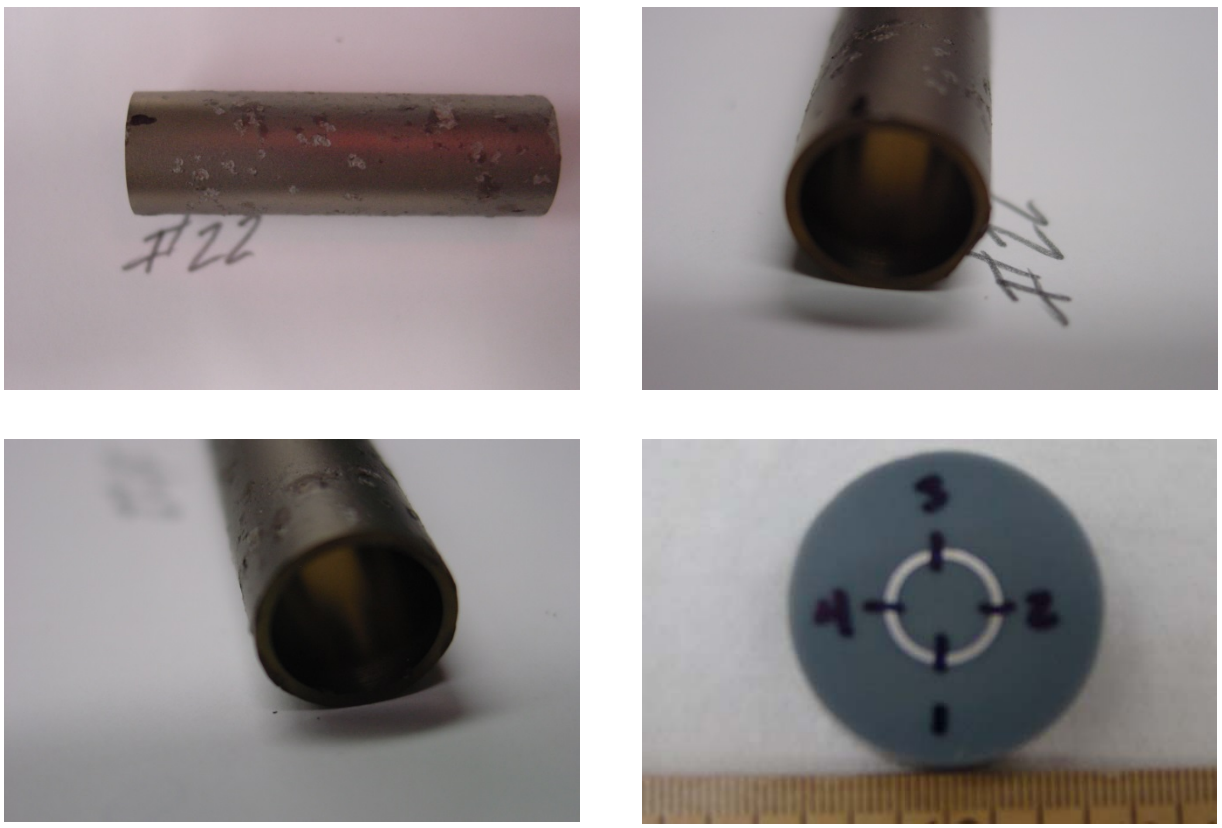
Sample 21 / Quadrant 4 / 200x Magnification

Sample ID: 22

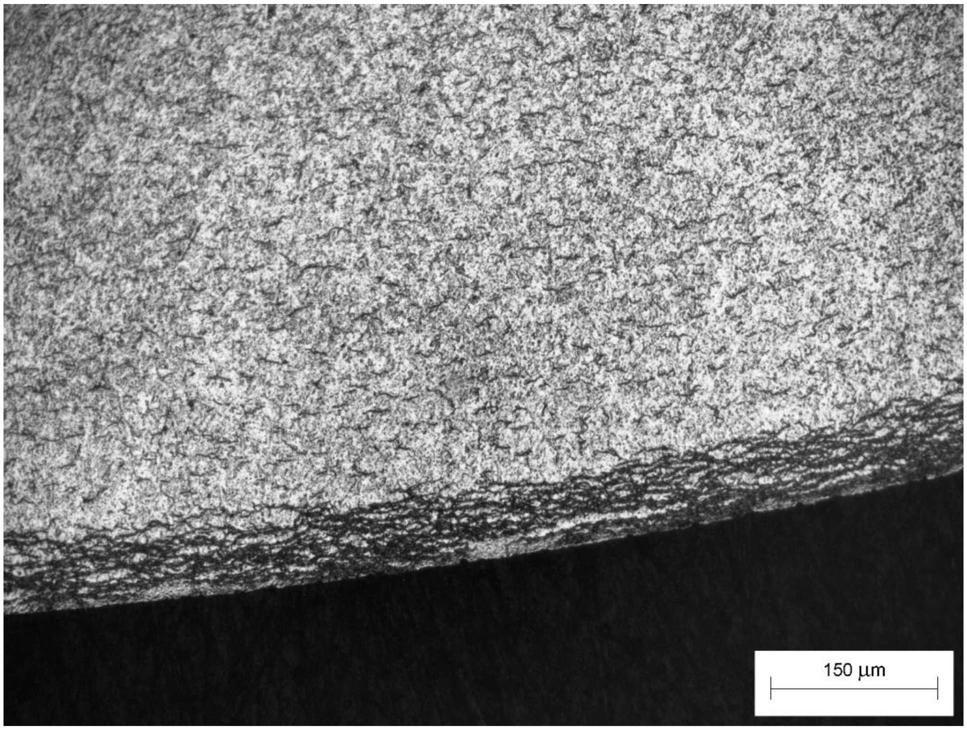
Furnace: CAMCo
Location: Hung
Temperature: 270°C
OD Surface: Blasted
Outside Diameter: 0.451 in (11.5 mm)
Wall Thickness: 0.036 in (0.90mm)

Hydrogen Concentration Results

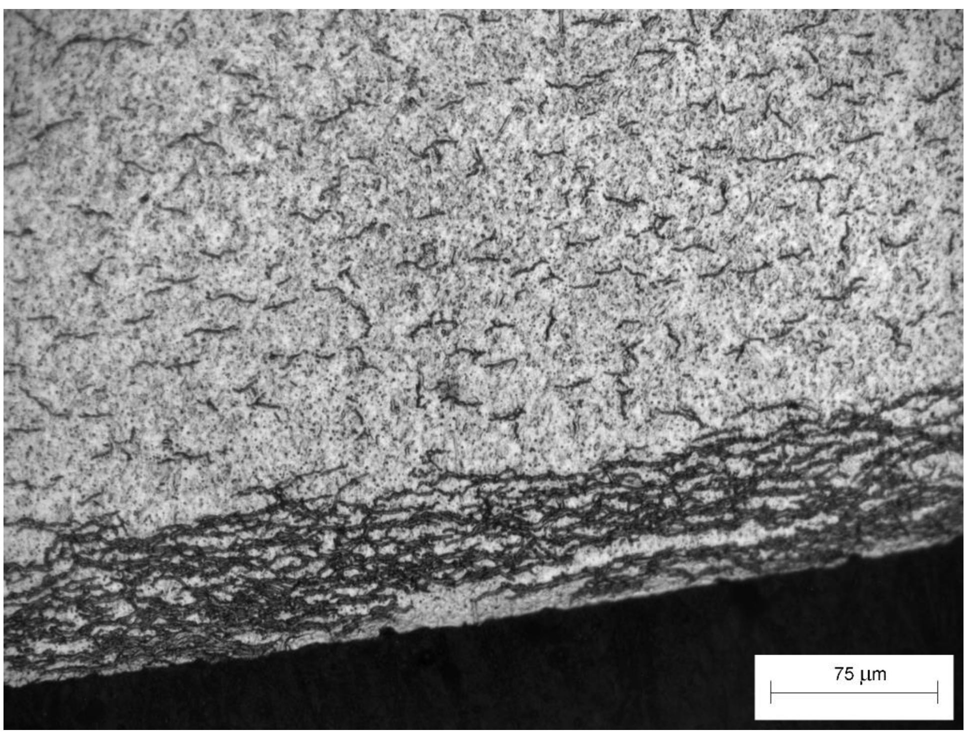
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-Top	670	560	410	690	583	128	22%
Middle-Bot	1400	1800	1100	640	1235	489	40%



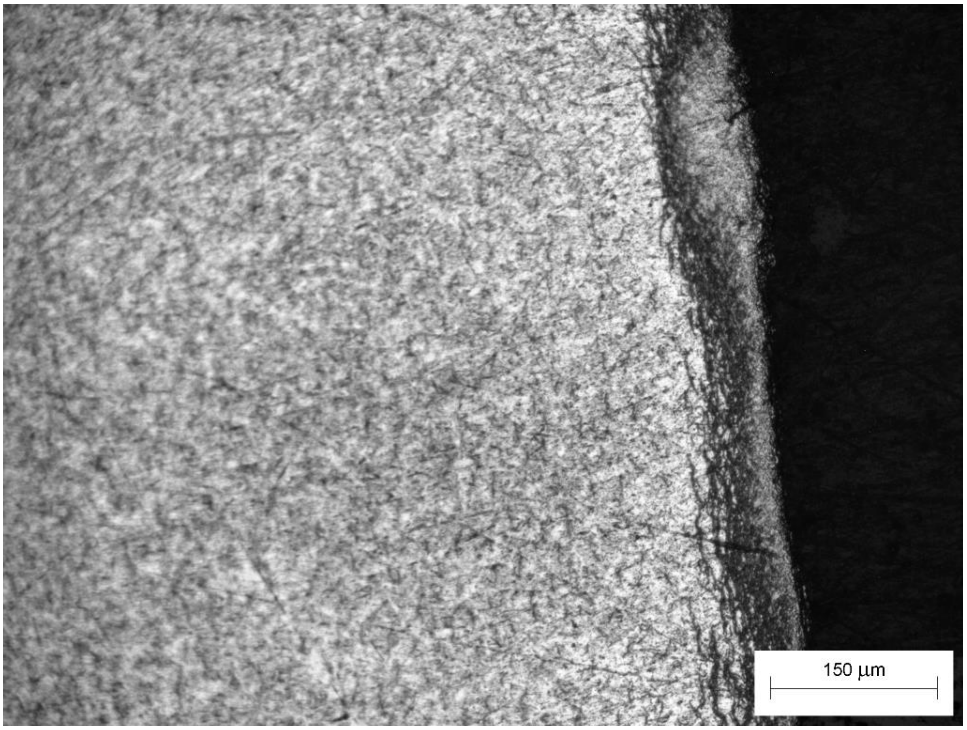
Images of Sample 22 After Hydriding and Mounted in Epoxy



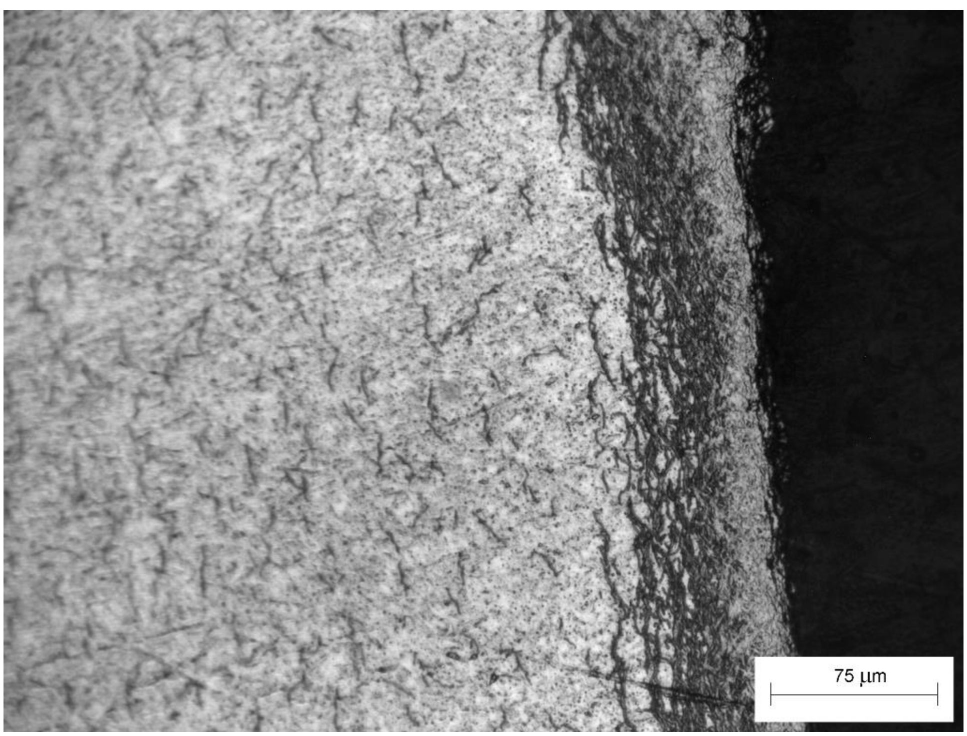
Sample 22 / Quadrant 1 / 100x Magnification



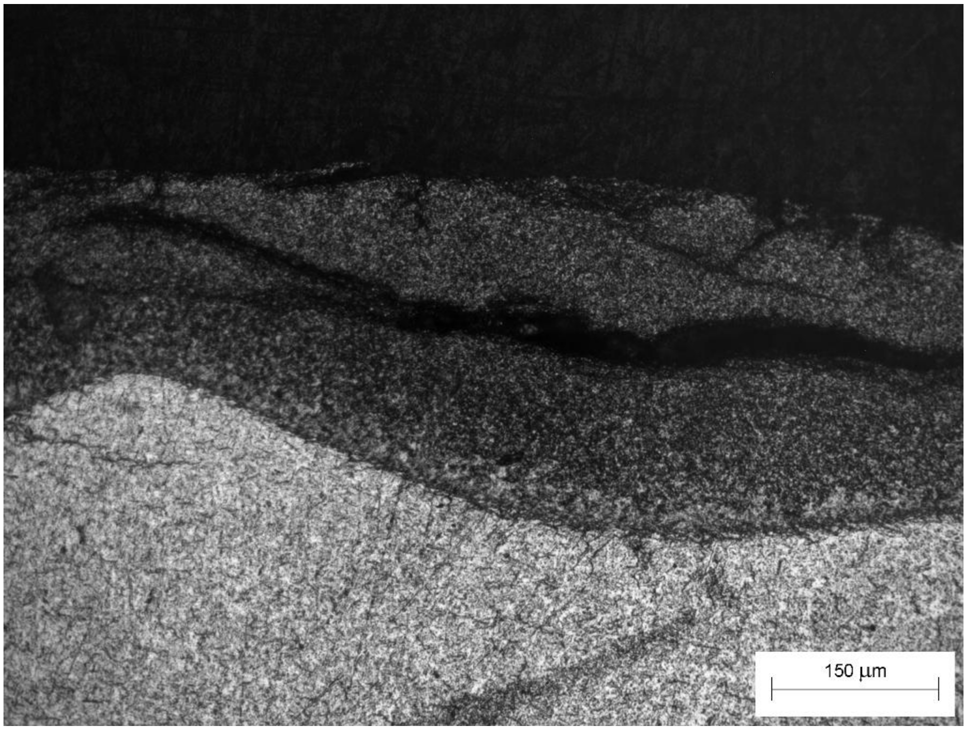
Sample 22 / Quadrant 1 / 200x Magnification



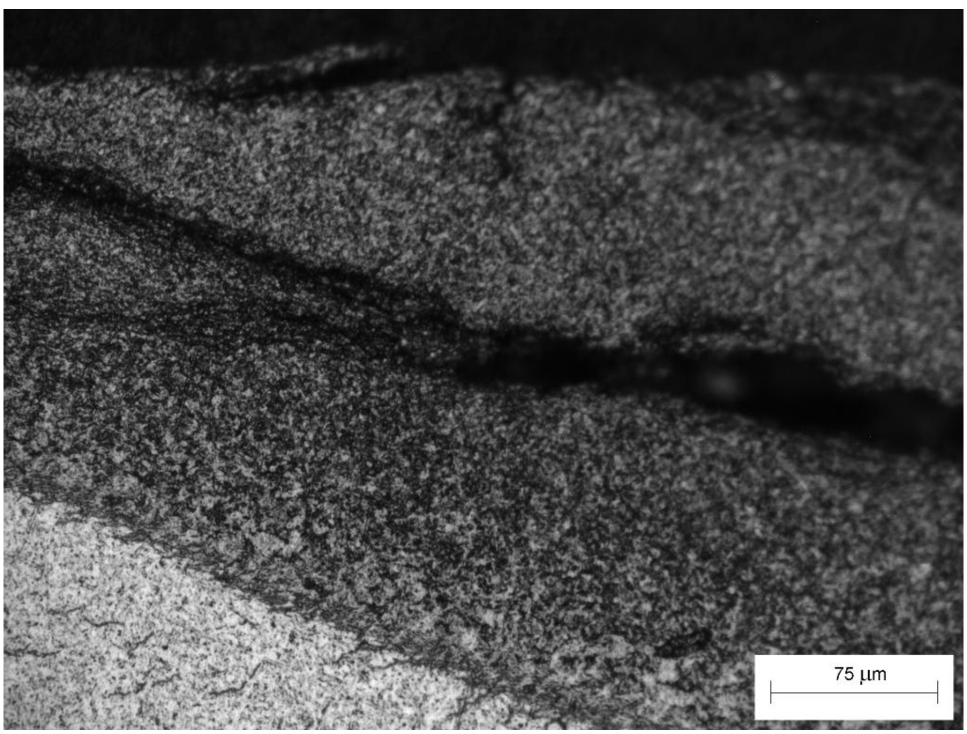
Sample 22 / Quadrant 2 / 100x Magnification



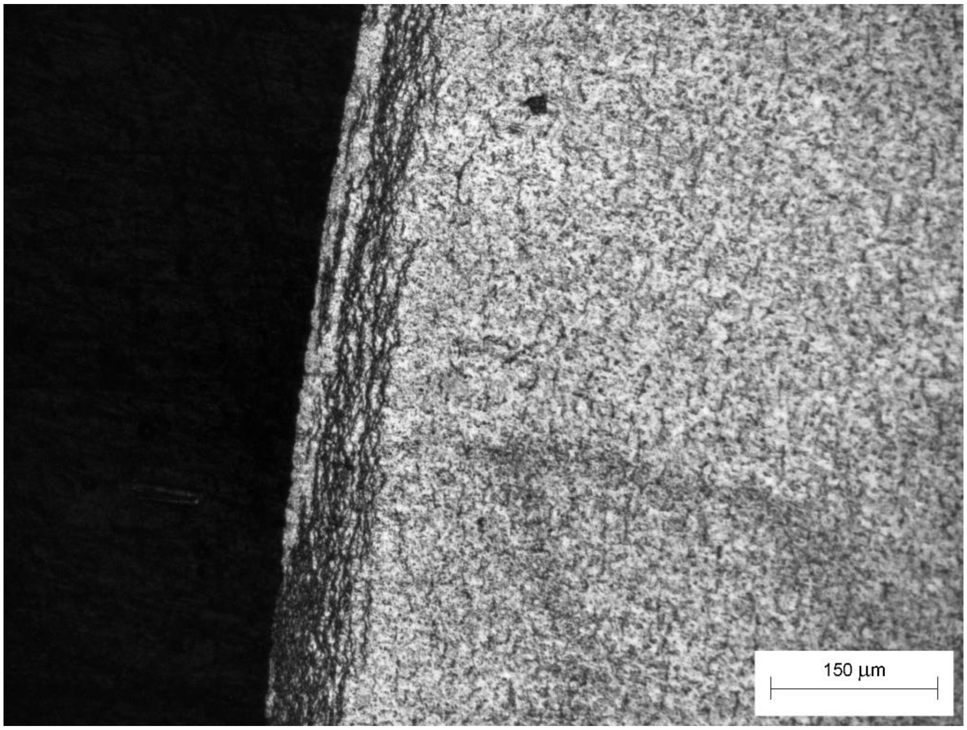
Sample 22 / Quadrant 2 / 200x Magnification



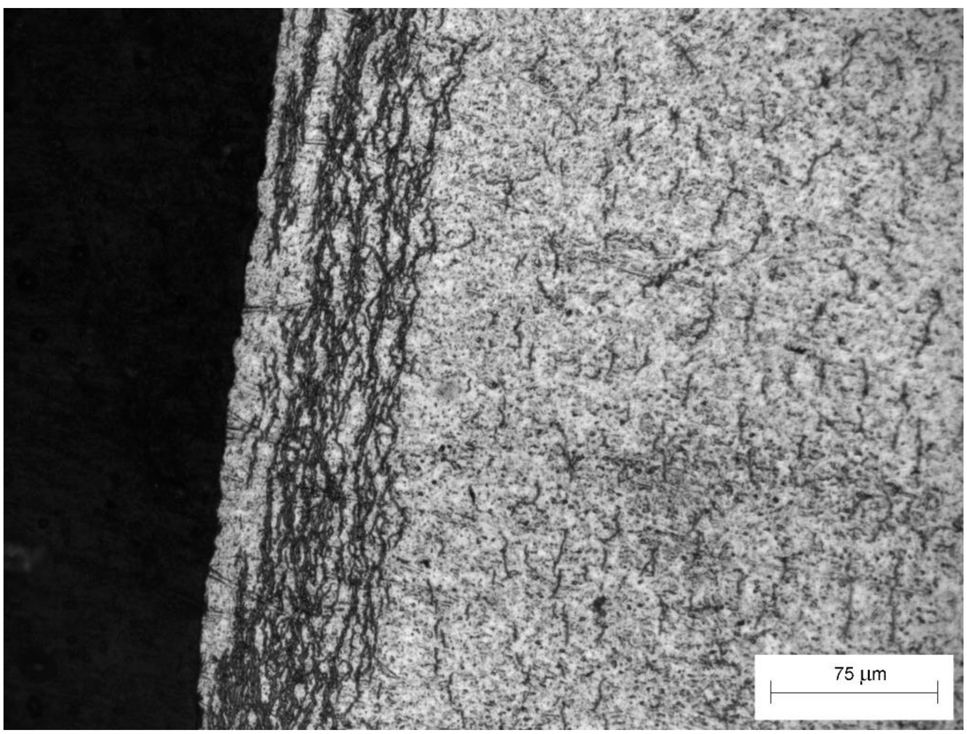
Sample 22 / Quadrant 3 / 100x Magnification



Sample 22 / Quadrant 3 / 200x Magnification



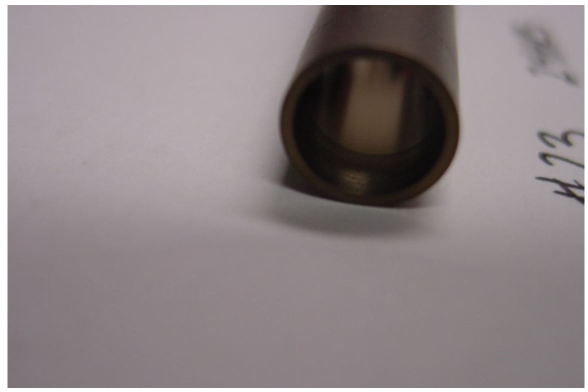
Sample 22 / Quadrant 4 / 100x Magnification



Sample 22 / Quadrant 4 / 200x Magnification

Sample ID: 23

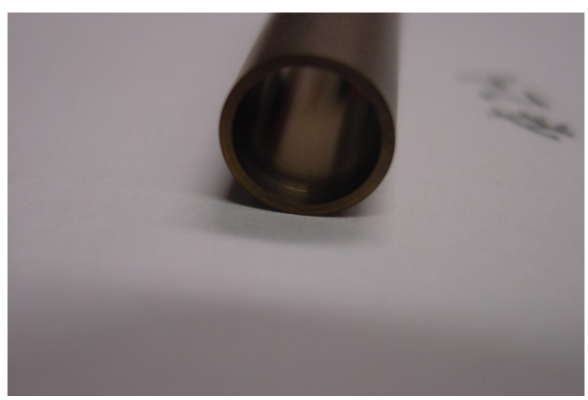
Furnace:	Thermolyne
Location:	Upright – tipped over
Temperature:	270°C
OD Surface:	Blasted, 24 hr Wait in Air
Outside Diameter:	0.451 in (11.5 mm)
Wall Thickness:	0.036 in (0.90mm)



Images of Sample 23 After Hydriding; No metallography or hydrogen analysis performed.

