Processing and Characterization of Pilgered 14YWT Tubing

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Abstract

The Nuclear Technology Research & Development program of the Office of Nuclear Energy has implemented a program to develop an advanced reactor cladding fabrication capability by using the extrusion and pilgering processes. Oxide-dispersion strengthened (ODS) steels are a promising class of advanced materials, but processes need to be developed to produce tubing for these advanced alloys. The Pacific Northwest National Laboratory (PNNL) has converted its rolling mill into a lab-scale pilger mill, which required designing pilger tooling and rolls that contained multiple pilgering grooves and designing a servo-driven feed mechanism. Upon assembling and checking out the system, pilgering runs on stainless steel, MA956, and 14YWT iron (Fe)-based alloys were completed. Once the feed rates and rotation rates were established, the reduction schedules were determined. This report will focus on the processing and characterization of pilgered 14YWT thin-wall tubing that was processed from an extruded thick-wall tube using the PNNL laboratory pilger rolling mill. Characterization included optical metallography of the pilgered tube and microhardness of the processed material. The 14YWT characterization is being done in cooperation with researchers at Los Alamos National Laboratory (LANL), with LANL performing mechanical property testing of pilgered 14YWT and 14YWT tubes produced by hydrostatic extrusion at Case Western Reserve University (CWRU).

Acronyms and Abbreviations

14YWT	steel cladding material that has been used in liquid metal reactors
CWRU	Case Western Reserve University
OD	outer diameter
ODS	Oxide-Dispersion Strengthened
PNNL	Pacific Northwest National Laboratory
ROA	reduction of area

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1. EXECUTIVE SUMMARY

Pilgering is a promising technique for the fabrication of advanced alloys such as oxide-dispersion strengthened (ODS) cladding (MA956 and 14YWT) suitable for producing nuclear reactor-grade tubing. To that end, this project is using a lab-scale pilger mill located at the Pacific Northwest National Laboratory (PNNL). Pilger processes on stainless steel, MA956, and 14YWT iron (Fe)-based alloys were completed. The feed rates and rotation rates were established, the reduction schedules were determined. This report will focus on the processing and characterization of a pilgered 14YWT thin-wall tubing that began the process as an extruded thick-wall tube. Characterization included optical metallography and microhardness of the tube sections. The 14YWT characterization is being done in cooperation with researchers at Los Alamos National Laboratory (LANL), with LANL performing mechanical property testing of pilgered 14YWT and 14YWT tubes produced by hydrostatic extrusion at Case Western Reserve University.

2. PNNL LAB-SCALE PILGER MILL

PNNL has been focused on the development of a pilger processes for cladding fabrication with nanostructured ferritic materials such as 14YWT. To that end, PNNL has modified a rolling mill to perform lab-scale pilgering. Figure 2.1 shows the PNNL setup. Commercial pilgering is a large-scale operation, therefore PNNL's lab-scale pilger mill allows for the greater flexibility needed with research and development. The PNNL design has rolls with multiple pilger die grooves. This allows for different tube reduction ratios to be obtained without tool changes. The PNNL design is servo motor-driven versus mechanically-driven. This allows for incremental changes to the degree of die rotation as well as the tube feed rate and die rotation synchronization.

Figure 2.1. Modifications to the PNNL Rolling Mill for Lab-Scale Pilgering

3. PILGERING DESIGN METHODOLOGY

The conversion of the rolling mill to a pilger mill design allows for flexibility in the degree and speed of die rotation. The feed rate, rotation, and groove design all impact the resulting tube reduction of area (ROA). Consequently, it is possible for some of the grooves to be used for more than one of the reduction passes. For example, ROAs in the high 30s and low 40s may use the same groove, whereas the 11% ROA and the 77% ROA are drastically different than other ROAs, requiring their own groove designs. The pilger rolls developed contain six different grooves of similar design but with differing ROAs.

The flexibility inherent with this six-groove design permits multiple passes to be conducted without die changes. Furthermore, synchronization of the servo-die-feed rotation can be programed with software without the need for mechanical linkage modifications.

4. TUBE PILGERING PROCESS DEVELOPMENT

The development of the tube pilger process at PNNL utilized three Fe-based alloys. Initial process development runs were performed using 304 stainless steel, which is readily available from commercial suppliers. Following successful processing of the stainless steel, a second process development effort utilized available MA956 alloy. MA956 is a mechanically-alloyed Fe-based material that is oxide-dispersion strengthened (ODS) and thus similar to the 14YWT alloy that is the ultimate focus of this effort. Due to the limited quantities of suitable 14YWT alloy, it was the last material to be pilgered and utilized basic processing parameters developed from the MA956 reductions. Table 4.1 below lists the three Fe-based materials utilized in the tube pilgering process development effort.