Sample ID: 24

Furnace:	Thermolyne
Location:	Upright – tipped over
Temperature:	270°C
OD Surface:	Blasted, 24 hr
Outside Diameter:	0.451 in (11.5 mm)
Wall Thickness:	0.036 in (0.90mm)



Images of Sample 15 After Hydriding; No metallography or hydrogen analysis performed.

Sample ID: 25

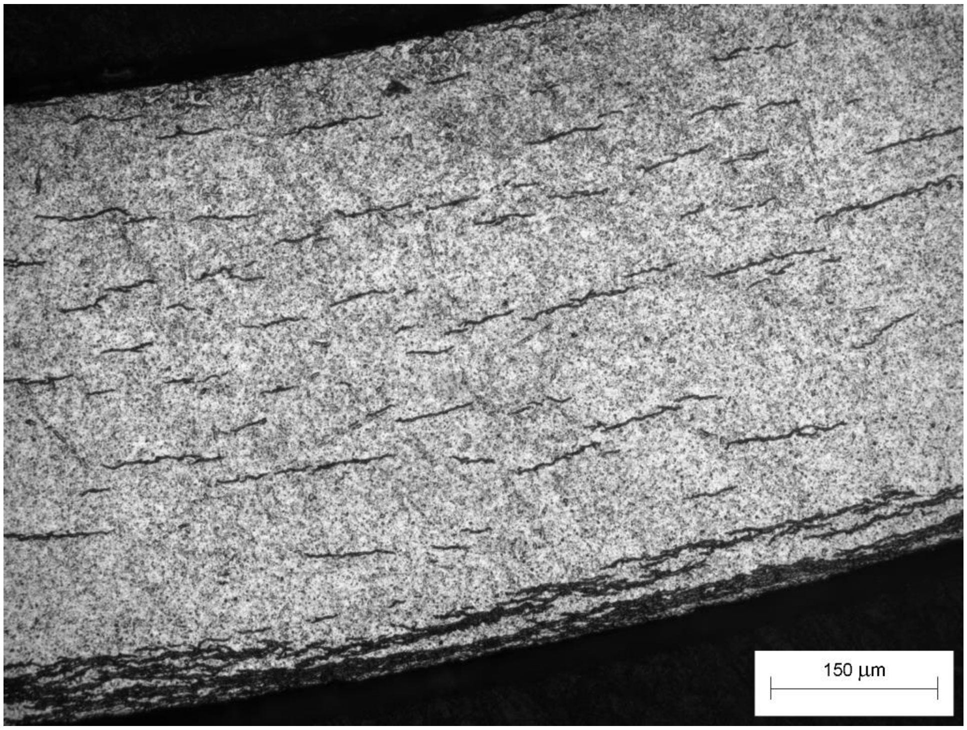
Furnace: Thermolyne
Location: Upright
Temperature: 270°C
OD Surface: Blasted, 24 hr Wait in Air
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

Hydrogen Concentration Results

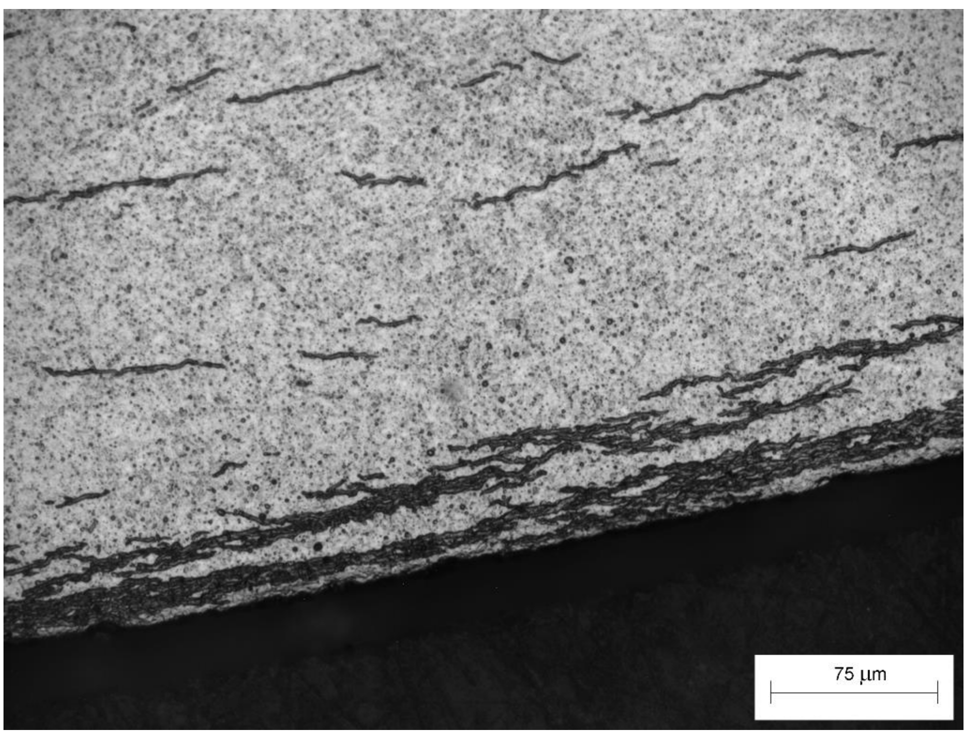
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	140	120	280	260	200	82	41%
Middle-2	250	160	250	230	223	43	19%



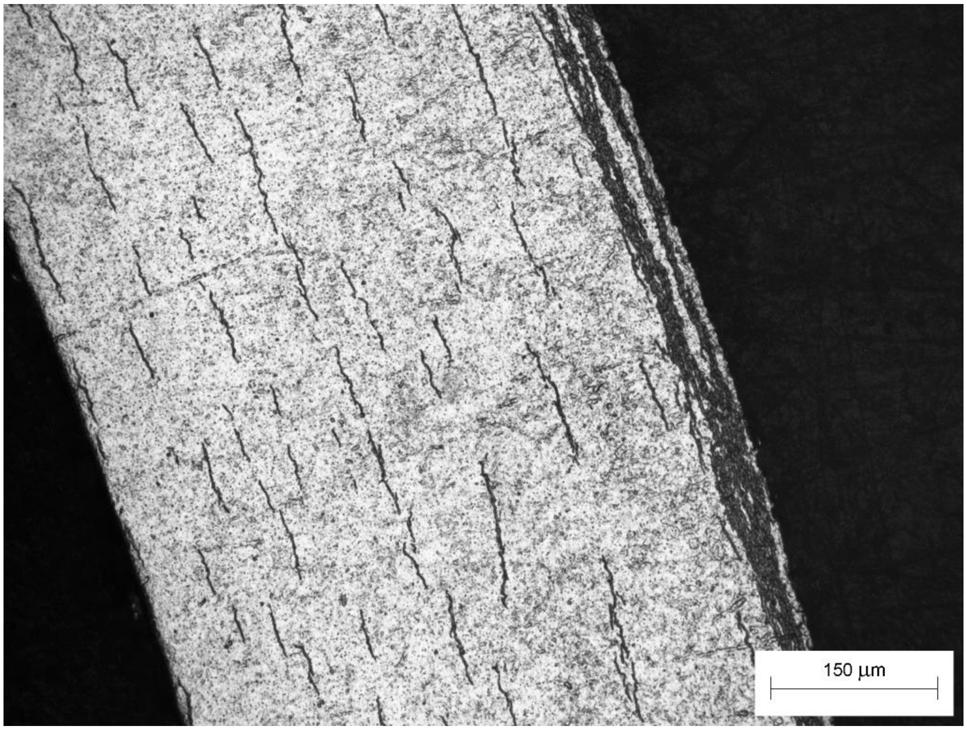
Images of Sample 25 After Hydriding and Mounted in Epoxy



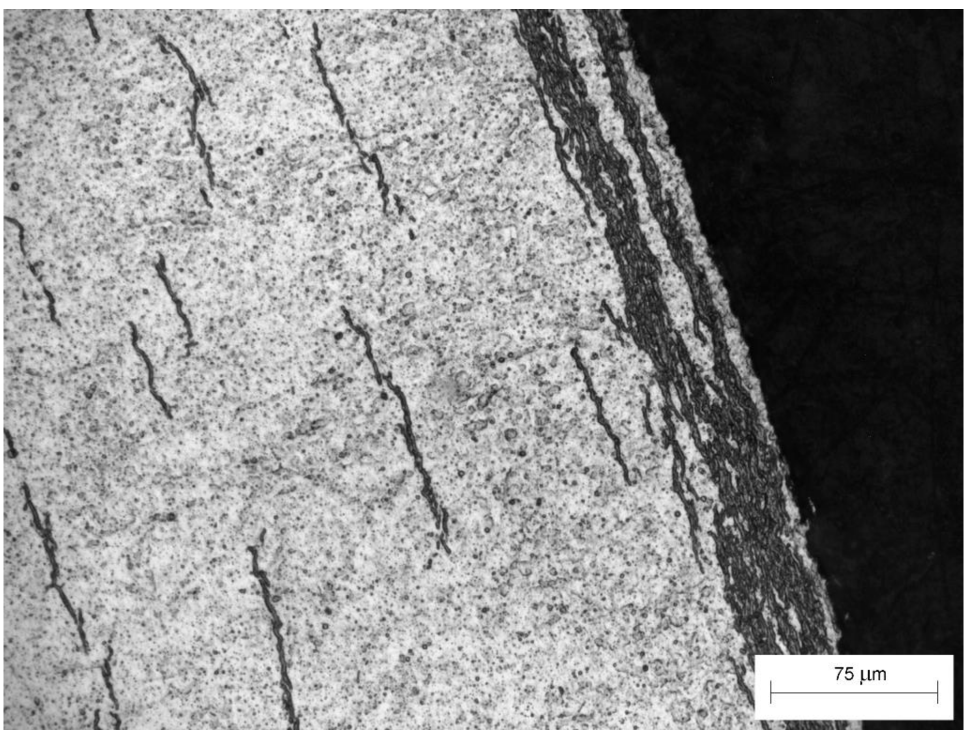
Sample 25 / Quadrant 1 / 100x Magnification



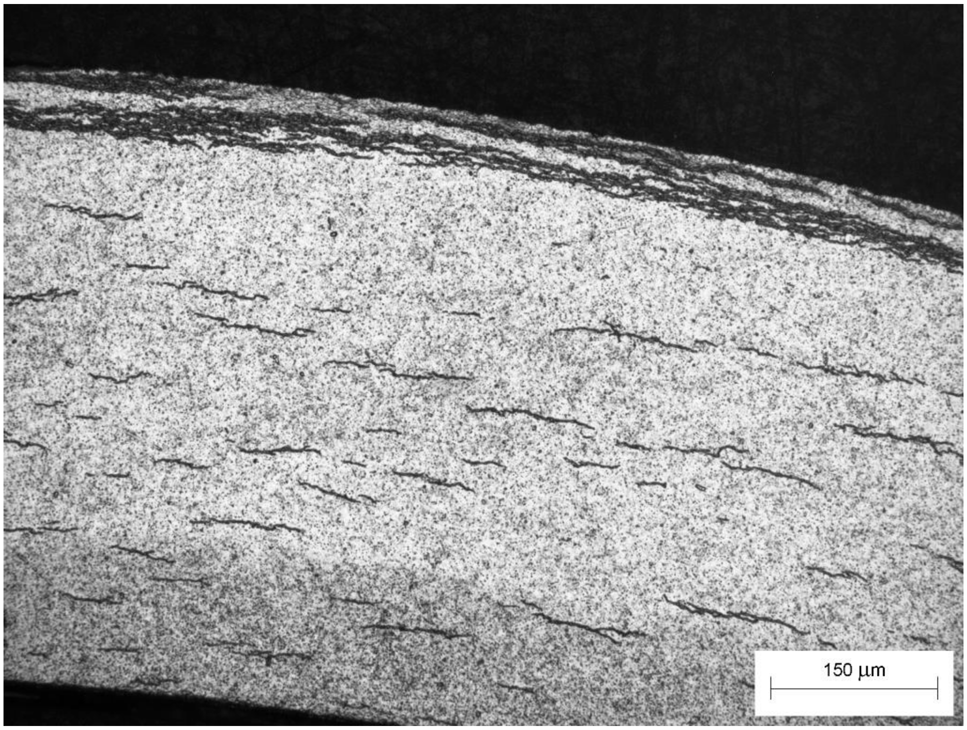
Sample 25 / Quadrant 1 / 200x Magnification



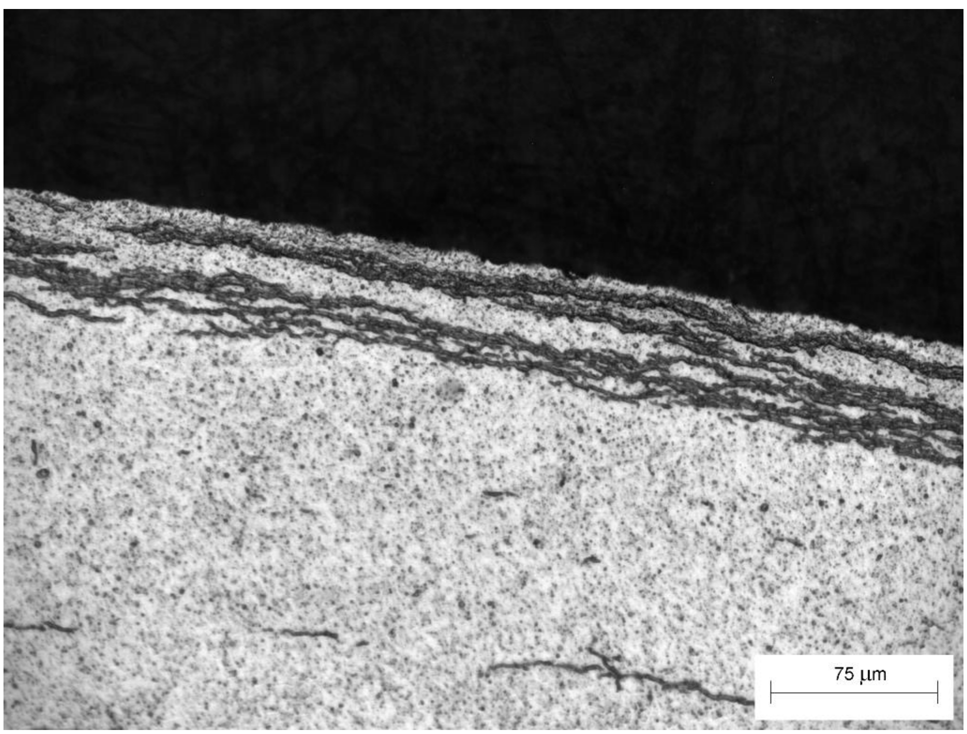
Sample 25 / Quadrant 2 / 100x Magnification



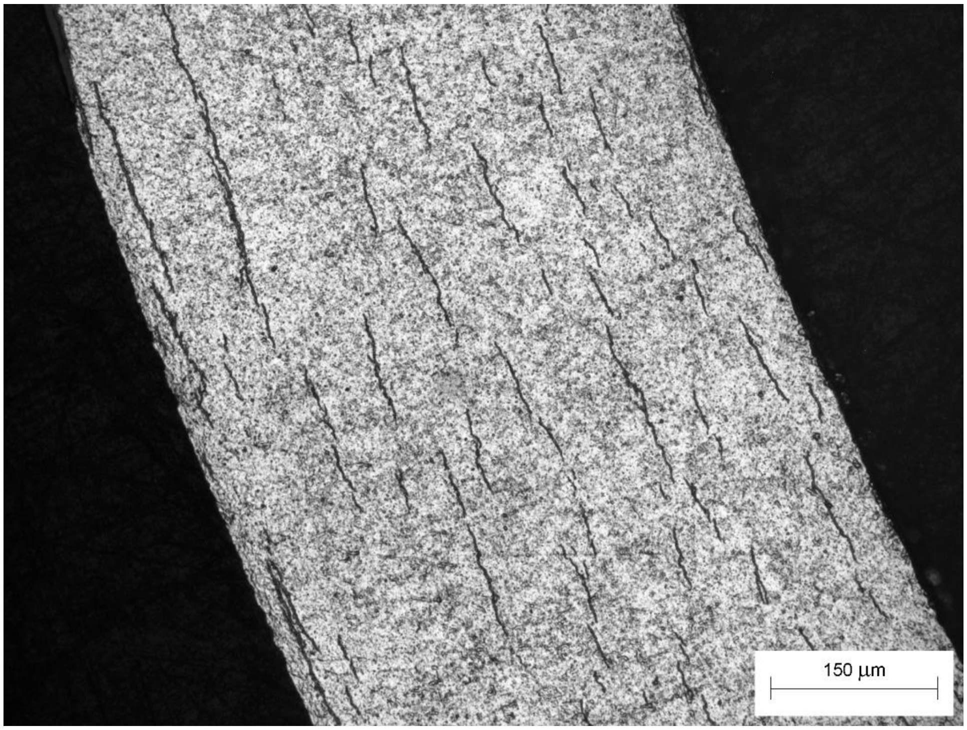
Sample 25 / Quadrant 2 / 200x Magnification



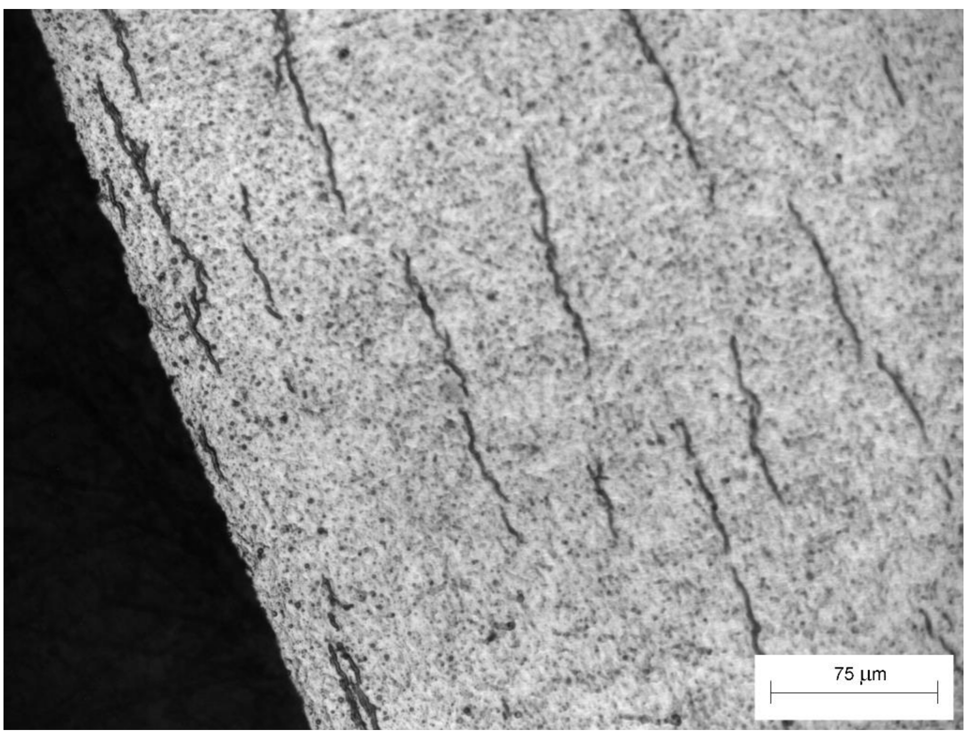
Sample 25 / Quadrant 3 / 100x Magnification



Sample 25 / Quadrant 3 / 200x Magnification



Sample 25 / Quadrant 4 / 100x Magnification



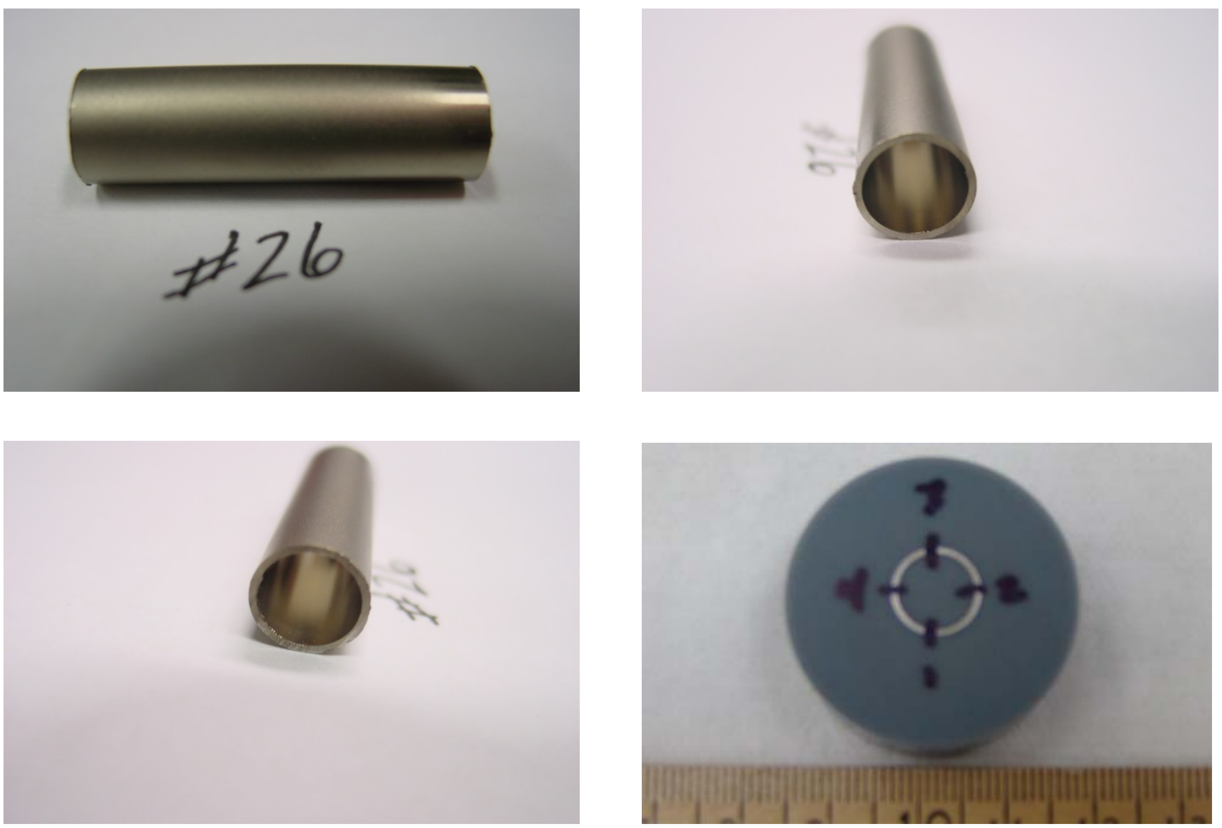
Sample 25 / Quadrant 4 / 200x Magnification

Sample ID: 26

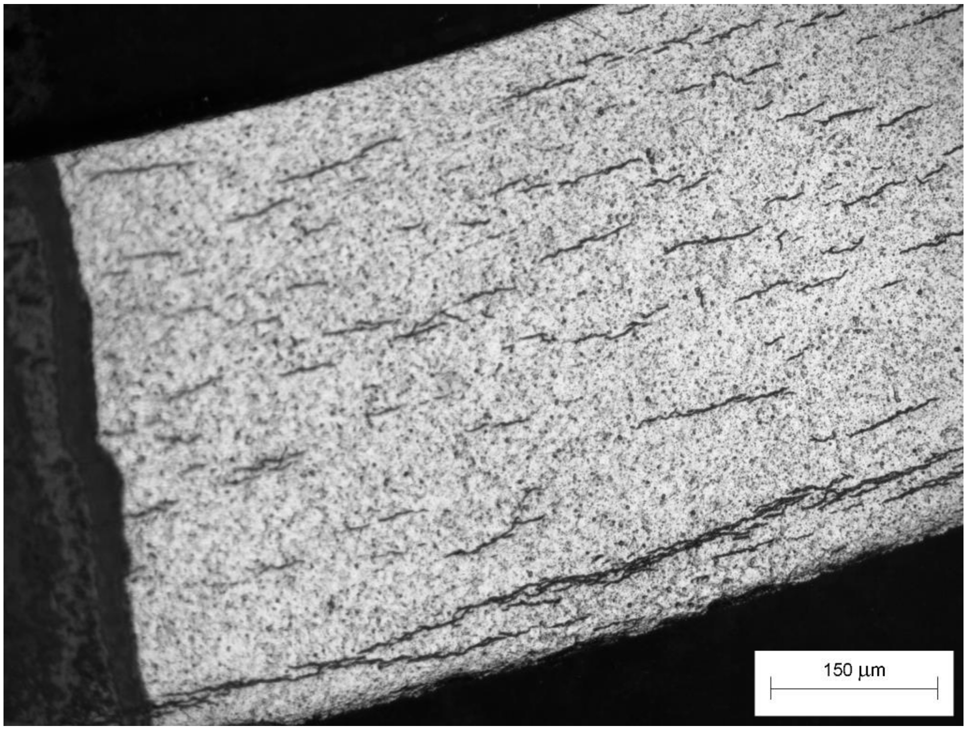
Furnace:	Thermolyne
Location:	Upright
Temperature:	270°C
OD Surface:	Blasted
Outside Diameter:	0.372 in (9.5 mm)
Wall Thickness:	0.022 in (0.56 mm)

Hydrogen Concentration Results

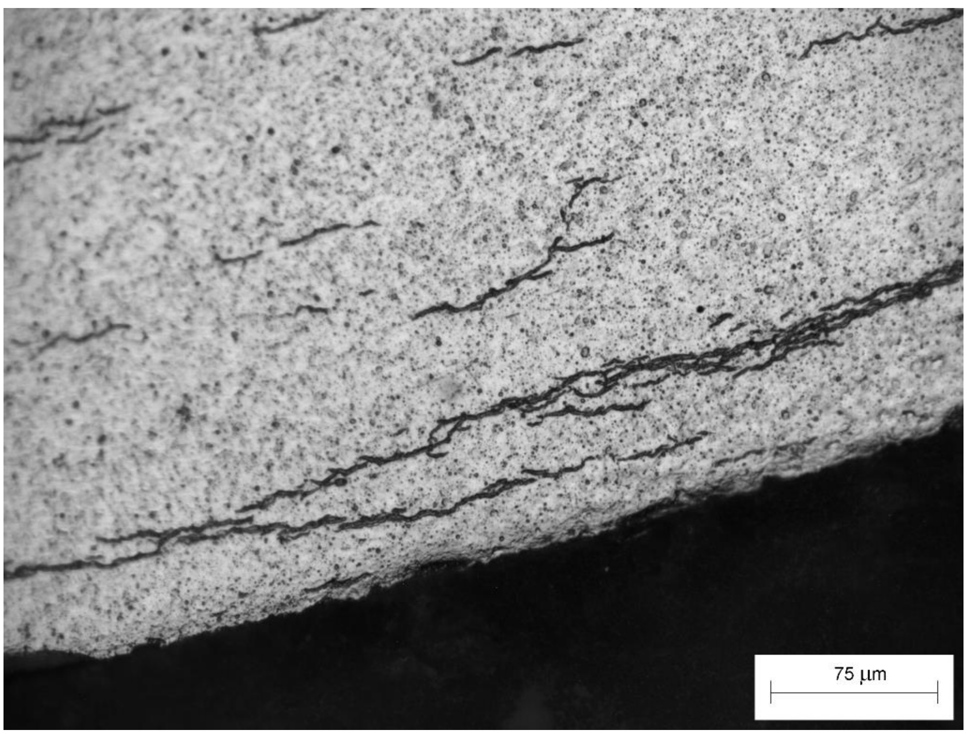
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	180	76	240	120	154	71	46%
Middle-2	210	180	140	130	165	37	22%



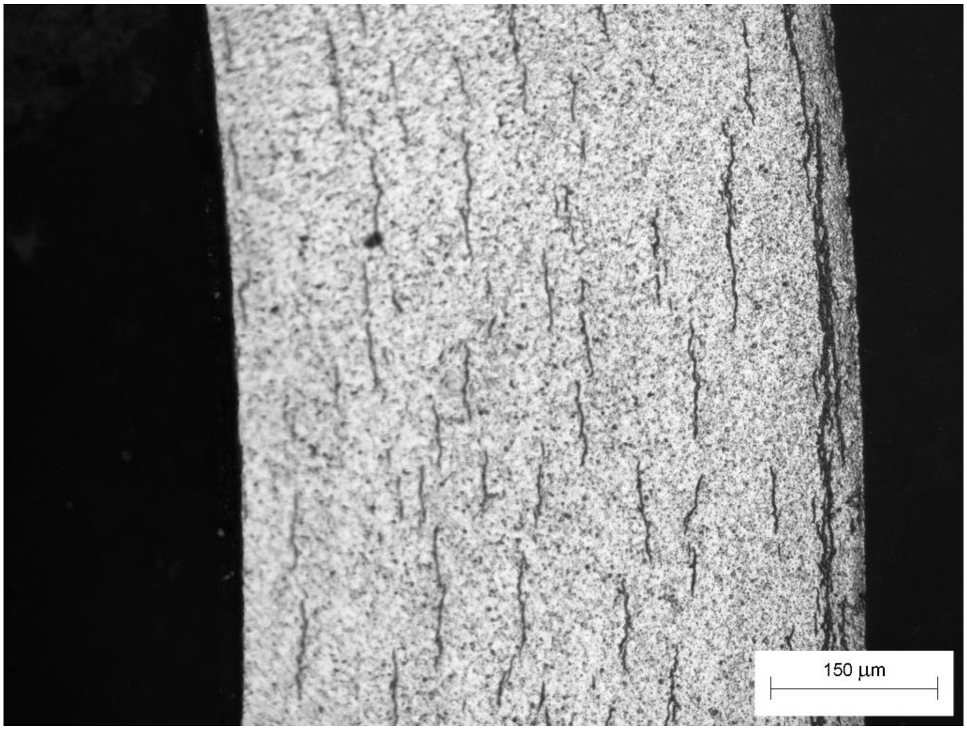
Images of Sample 26 After Hydriding and Mounted in Epoxy



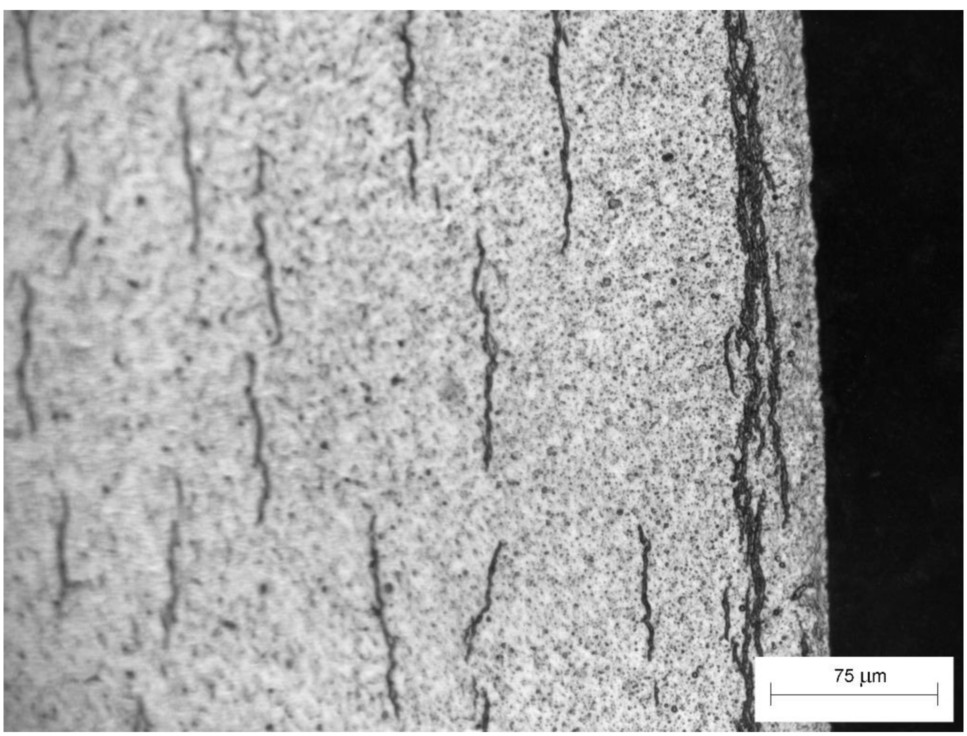
Sample 26 / Quadrant 1 / 100x Magnification



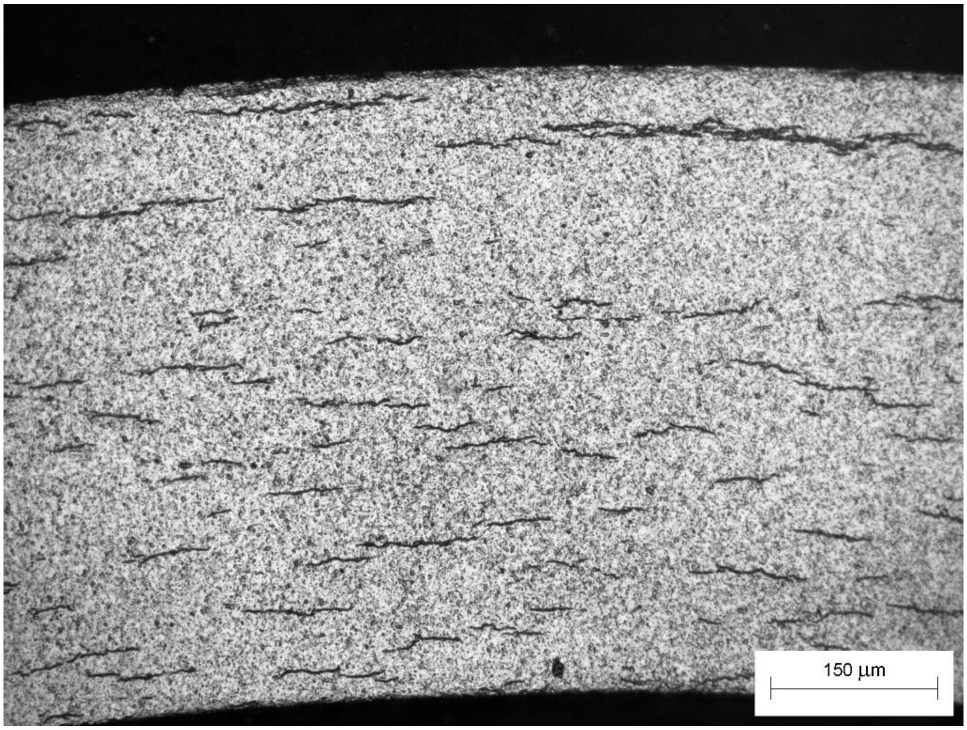
Sample 26 / Quadrant 1 / 200x Magnification



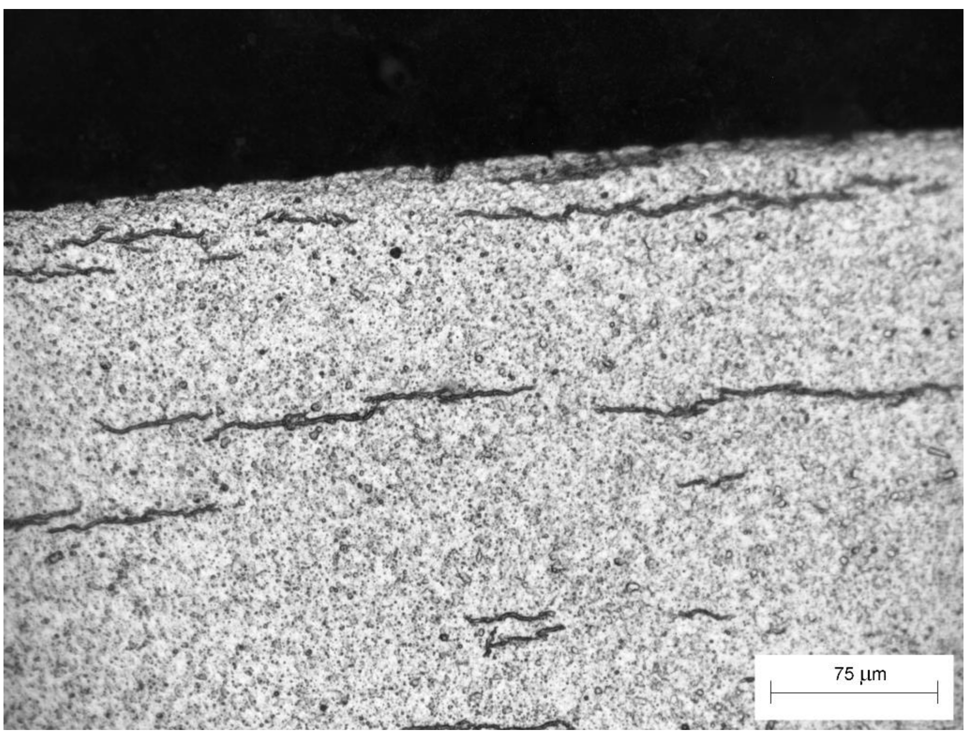
Sample 26 / Quadrant 2 / 100x Magnification



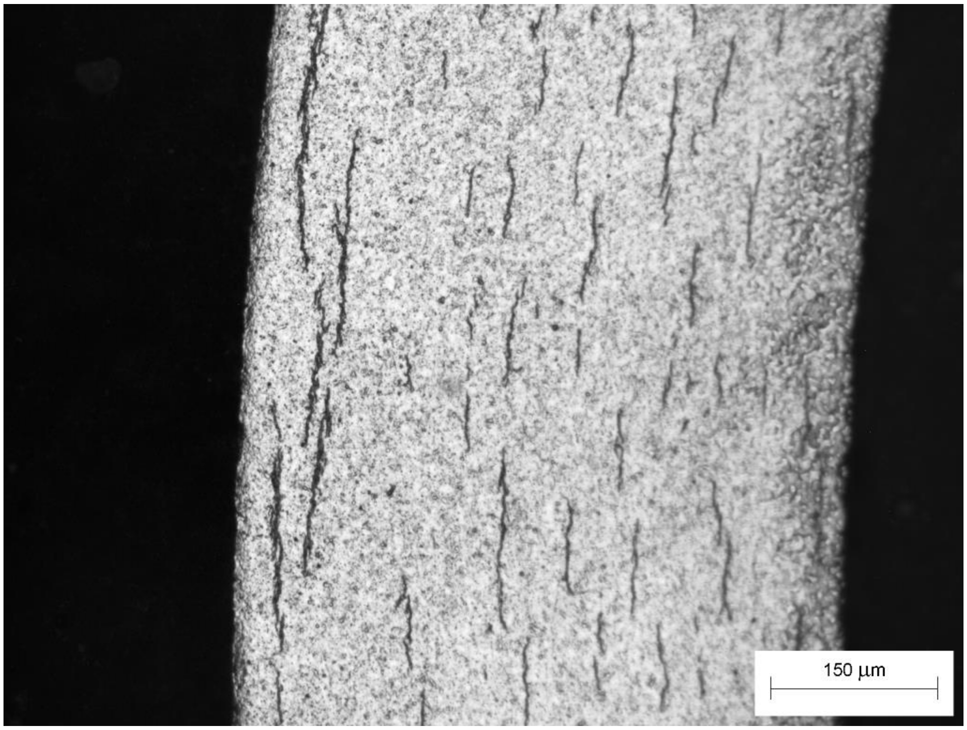
Sample 26 / Quadrant 2 / 200x Magnification



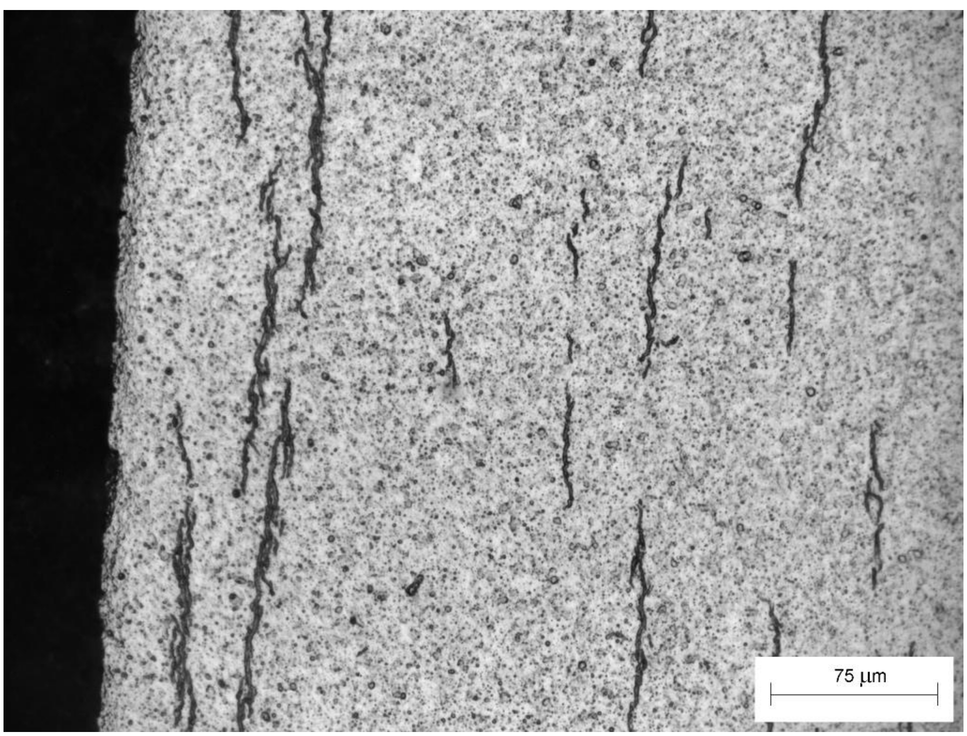
Sample 26 / Quadrant 3 / 100x Magnification



Sample 26 / Quadrant 3 / 200x Magnification



Sample 26 / Quadrant 4 / 100x Magnification



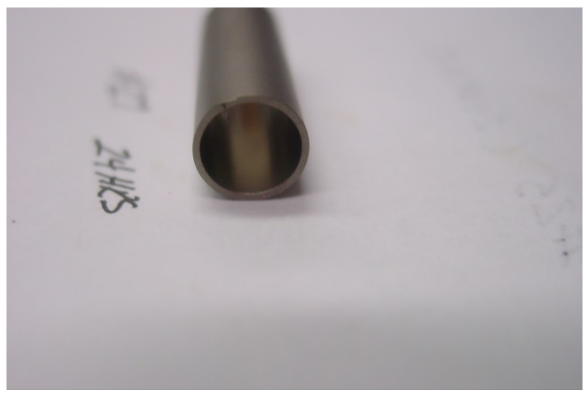
Sample 26 / Quadrant 4 / 200x Magnification

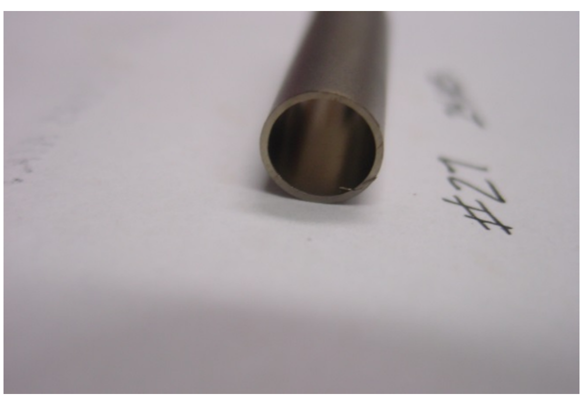
Sample ID: 27

Furnace: Thermolyne
Location: Upright
Temperature: 280°C
OD Surface: Blasted, 24 HR Wait in Air
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

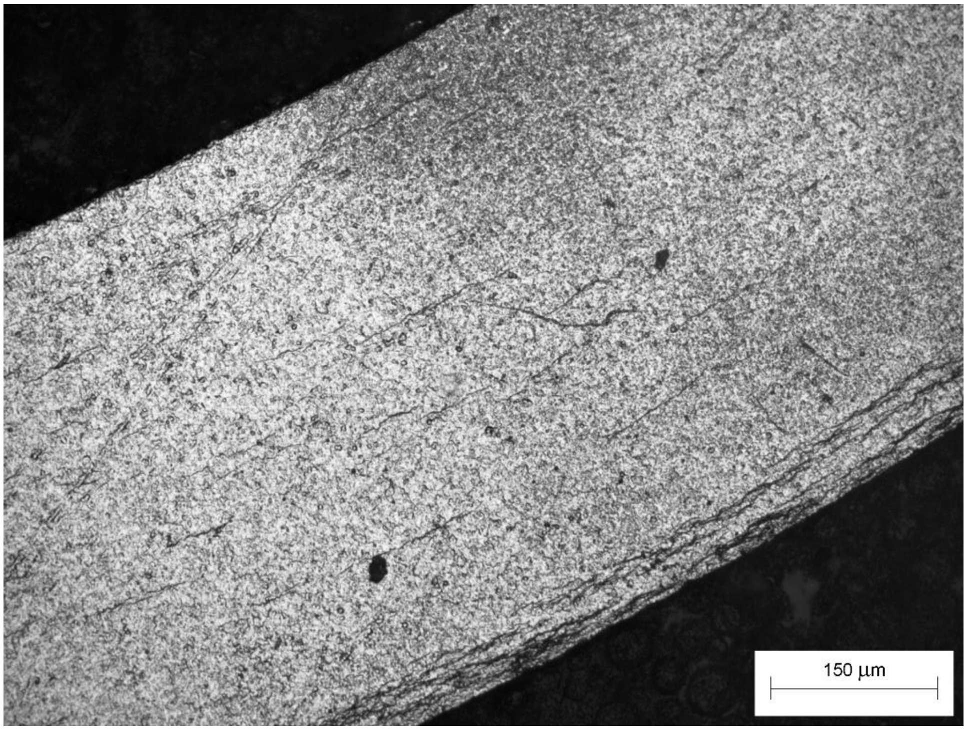
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	340	410	360	340	363	33	9%
Middle-2	310	310	340	420	345	52	15%

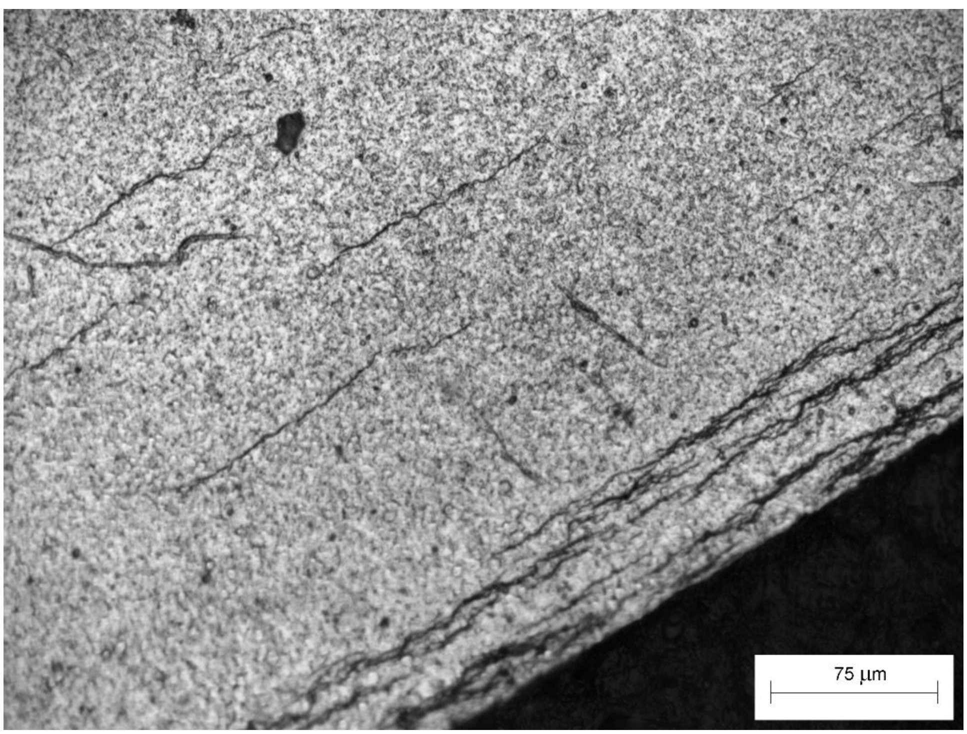




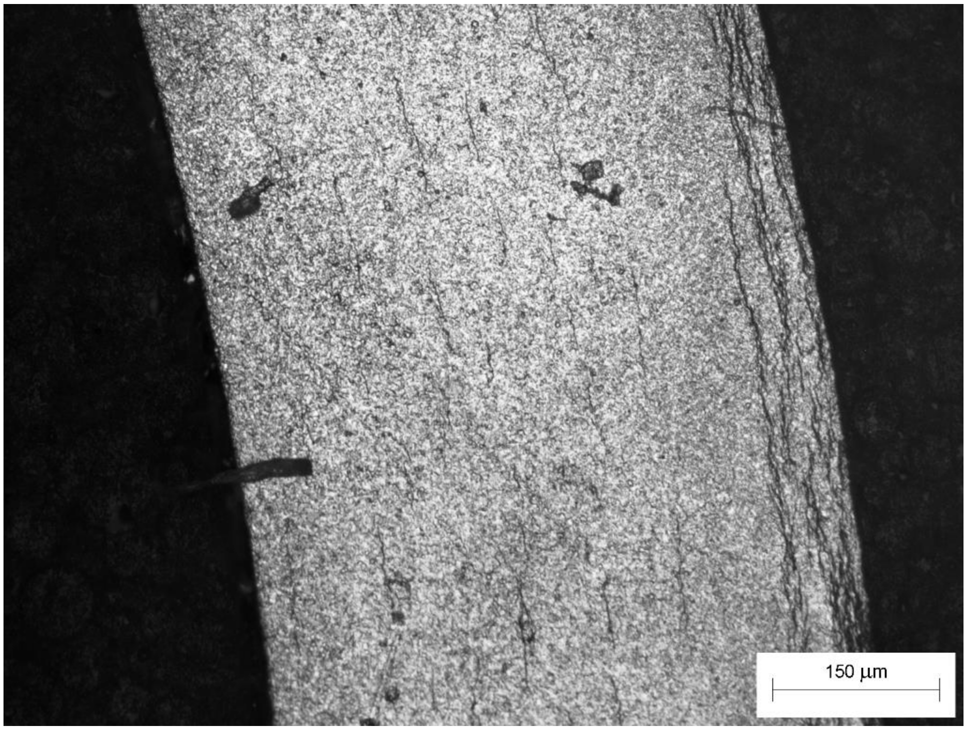
Images of Sample 27 After Hydriding and Mounted in Epoxy.