Table 4.1. Tube Pilgering Development Materials with Initial and Finished Dimensions

Material	**Approximate Initial OD (in)**	**Approximate Initial ID (in)**	**Approximate Finished OD (in)**	**Approximate Finished ID (in)**
304 Stainless Steel	0.375	0.209	0.242	0.192
MA956	0.380	0.209	0.242	0.209
14YWT	0.380	0.211	0.242	0.209

5. TUBE PILGERING PROCESS DEVELOPMENT FOR 14YWT

The final development task for the pilgering process demonstration was the pilgering of an extruded thick-wall 14YWT starting tube, to a finished thin-wall tube having an OD of approximately 0.240 inch with a wall thickness of 0.020 inch. Following a similar ROA schedule to that of the MA956 tube, 14YWT was processed to the target finished dimensions using the PNNL tube pilger mill. As with MA956, the 14YWT was processed with an intermediate anneal to reduce hardness and increase ductility during the high overall ROA. Figure 5.1 shows the starting 14YWT thick-wall tube blank with the finished 14YWT thin-wall tube pictured below.

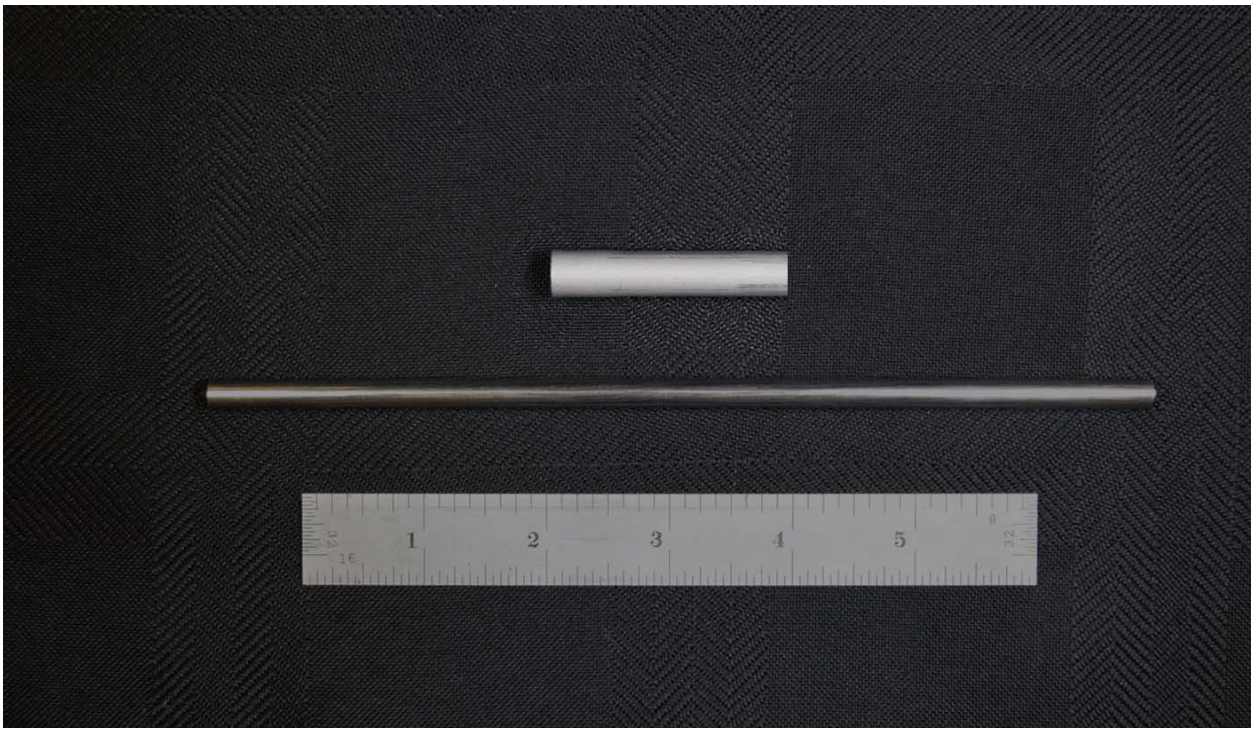

Figure 5.1. Photograph Showing 14YWT Starting Tube Blank (top) with Finished 14YWT Thin-Wall Tube (bottom)

6. OPTICAL METALLOGRAPHY CHARACTERIZATION OF PILGERED 14YWT TUBING

Optical microscopy was performed on a 14YWT solid billet as well as-extruded thick-wall and thin-wall pilgered tubes. The pilgered tube sections analyzed include intermediate, stress relieved, and finished ROA samples. The microscopy sample information is shown in Table 6.1.

Table 6.1. 14YWT Optical Microscopy Tube Sections

Condition	Approximate OD (inch)	Approximate ID (inch)
As-Received[1]	0.86”	N/A
As-Extruded	0.443	0.202
Heat Treated[2]	0.380	0.211
As-Pilgered	0.340	0.209
Heat Treated	0.340	0.209
As-Pilgered	0.300	0.209
Heat Treated	0.300	0.209
As-Pilgered	0.265	0.209
0.265 heat treatment sample was not analyzed		
As-Pilgered	0.242	0.209

A heterogeneous microstructure is present in the micrographs of the as-received billet, shown in Figures 6.1 and 6.2. The sample was taken as a thin section from the axial end of the billet. The heterogeneous structure present is well documented in 14YWT alloys.[1–3] The primary microstructure displays micron sized grains with sub-micron sized precipitates at grain boundaries. The secondary structure is of larger precipitates or agglomerates that do not appear to represent the same elemental composition of the primary structure.