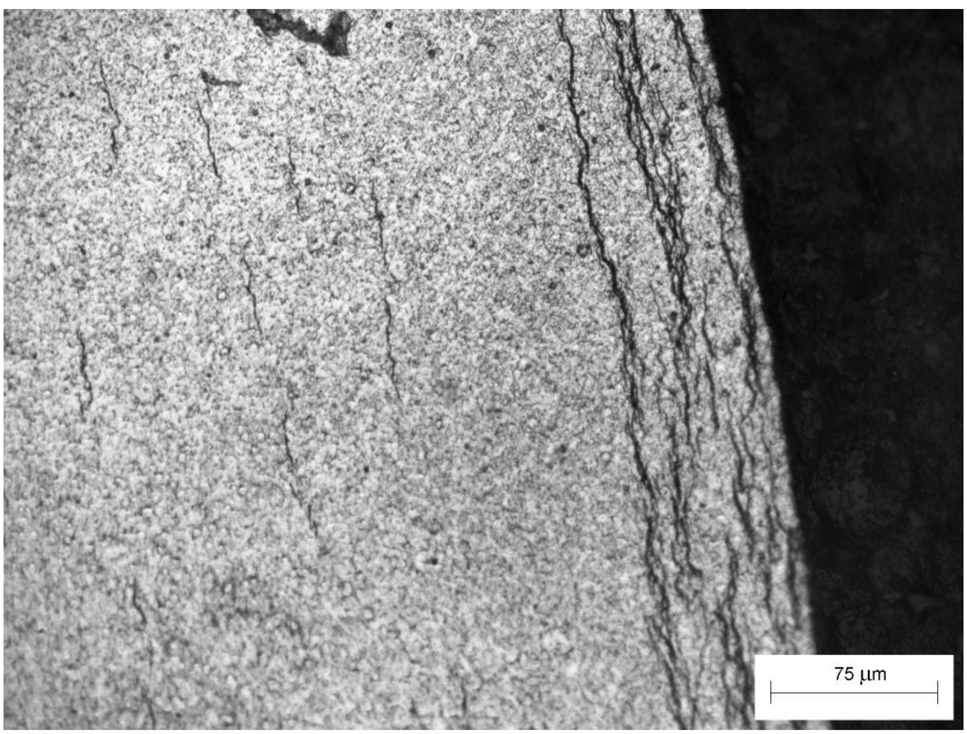
Sample 27 / Quadrant 1 / 100x Magnification



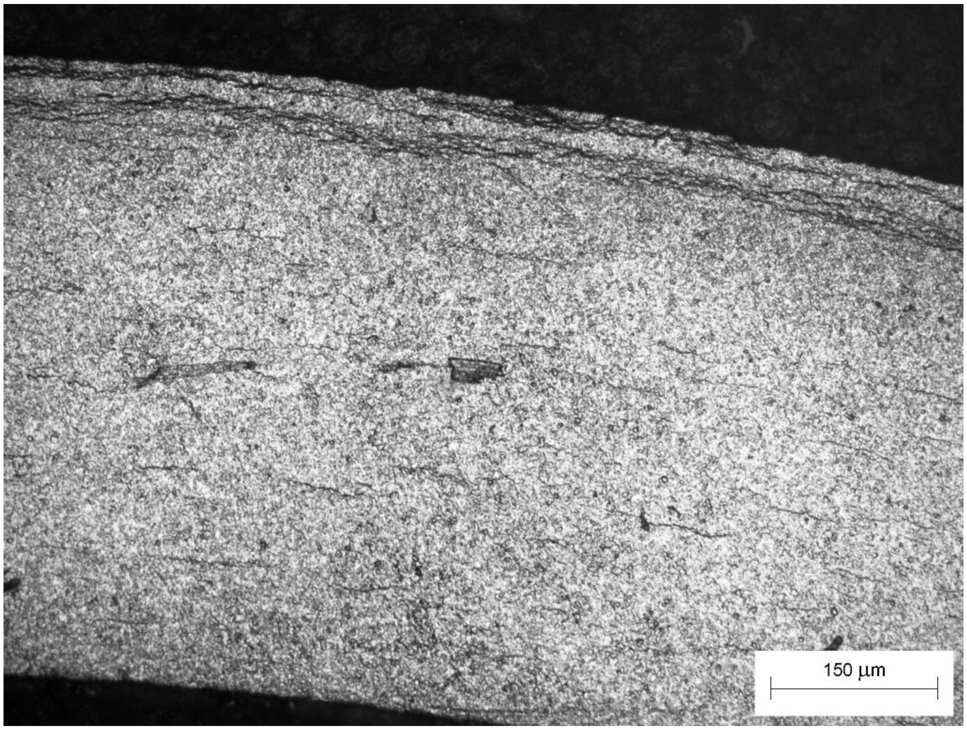
Sample 27 / Quadrant 1 / 200x Magnification



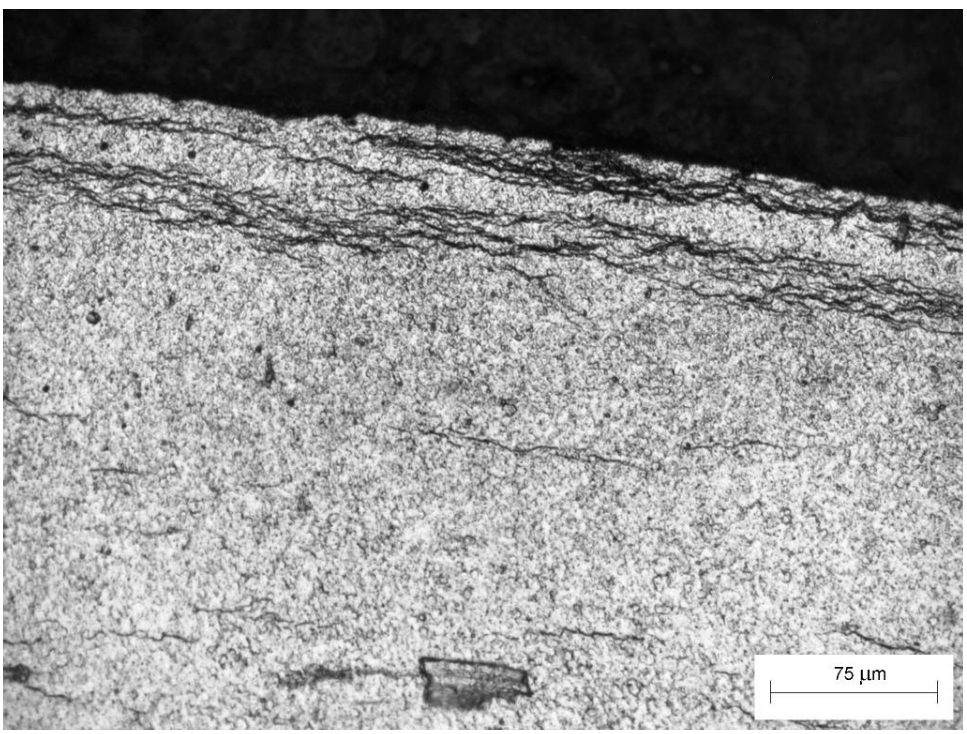
Sample 27 / Quadrant 2 / 100x Magnification



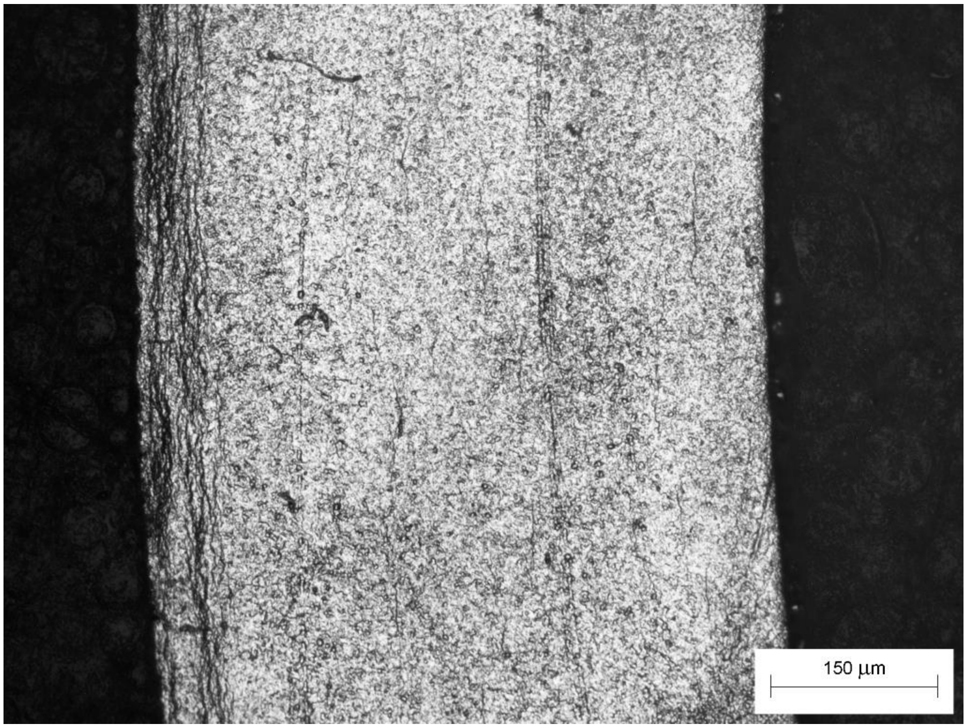
Sample 27 / Quadrant 2 / 200x Magnification



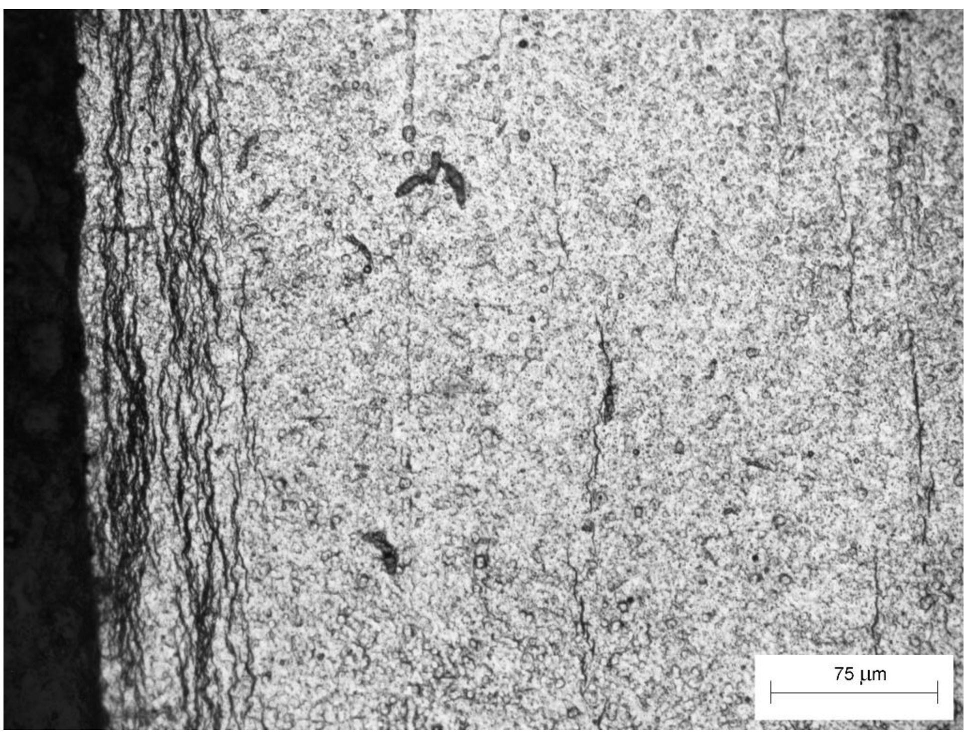
Sample 27 / Quadrant 3 / 100x Magnification



Sample 27 / Quadrant 3 / 200x Magnification



Sample 27 / Quadrant 4 / 100x Magnification



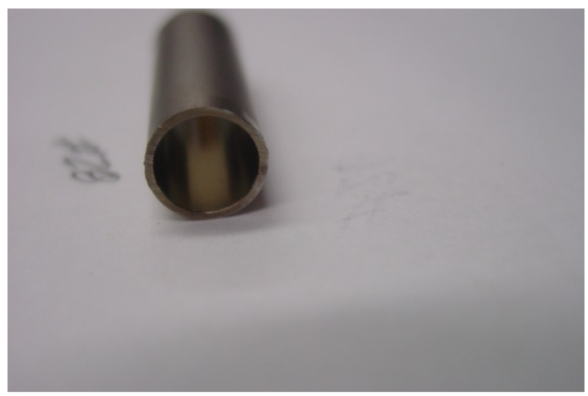
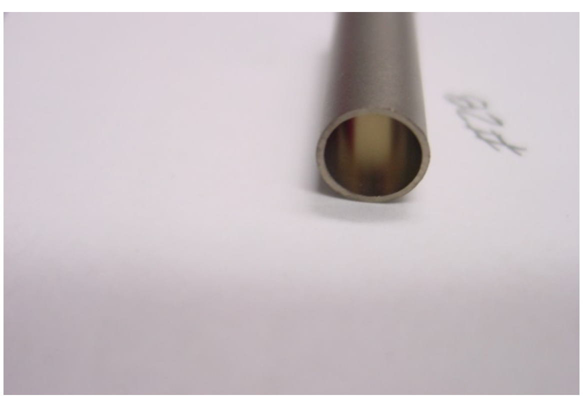
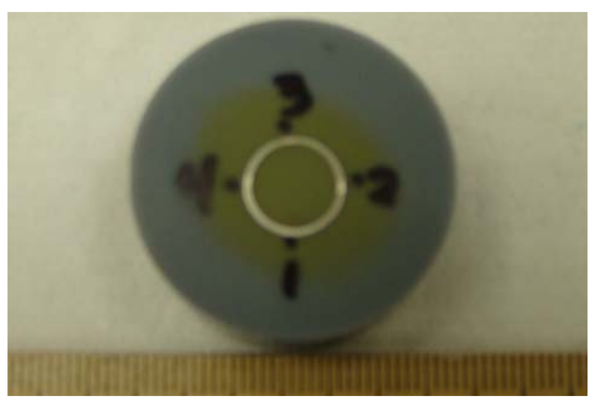
Sample 27 / Quadrant 4 / 200x Magnification

Sample ID: 28

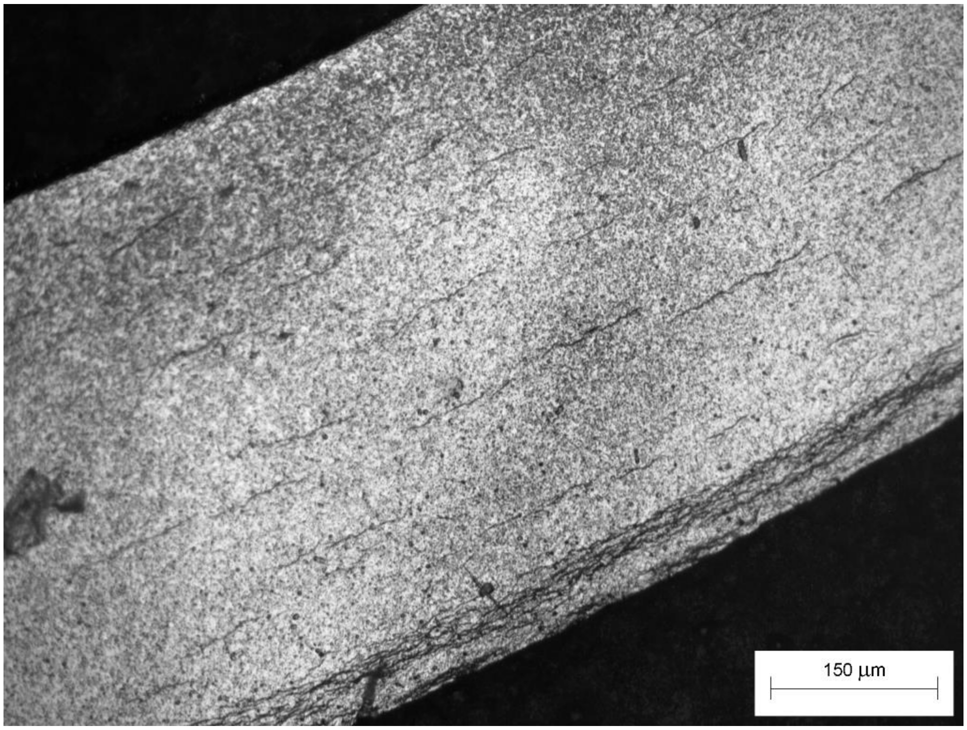
Furnace: Thermolyne
Location: Upright
Temperature: 280°C
OD Surface: Blasted
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

Hydrogen Concentration Results

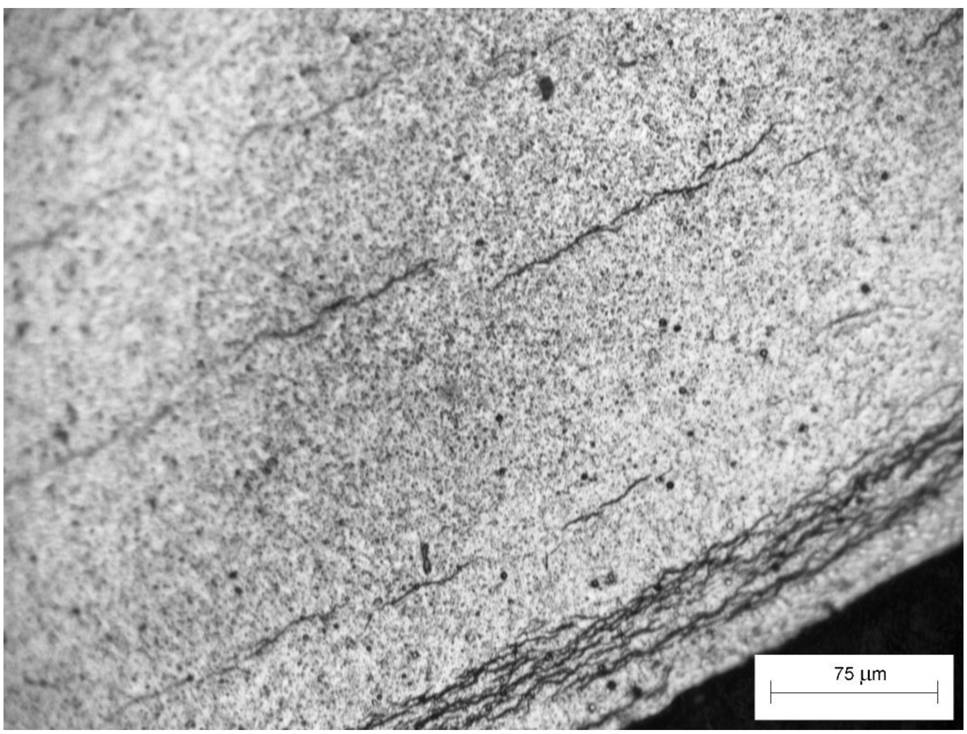
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	350	360	360	300	343	29	8%
Middle-2	270	250	190	180	223	44	20%

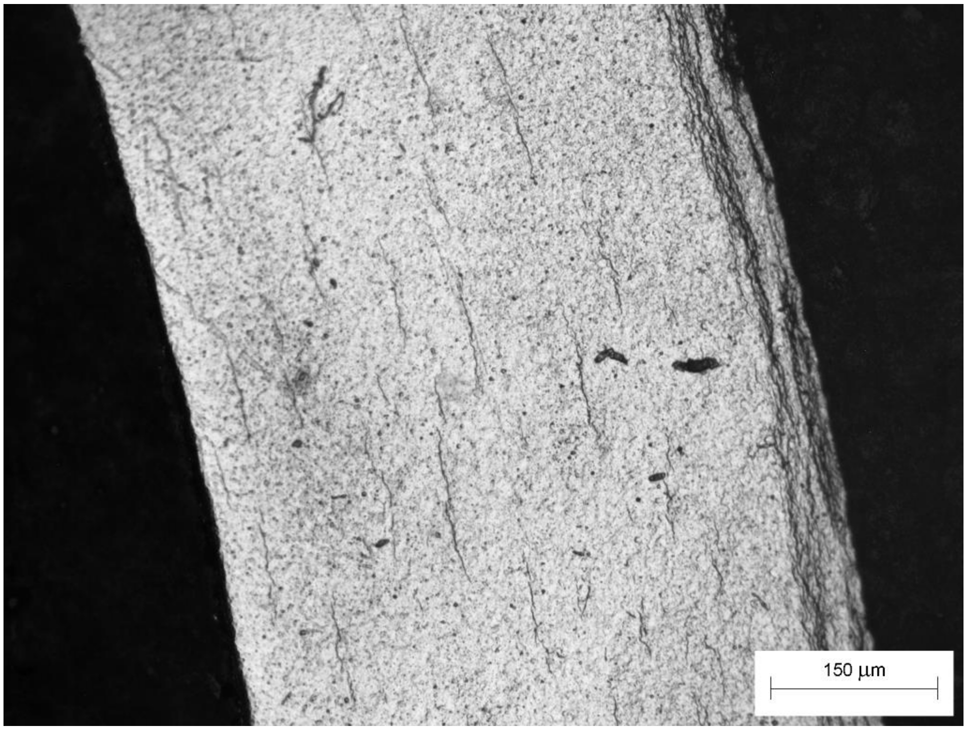
Images of Sample 28 After Hydriding and Mounted in Epoxy.



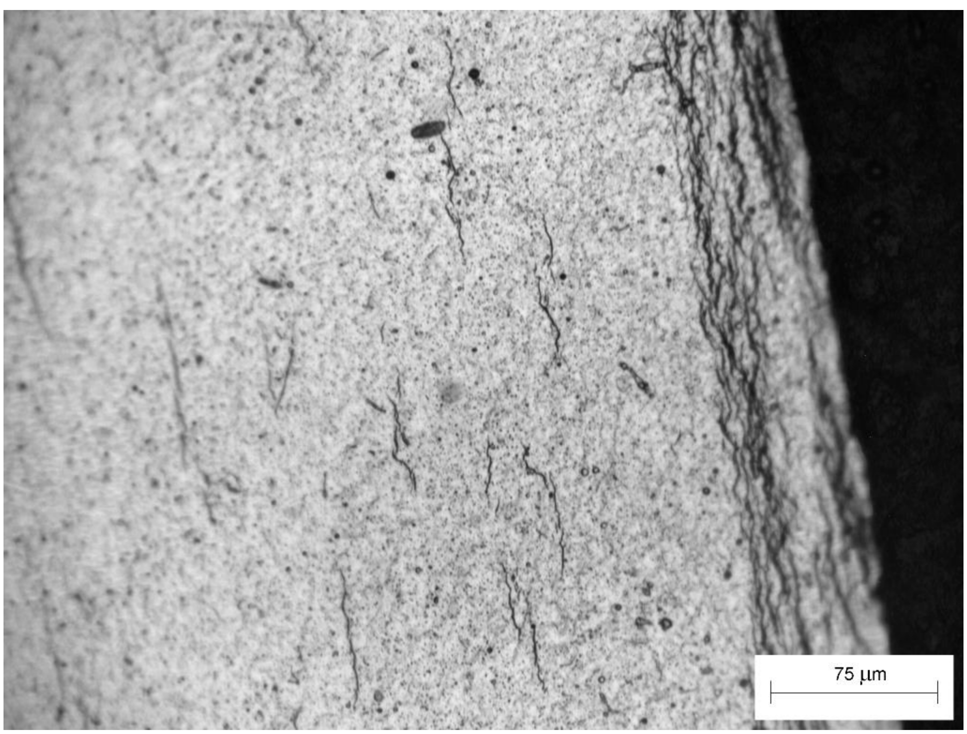
Sample 28 / Quadrant 1 / 100x Magnification



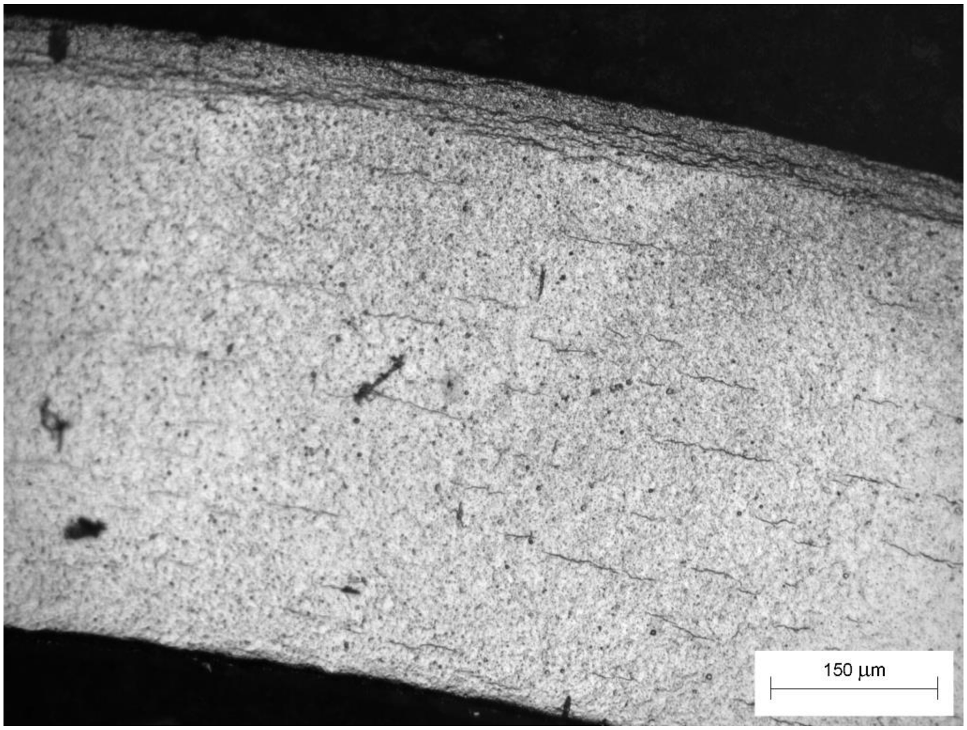
Sample 28 / Quadrant 1 / 200x Magnification



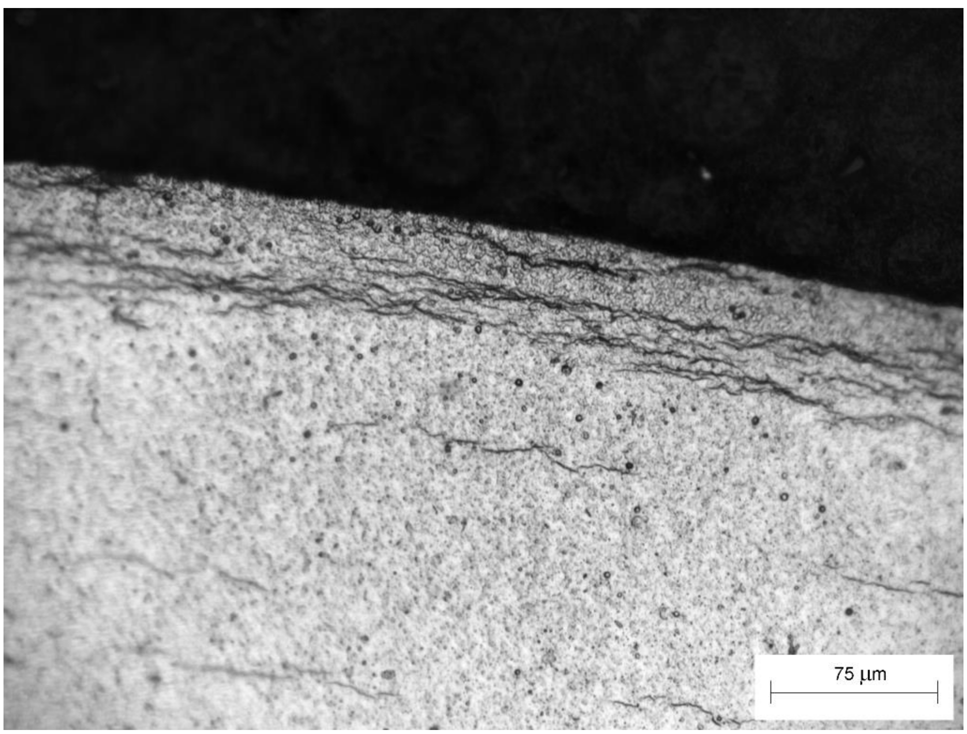
Sample 28 / Quadrant 2 / 100x Magnification



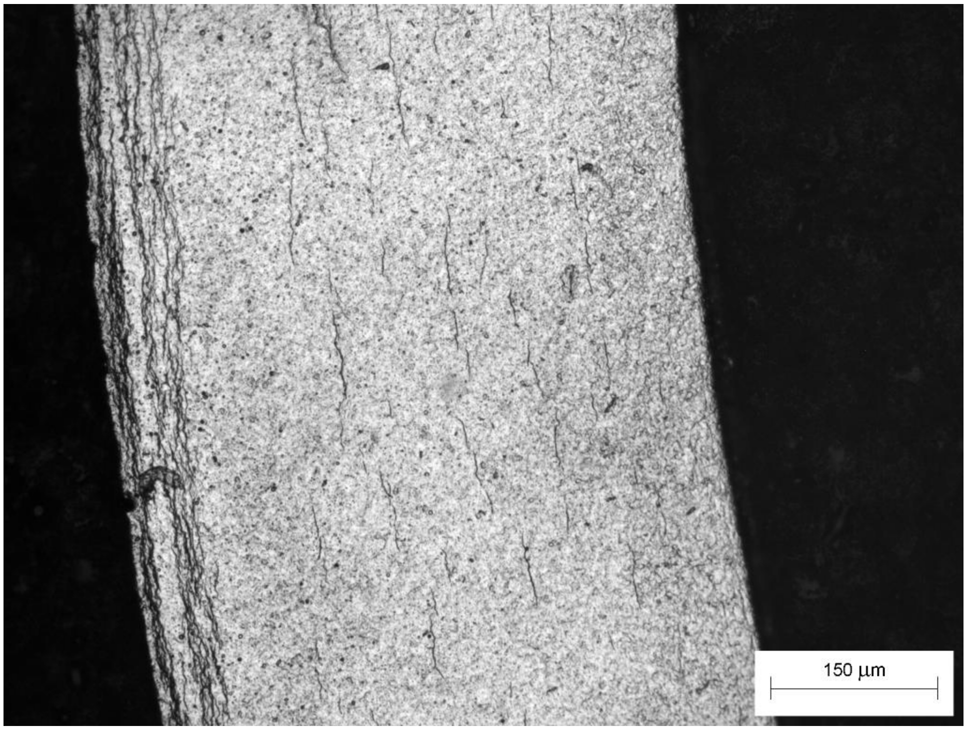
Sample 28 / Quadrant 2 / 200x Magnification



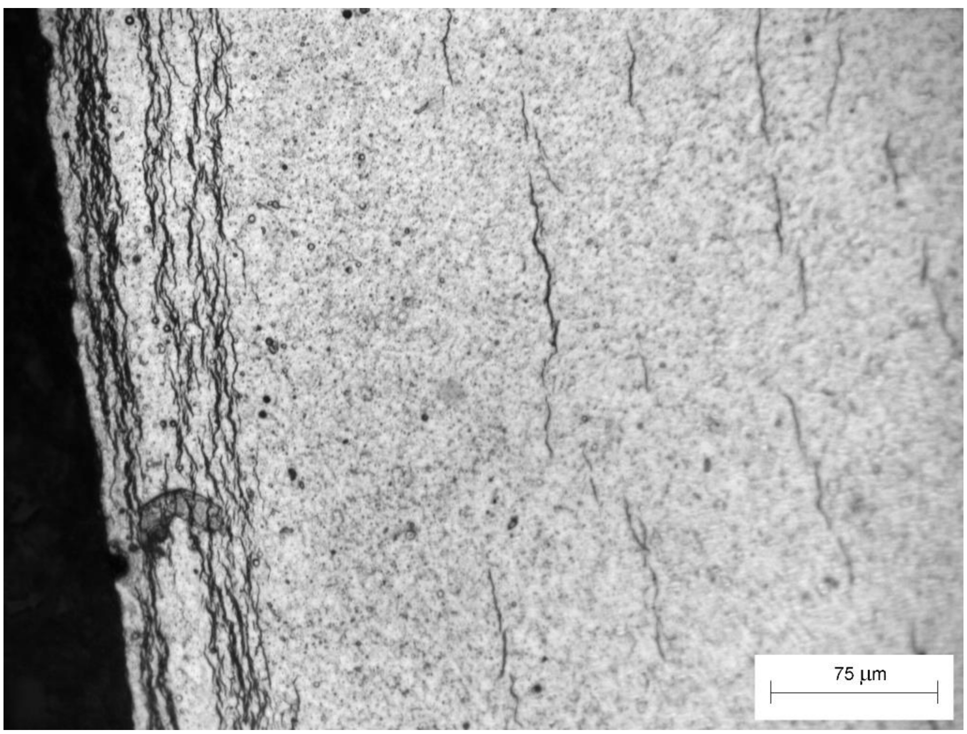
Sample 28 / Quadrant 3 / 100x Magnification



Sample 28 / Quadrant 3 / 200x Magnification



Sample 28 / Quadrant 4 / 100x Magnification



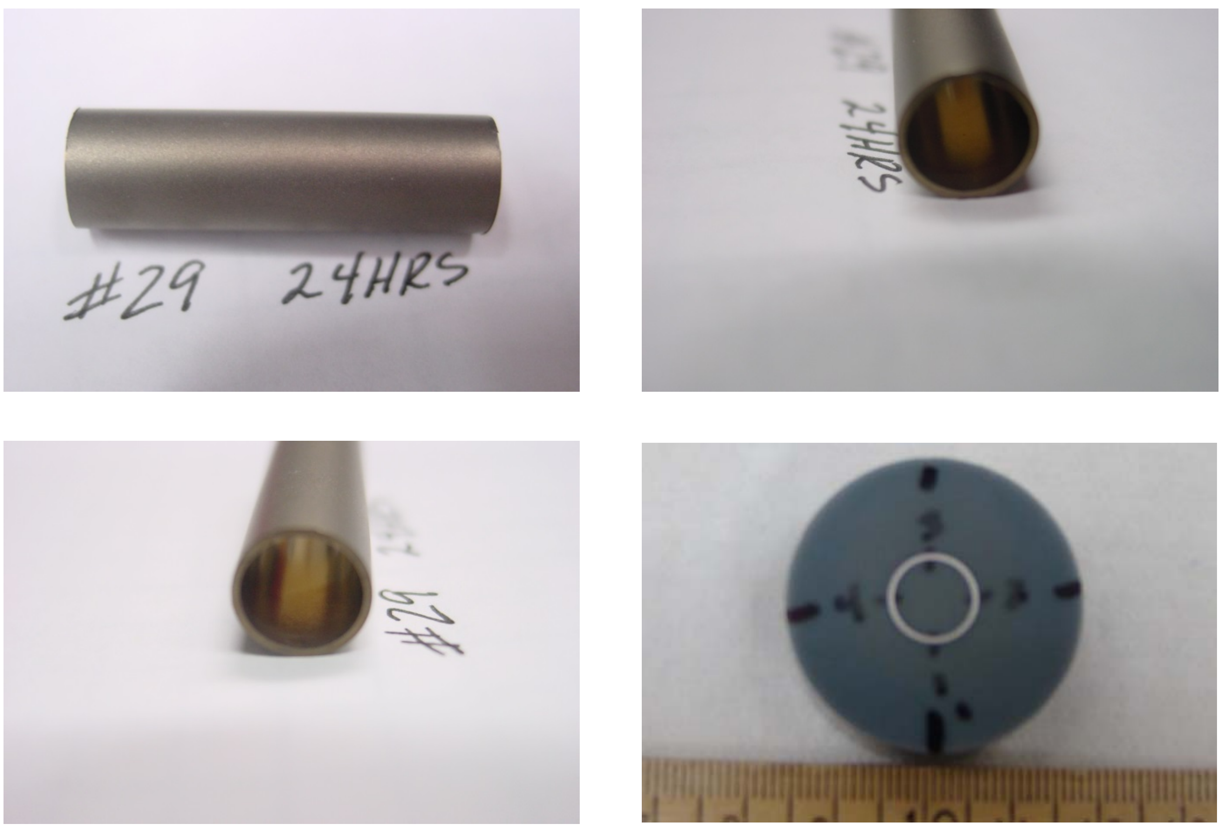
Sample 28 / Quadrant 4 / 200x Magnification

Sample ID: 29

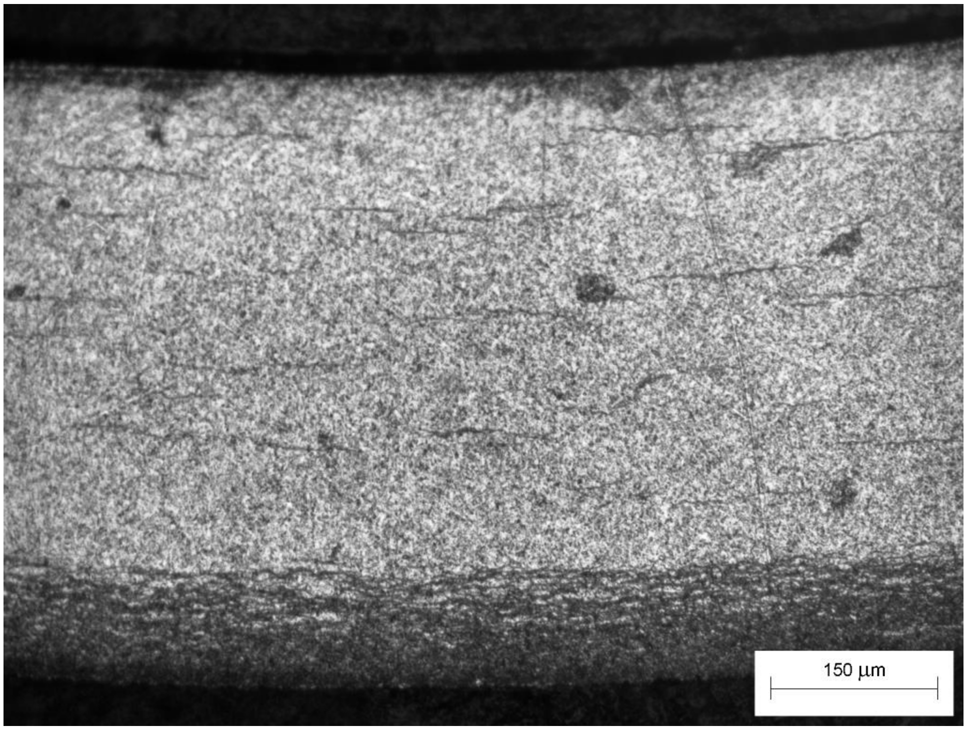
Furnace: Thermolyne
Location: Upright
Temperature: 290°C
OD Surface: Blasted, 24 HR Wait in Air
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

Hydrogen Concentration Results

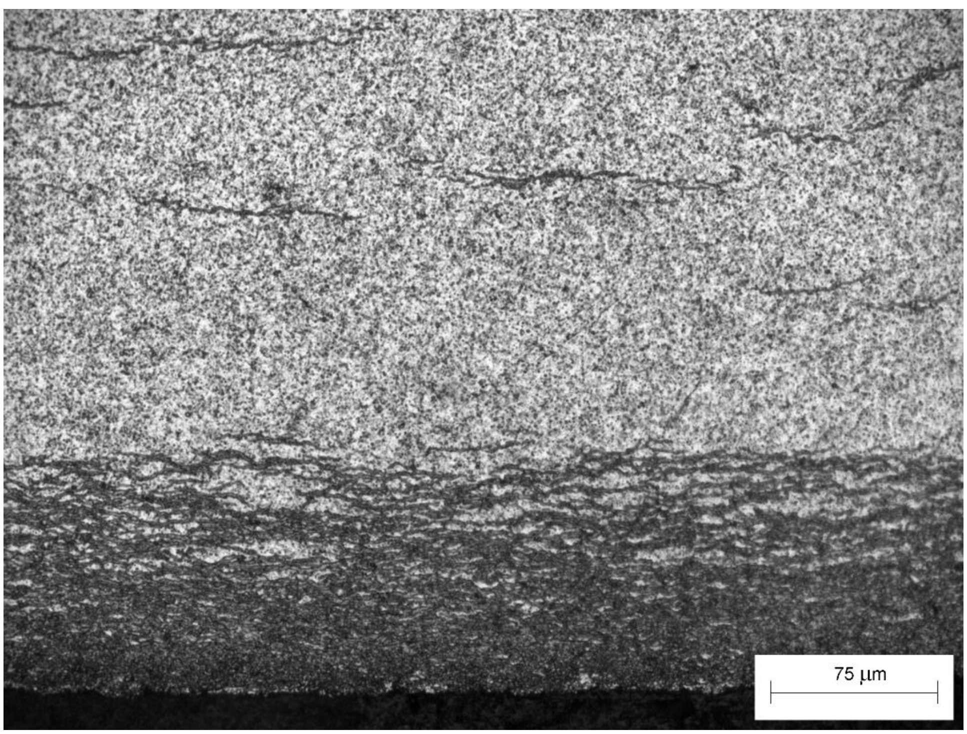
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	1200	1400	1100	700	1100	294	27%
Middle-2	1200	1300	840	910	1063	222	21%



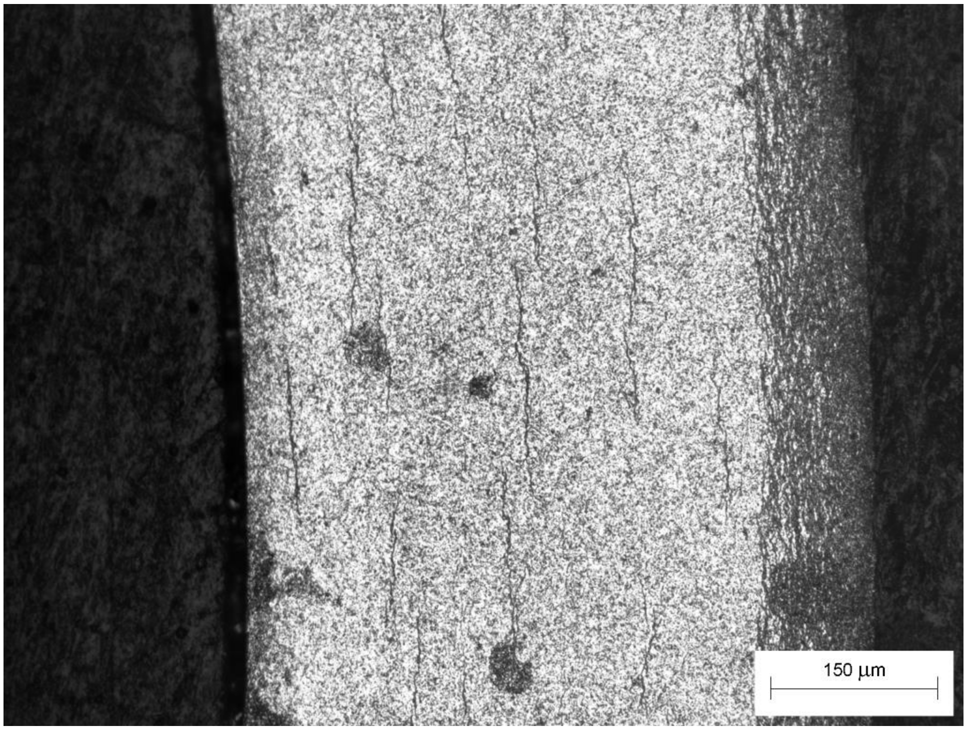
Images of Sample 29 After Hydriding and Mounted in Epoxy.



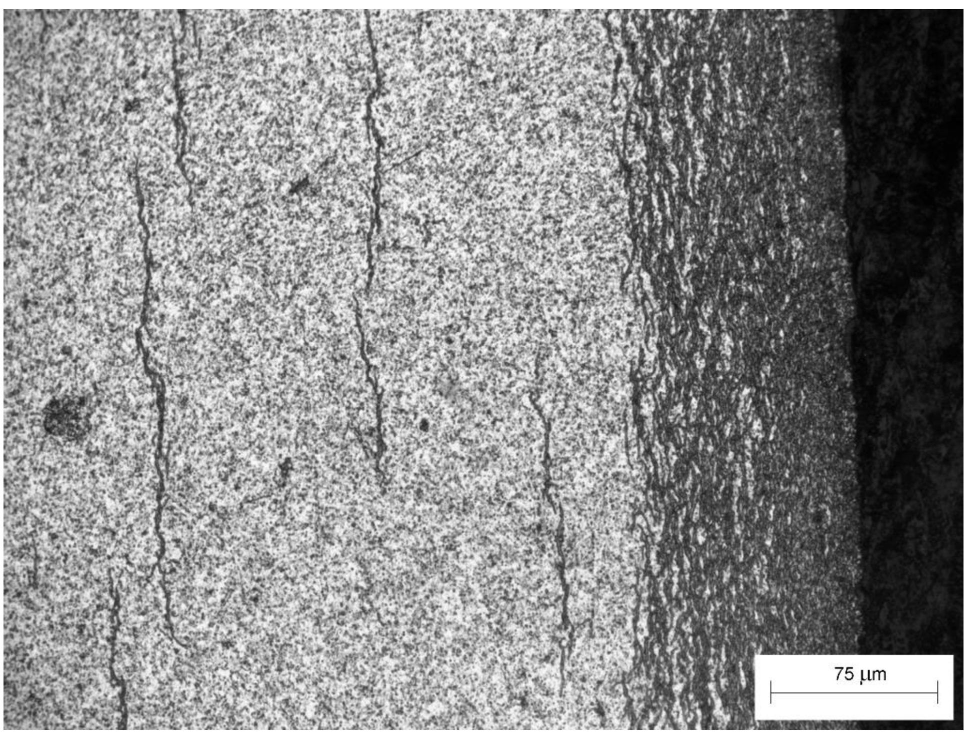
Sample 29 / Quadrant 1 / 100x Magnification



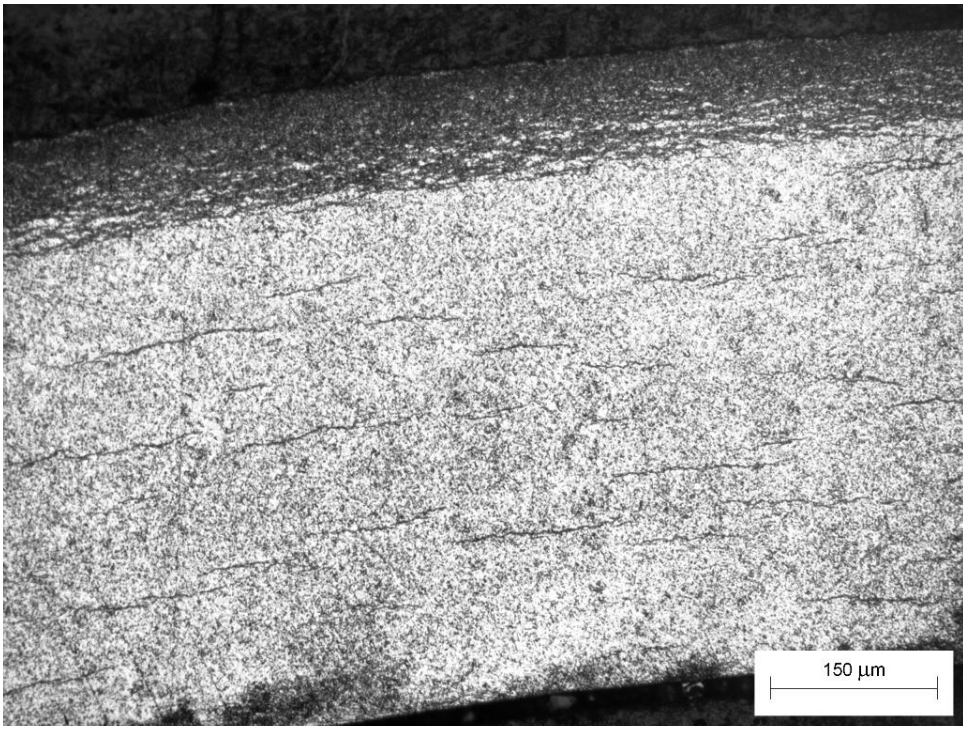
Sample 29 / Quadrant 1 / 200x Magnification



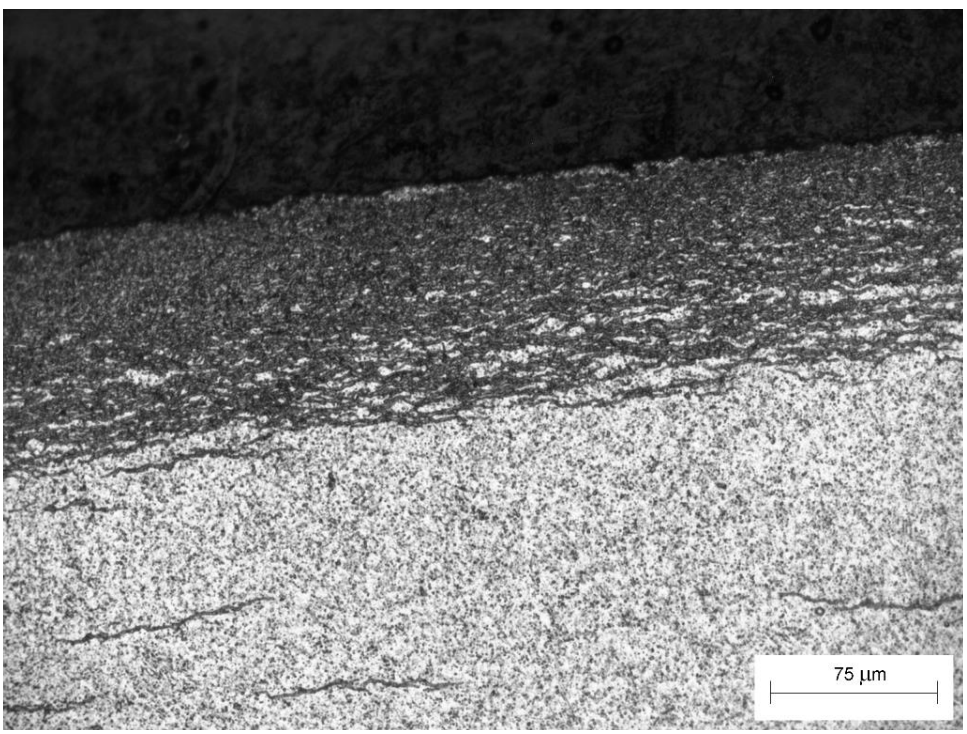
Sample 29 / Quadrant 2 / 100x Magnification



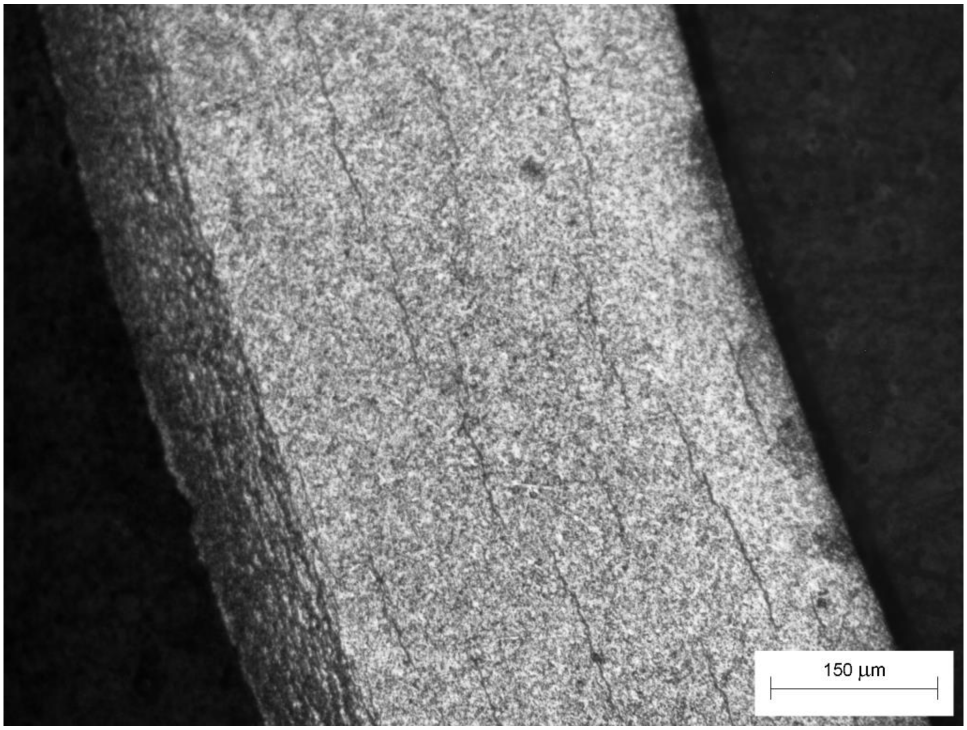
Sample 29 / Quadrant 2 / 200x Magnification



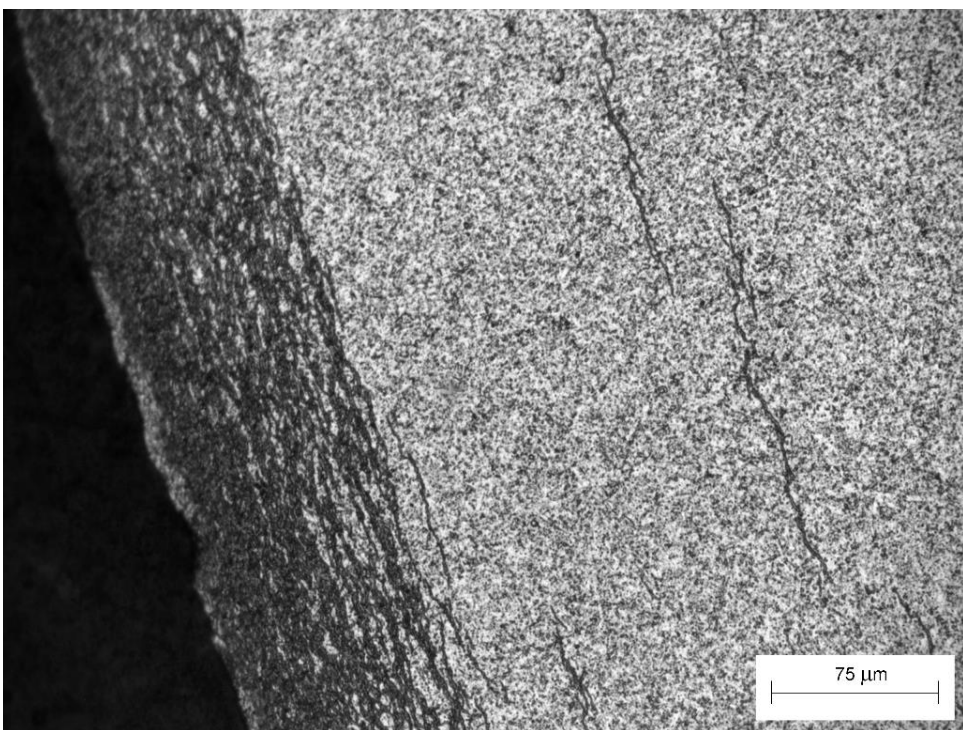
Sample 29 / Quadrant 3 / 100x Magnification



Sample 29 / Quadrant 3 / 200x Magnification



Sample 29 / Quadrant 4 / 100x Magnification



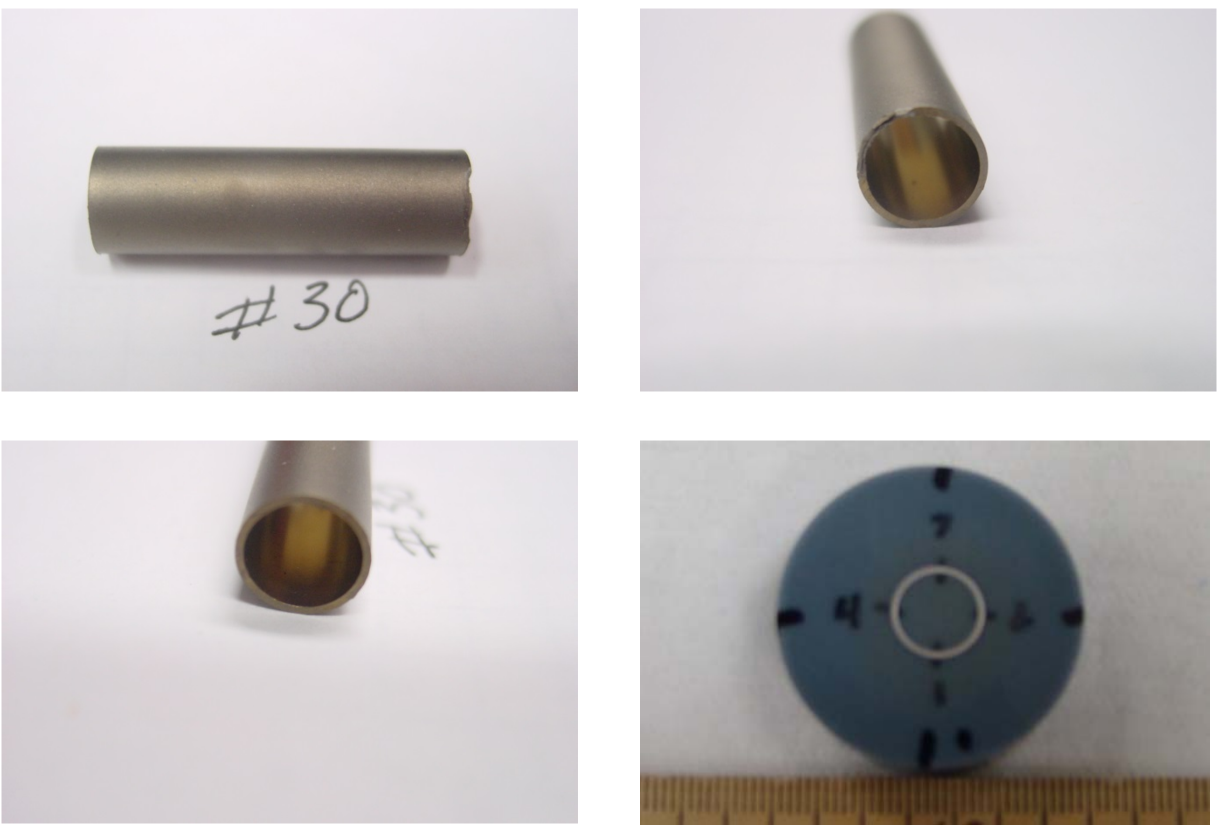
Sample 29 / Quadrant 4 / 200x Magnification

Sample ID: 30

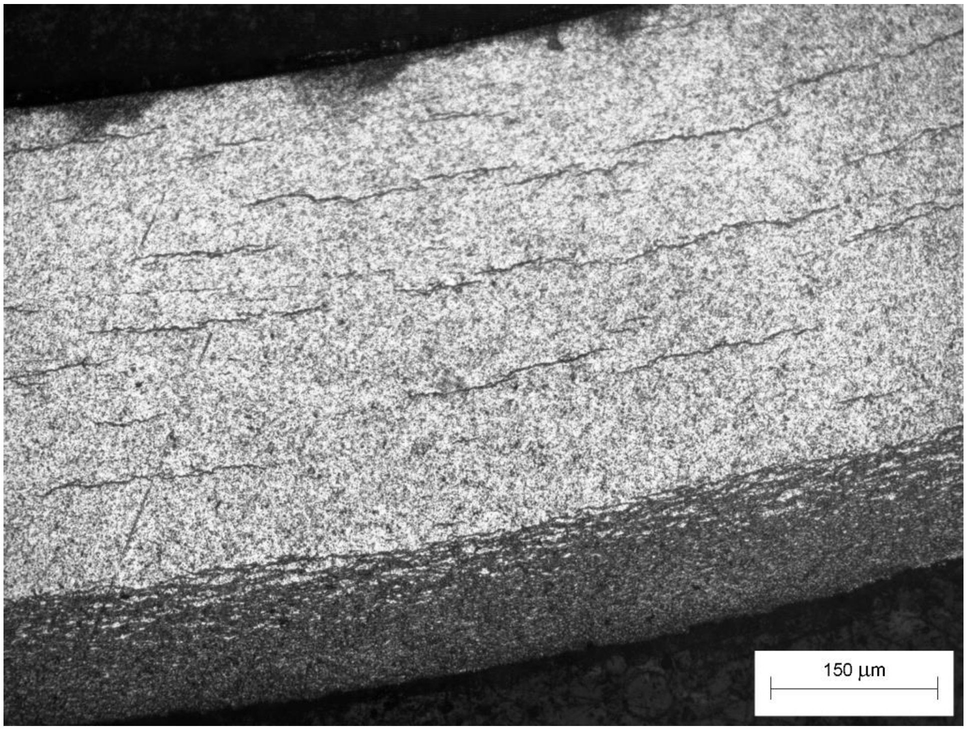
Furnace: Thermolyne
Location: Upright
Temperature: 290°C
OD Surface: Blasted
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

Hydrogen Concentration Results

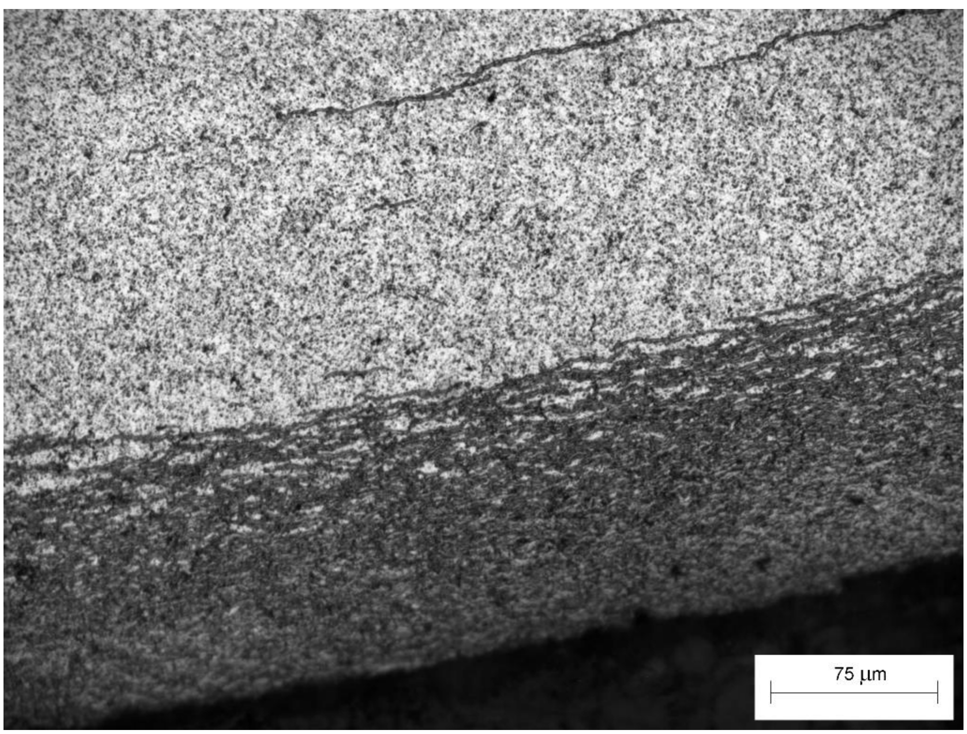
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	1300	1000	1300	1100	1175	150	13%
Middle-2	1100	1100	910	920	1008	107	11%



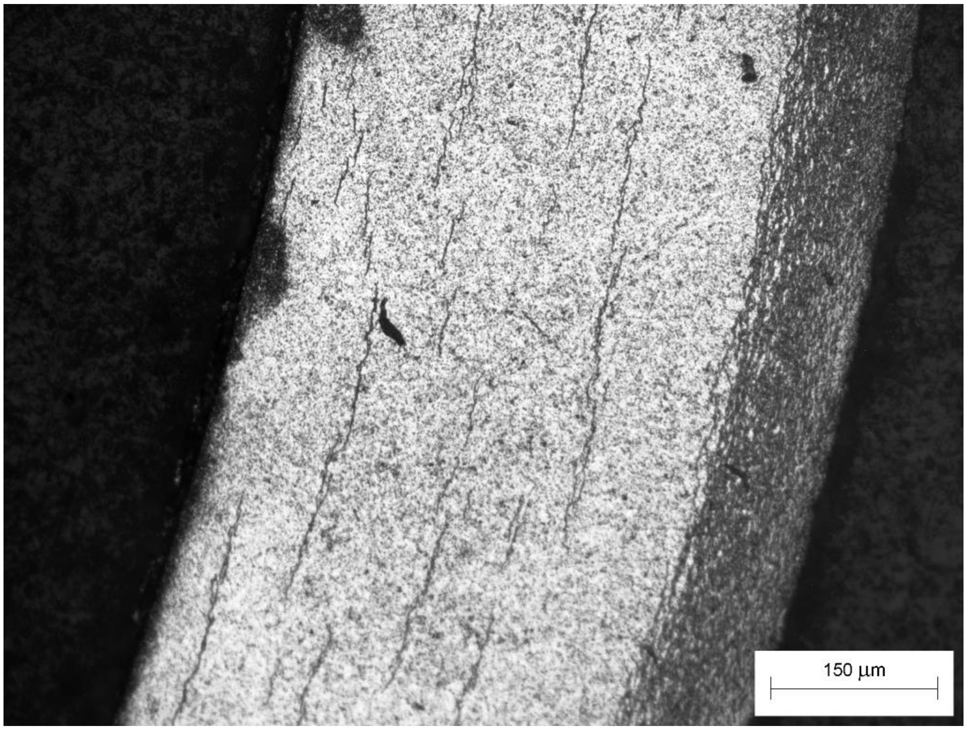
Images of Sample 30 After Hydriding and Mounted in Epoxy.



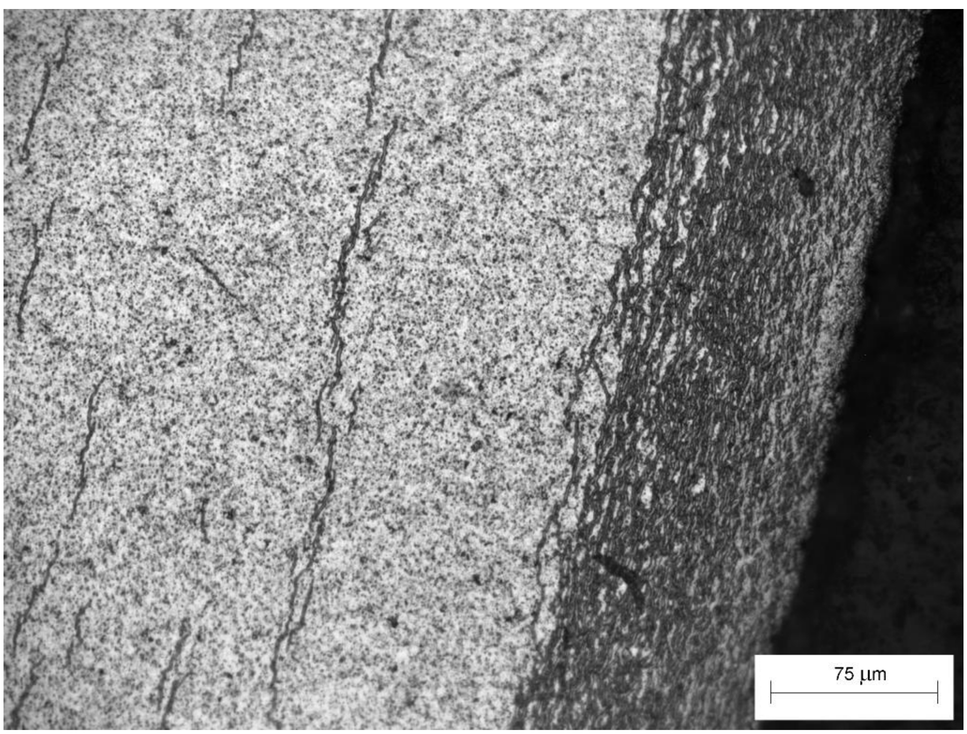
Sample 30 / Quadrant 1 / 100x Magnification



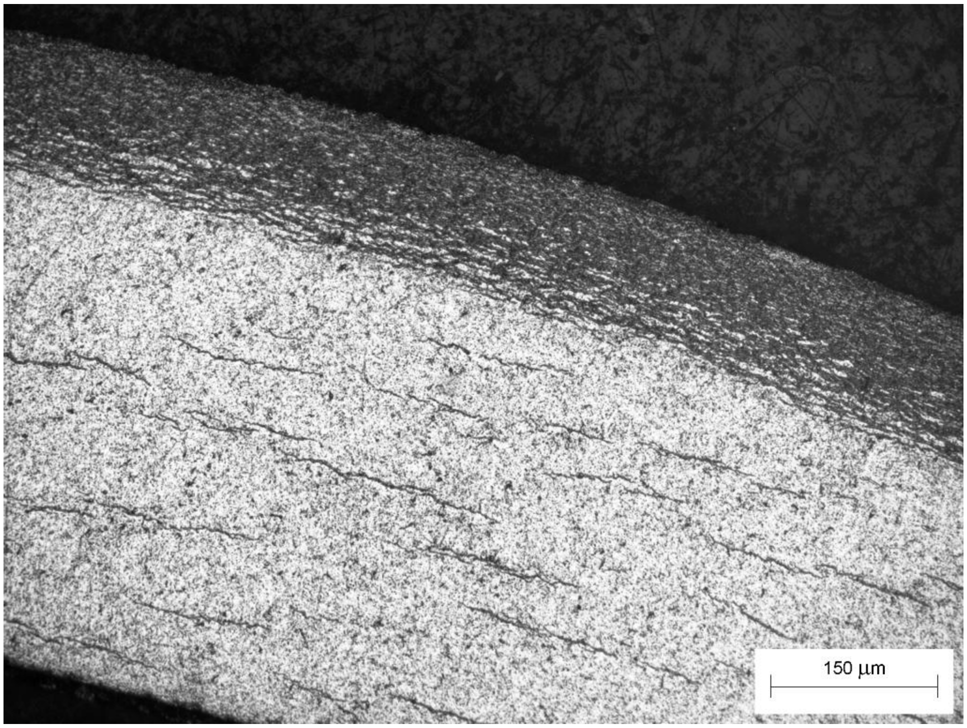
Sample 30 / Quadrant 1 / 200x Magnification



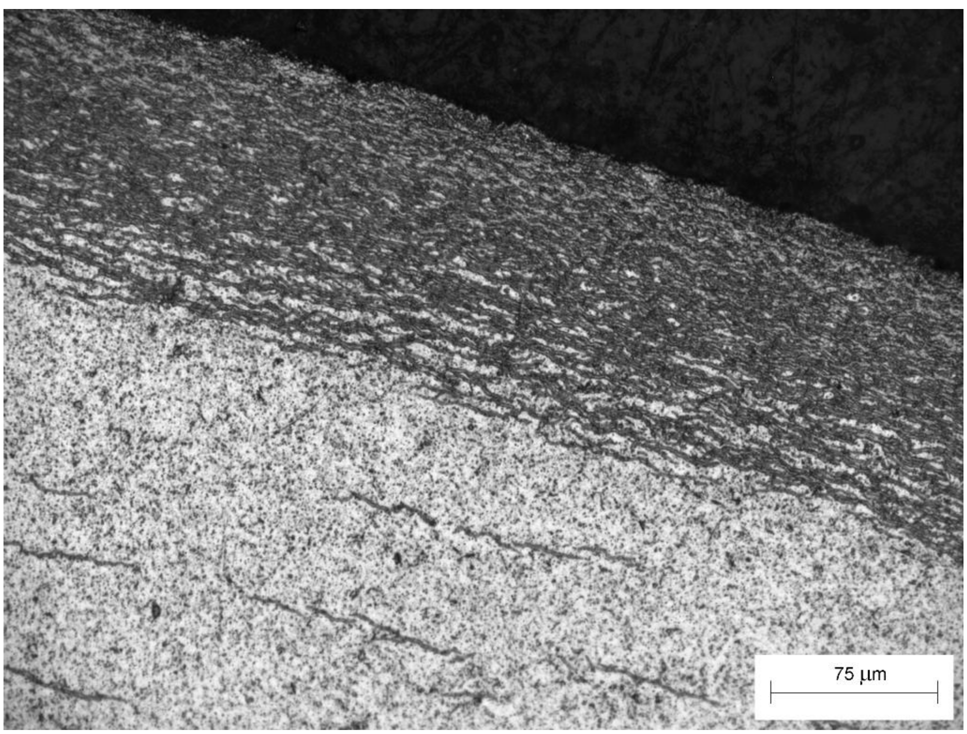
Sample 30 / Quadrant 2 / 100x Magnification



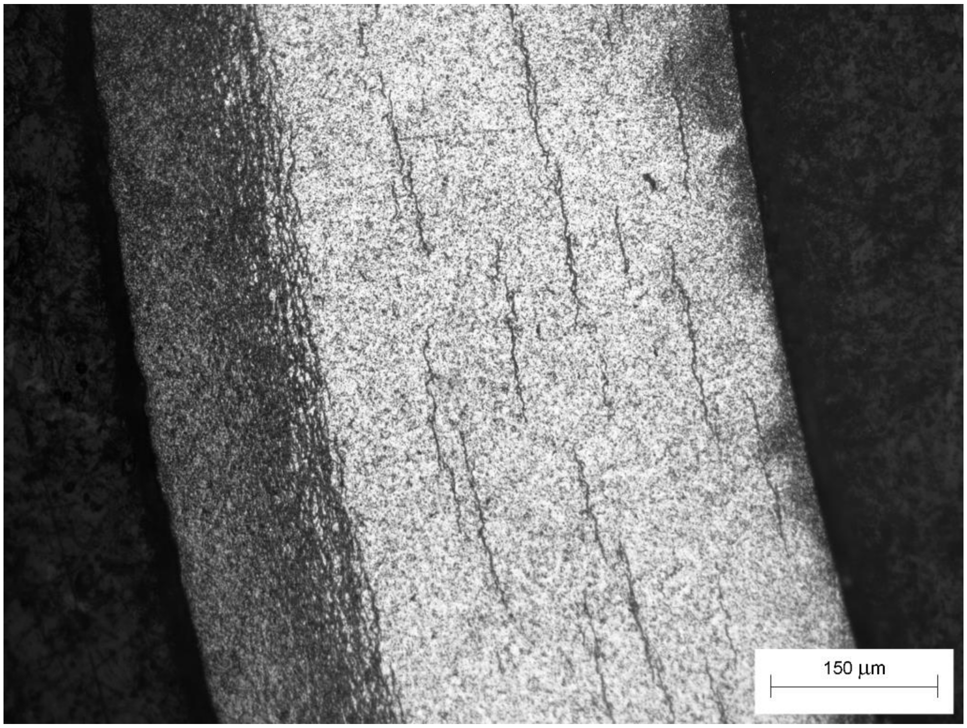
Sample 30 / Quadrant 2 / 200x Magnification



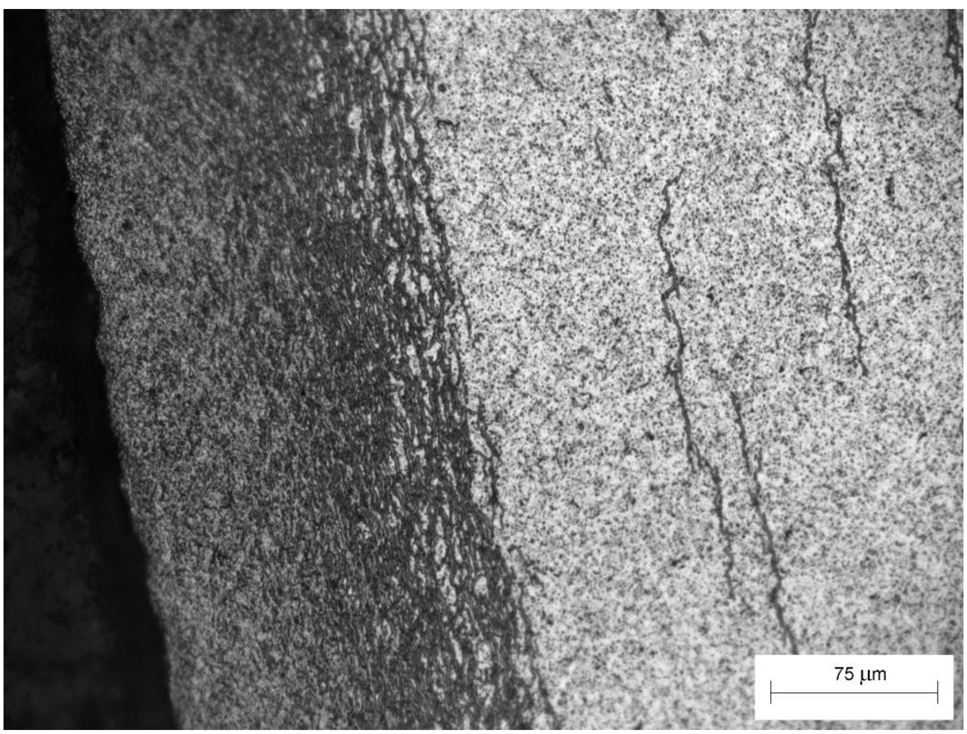
Sample 30 / Quadrant 3 / 100x Magnification



Sample 30 / Quadrant 3 / 200x Magnification



Sample 30 / Quadrant 4 / 100x Magnification



Sample 30 / Quadrant 4 / 200x Magnification

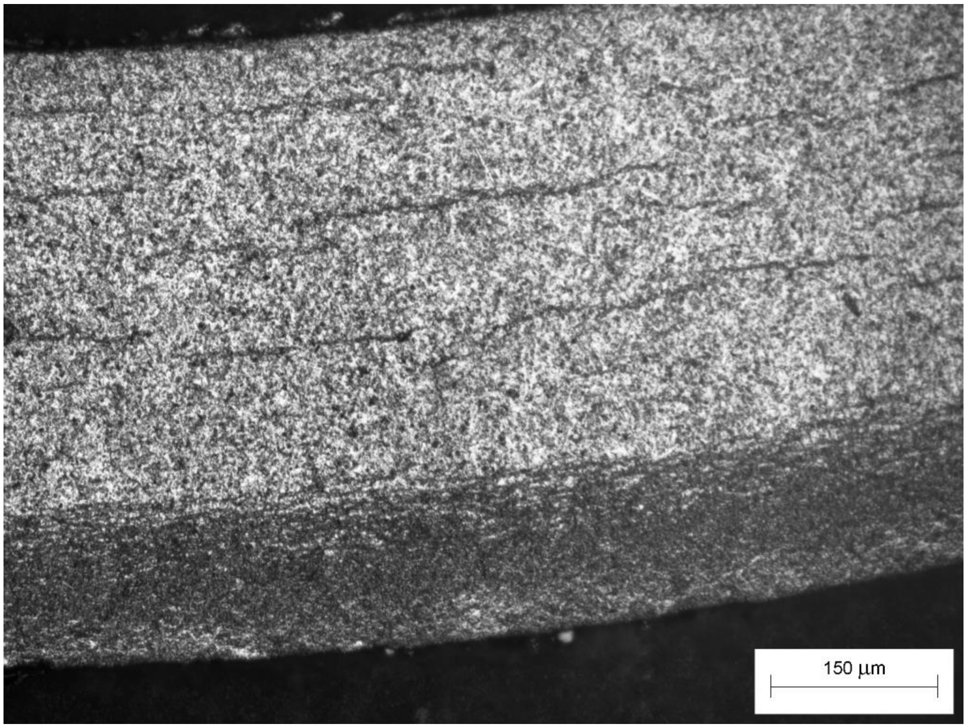
Sample ID: 31

Furnace: Thermolyne
Location: Flat
Temperature: 300°C
OD Surface: Blasted, 24 hr
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

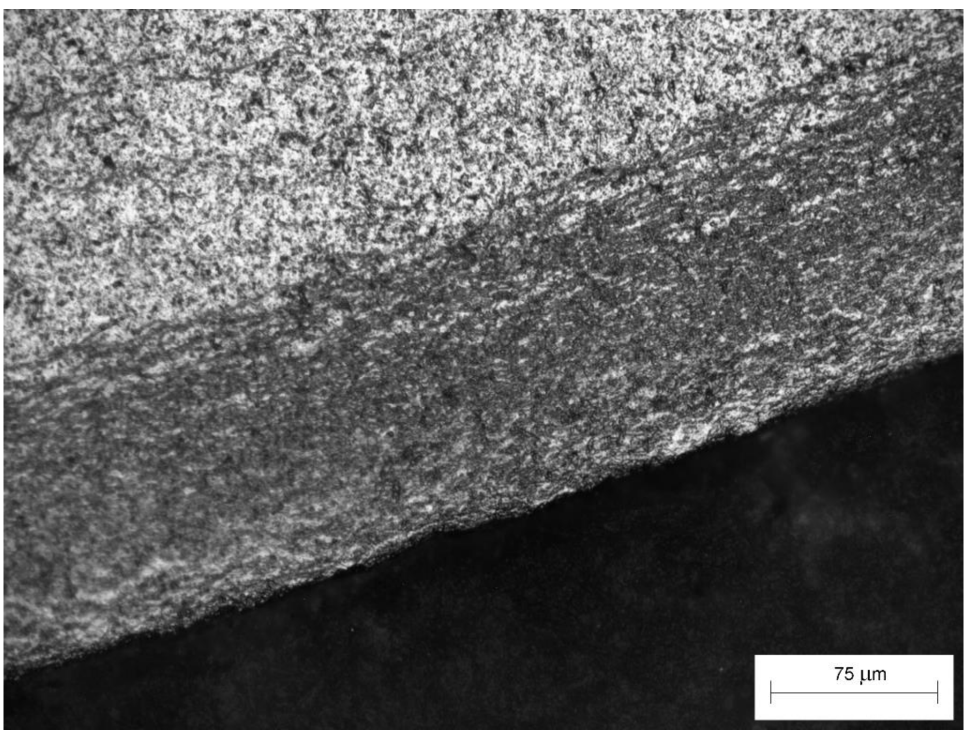
Hydrogen Concentration Results

Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	680	620	570	890	690	141	20%
Middle-2	890	410	670	1100	768	296	39%

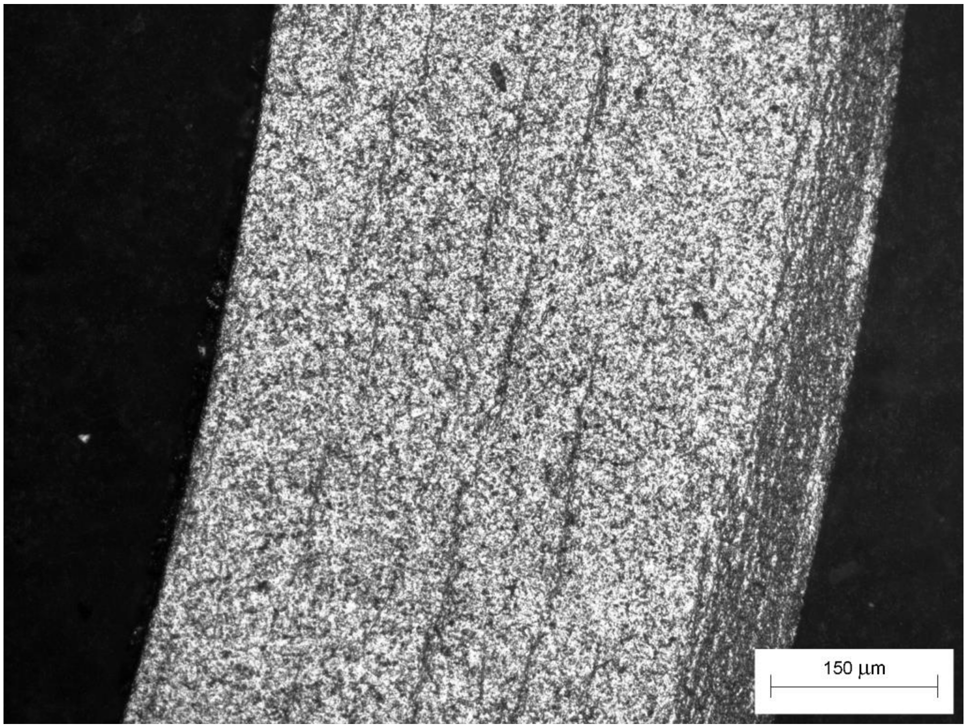
Images of Sample 31 After Hydriding and Mounted in Epoxy.



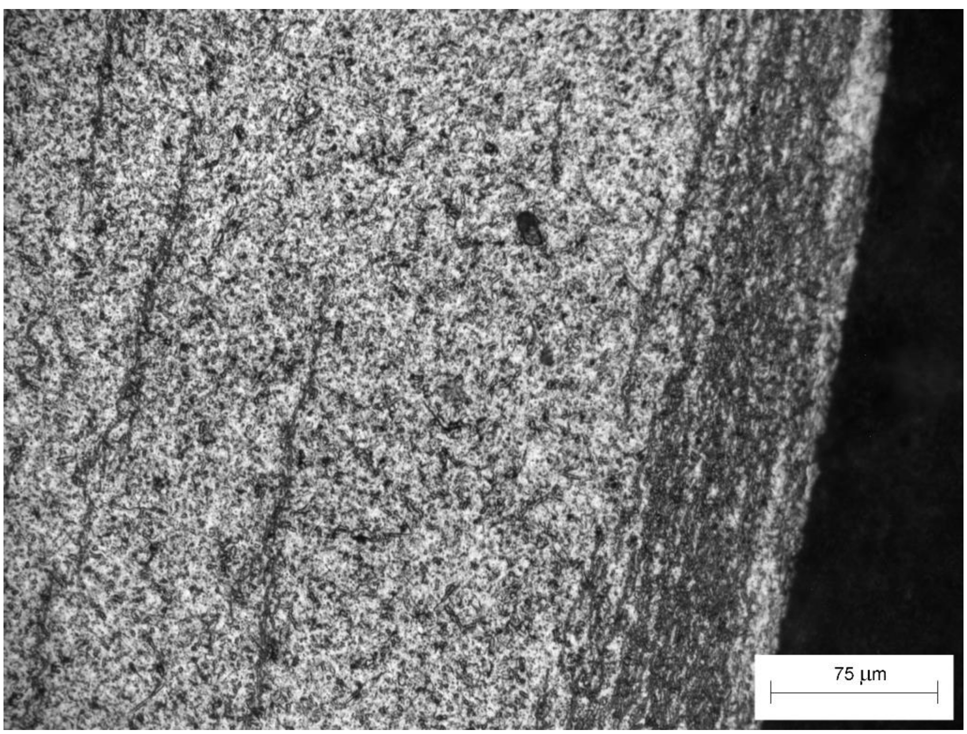
Sample 31 / Quadrant 1 / 100x Magnification



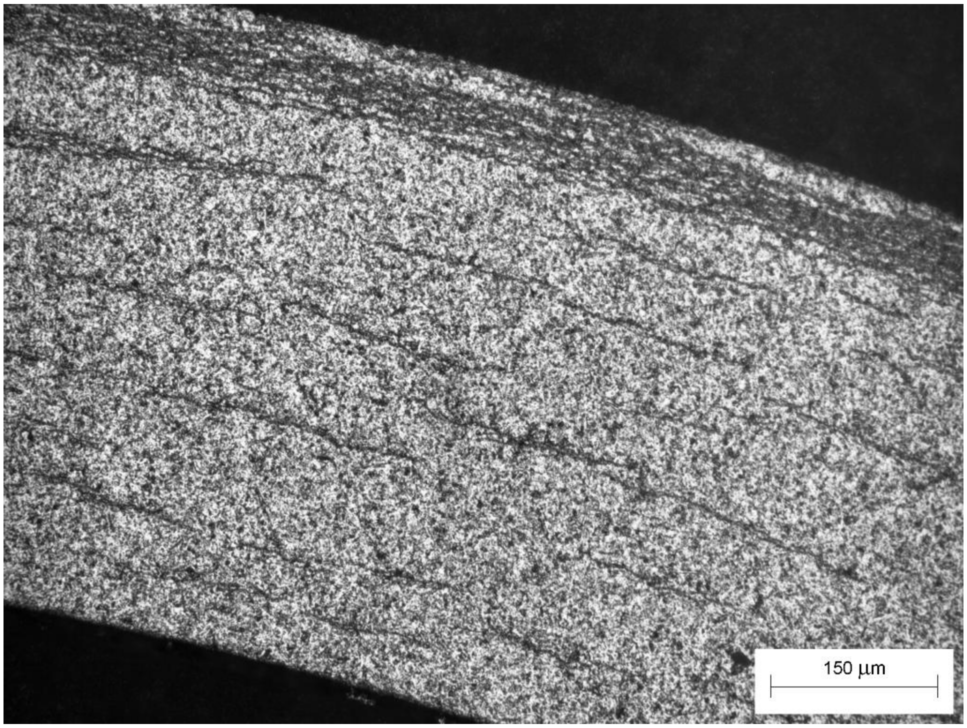
Sample 31 / Quadrant 2 / 200x Magnification



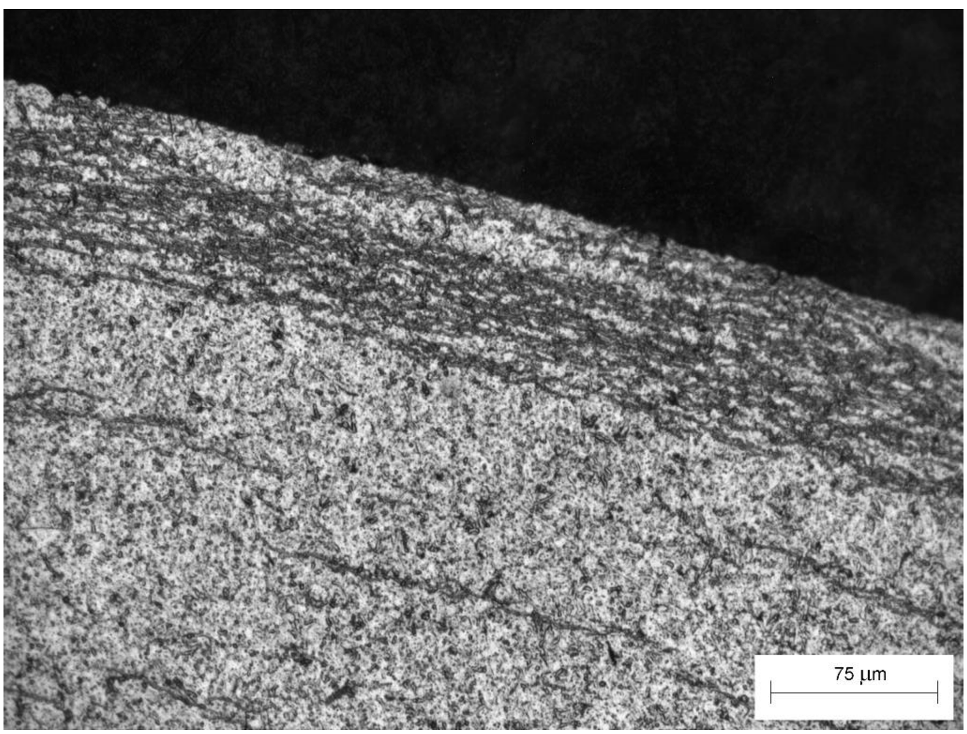
Sample 31 / Quadrant 2 / 100x Magnification



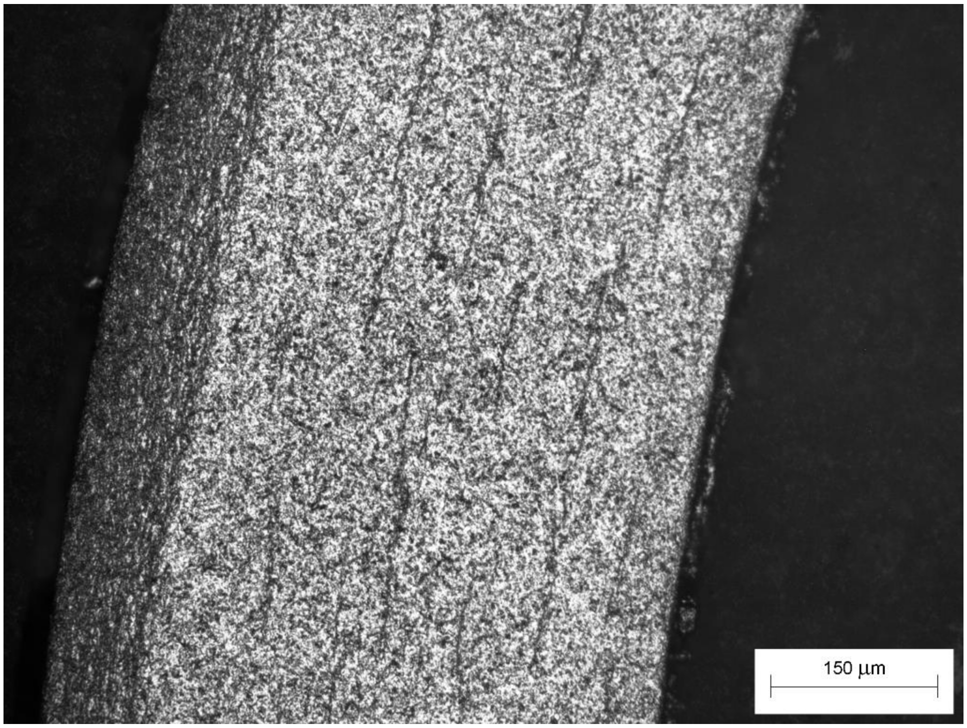
Sample 31 / Quadrant 2 / 200x Magnification



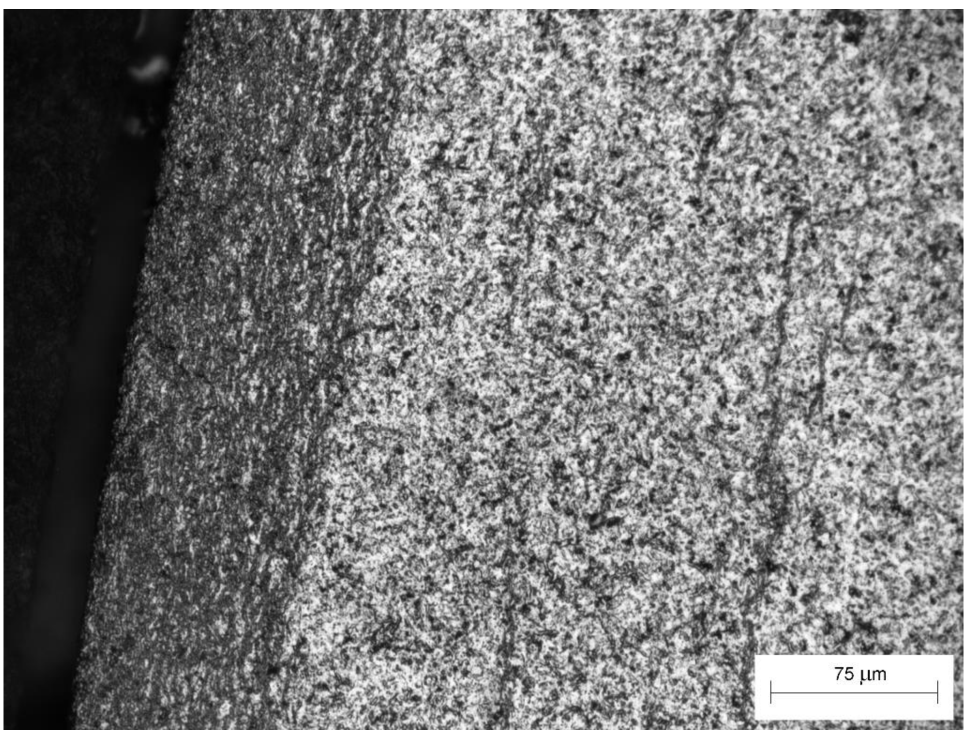
Sample 31 / Quadrant 3 / 100x Magnification



Sample 31 / Quadrant 3 / 200x Magnification



Sample 31 / Quadrant 4 / 100x Magnification



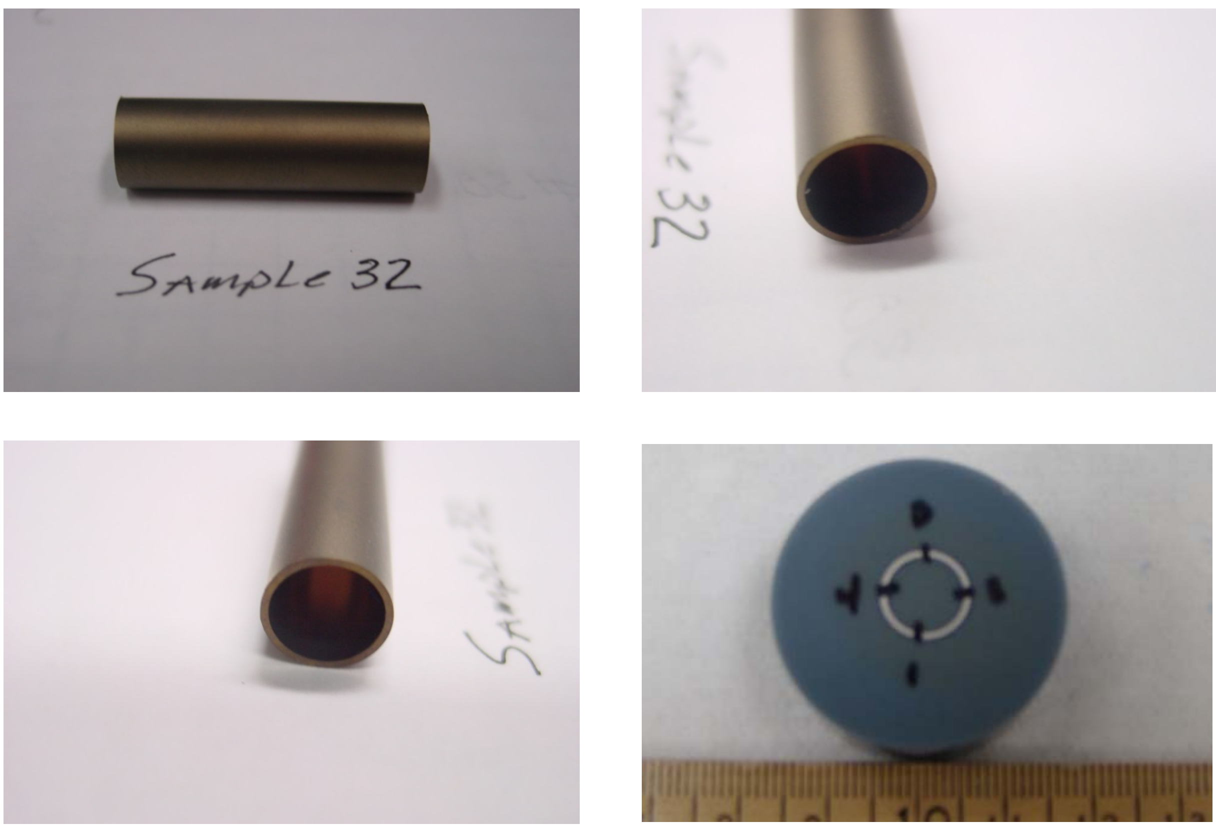
Sample 31 / Quadrant 4 / 200x Magnification

Sample ID: 32

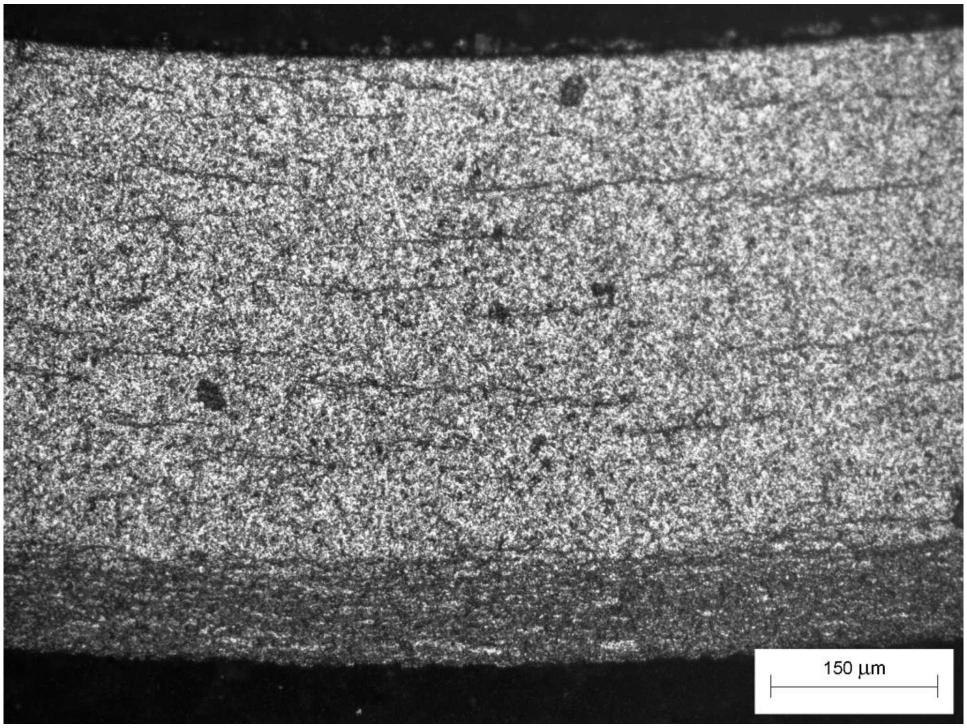
Furnace: Thermolyne
Location: Flat
Temperature: 300°C
OD Surface: Blasted
Outside Diameter: 0.372 in (9.5 mm)
Wall Thickness: 0.022 in (0.56 mm)

Hydrogen Concentration Results

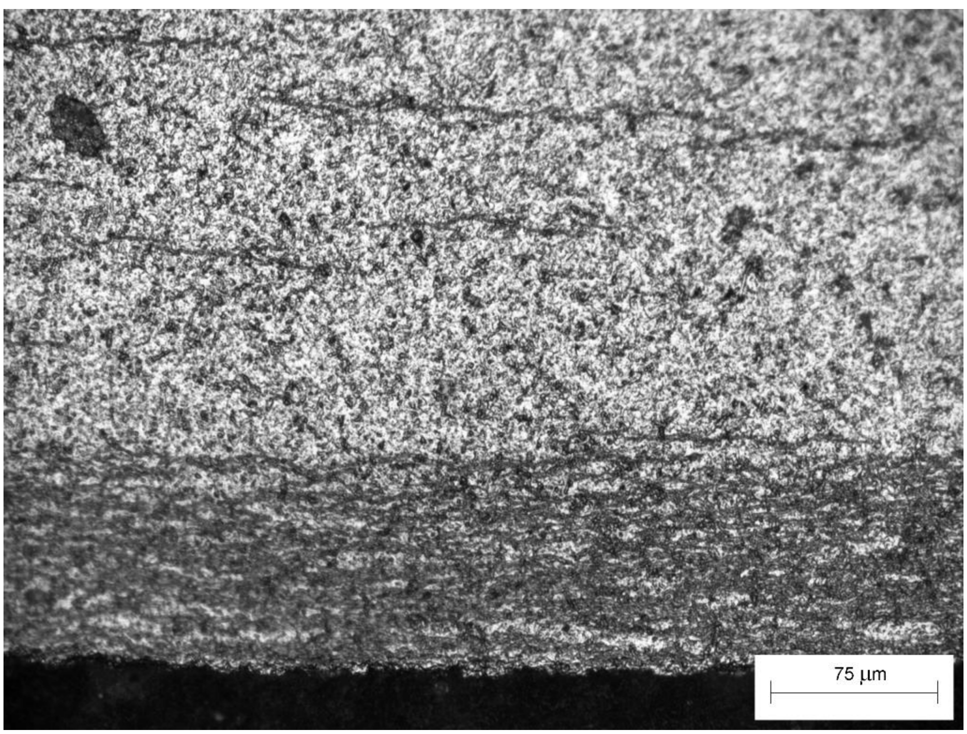
Sample Location	Hydrogen Concentration (wppm)						RPD (%)
	1	2	3	4	AVERAGE	STANDARD	
Middle-1	700	350	960	990	750	297	40%
Middle-2	500	840	910	1200	863	288	33%



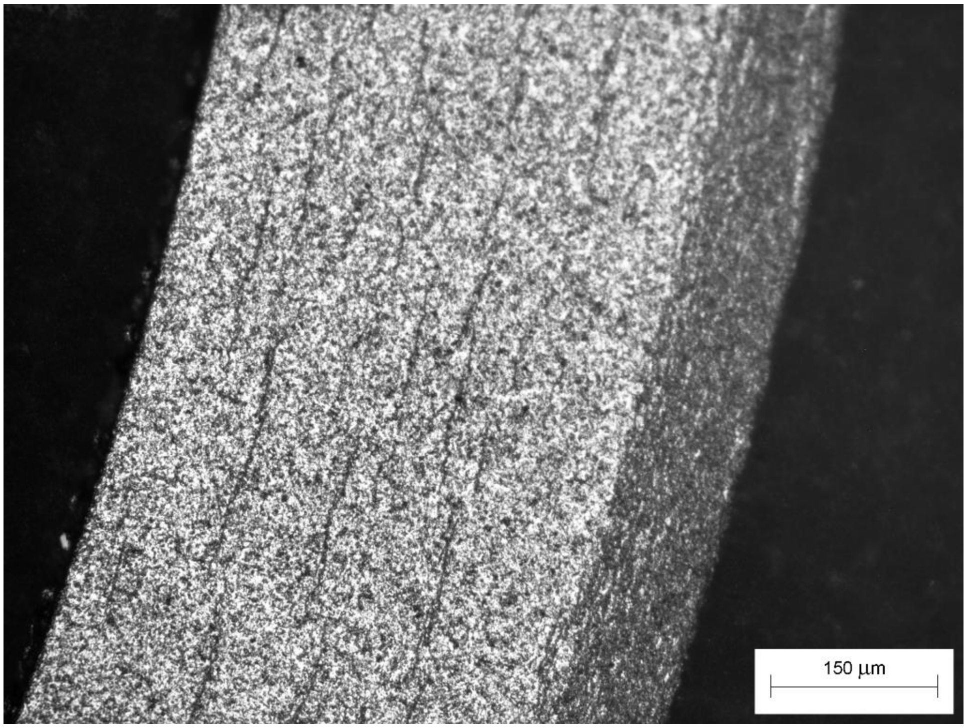
Images of Sample 32 After Hydriding and Mounted in Epoxy.



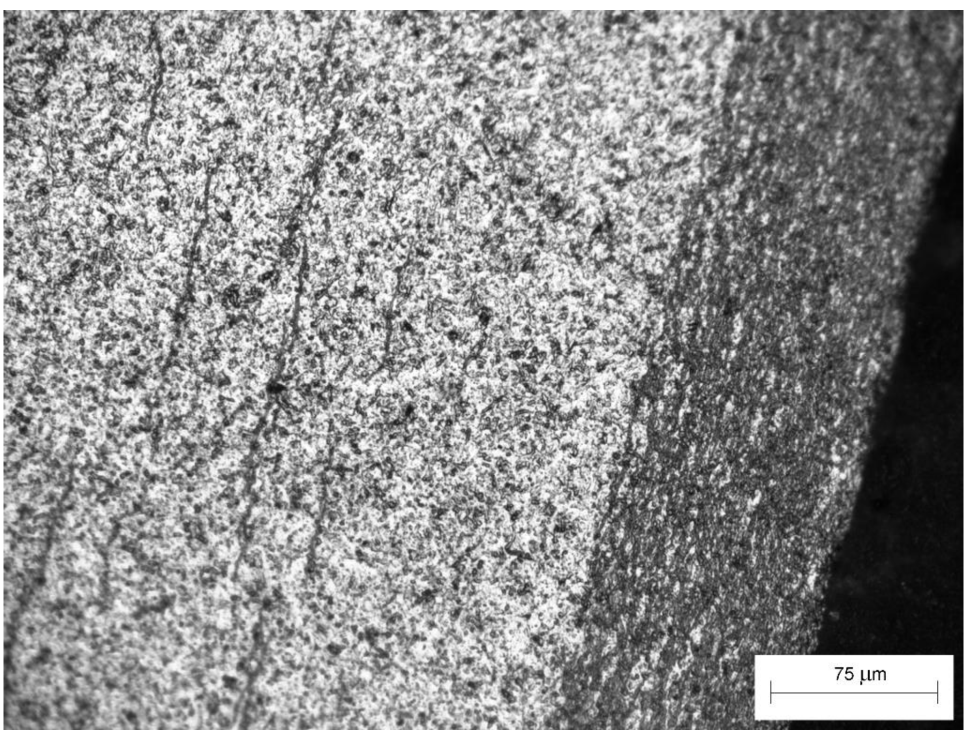
Sample 32 / Quadrant 1 / 100x Magnification



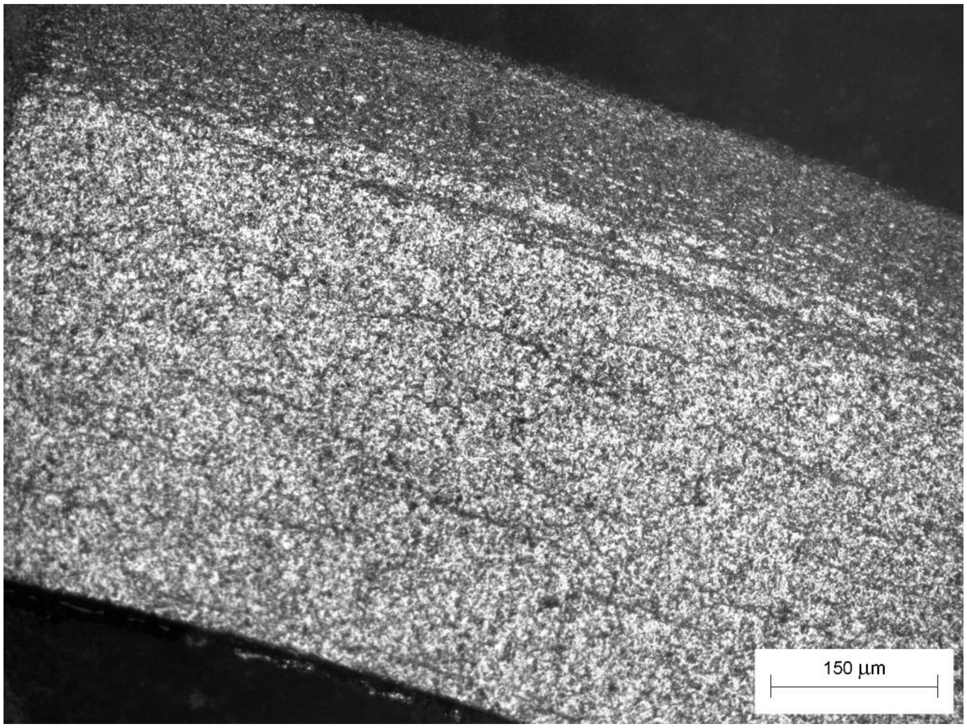
Sample 32 / Quadrant 1 / 200x Magnification



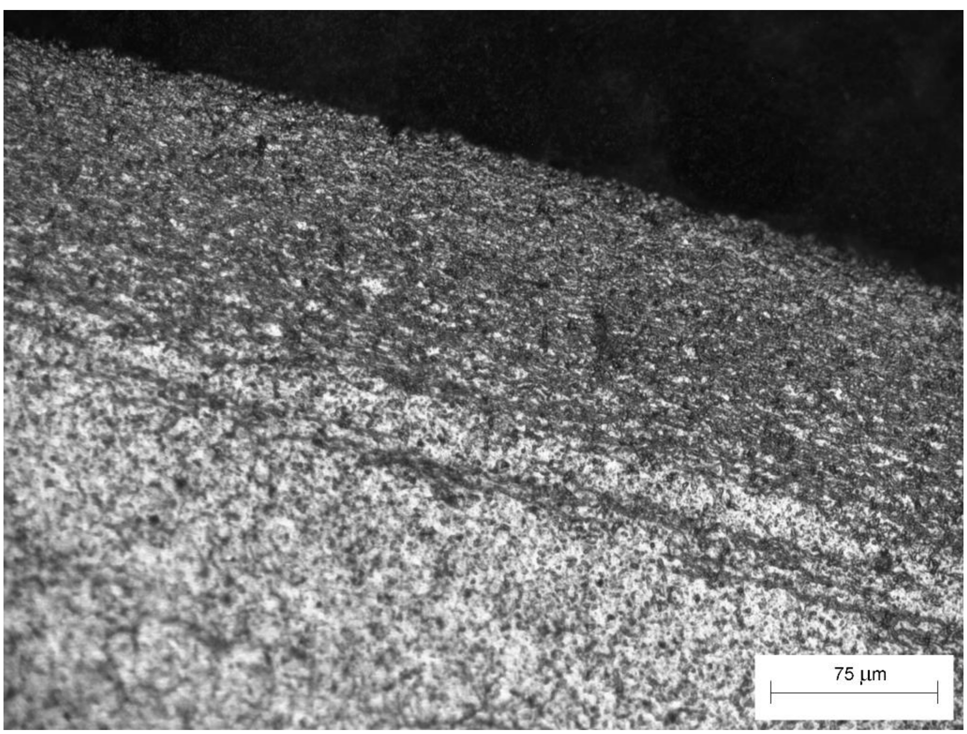
Sample 32 / Quadrant 2 / 100x Magnification



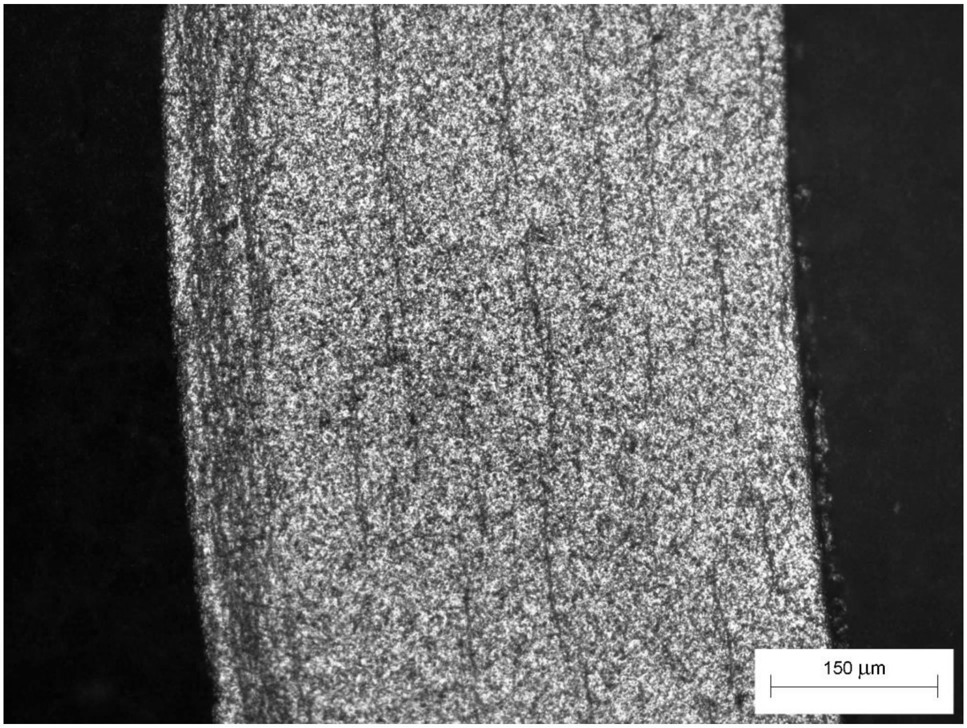
Sample 32 / Quadrant 2 / 200x Magnification



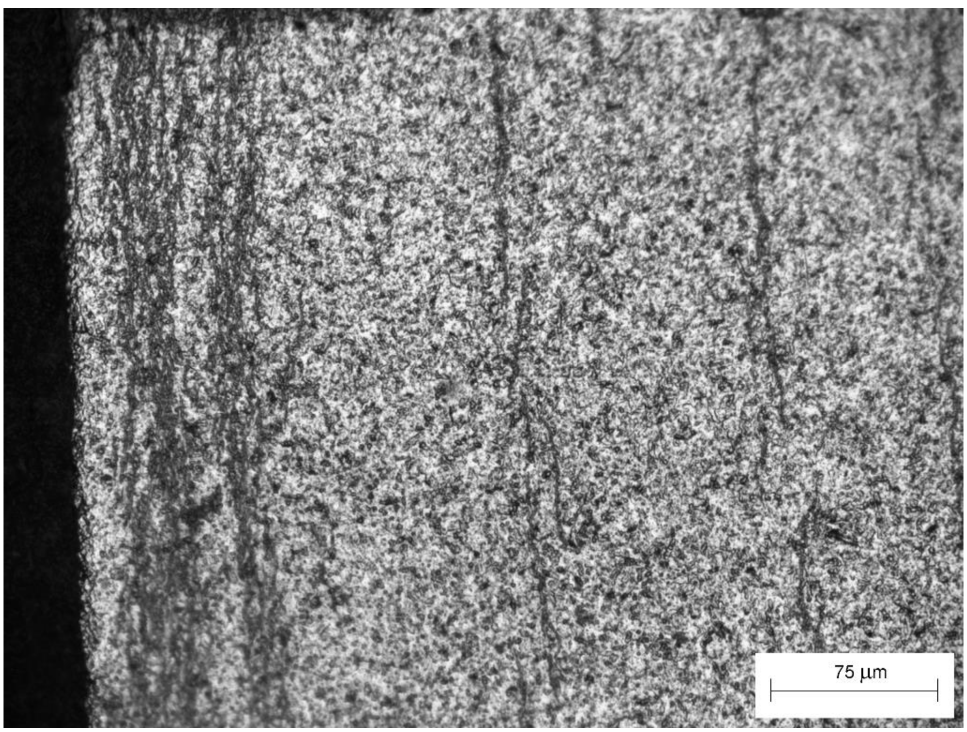
Sample 32 / Quadrant 3 / 100x Magnification



Sample 32 / Quadrant 3 / 200x Magnification



Sample 32 / Quadrant 4 / 100x Magnification



Sample 32 / Quadrant 4 / 200x Magnification