[1] The as-received billet analyzed was not the billet that produced the final thin-wall tube.
[2] The extruded thick-wall tube used in the pilger process was not heat treated. It was pilgered as-extruded.

This heterogeneous structure is maintained in the micrographs of the extruded tube sections, shown in Figures 6.3 and 6.4 as well as the pilgered tube micrographs show in Figures 6.5-6.11. As expected, the extrusion process elongated both modes. This elongation is carried through the pilger process. Furthermore, the pilger process appears to concentrate the secondary structure near the tube mid-wall. This effect would be favorable with respect to resistance to radial crack propagation similar to that seen with zircoloy tube microstructures. [4]

Comparison of the as-pilgered and stress relieved tube micrographs, (Figures 6.5 and 6.6 for as-pilgered and Figures 6.7 and 6.8 for stress relieved), show that the intermediate anneals had no significant impact to the gross microstructure of the tube.

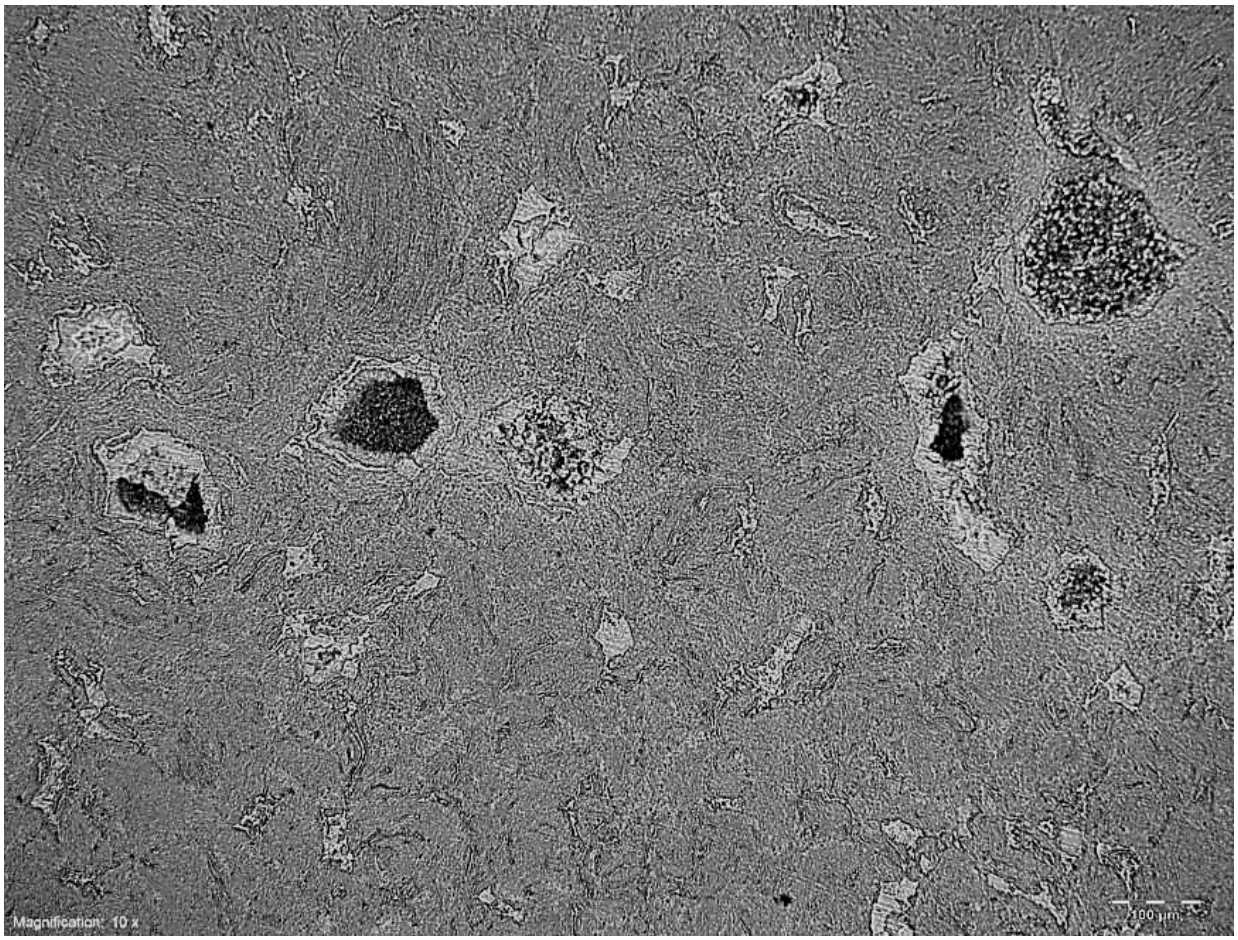

Figure 6.1. As-received 14YWT 0.86" OD (Billet)

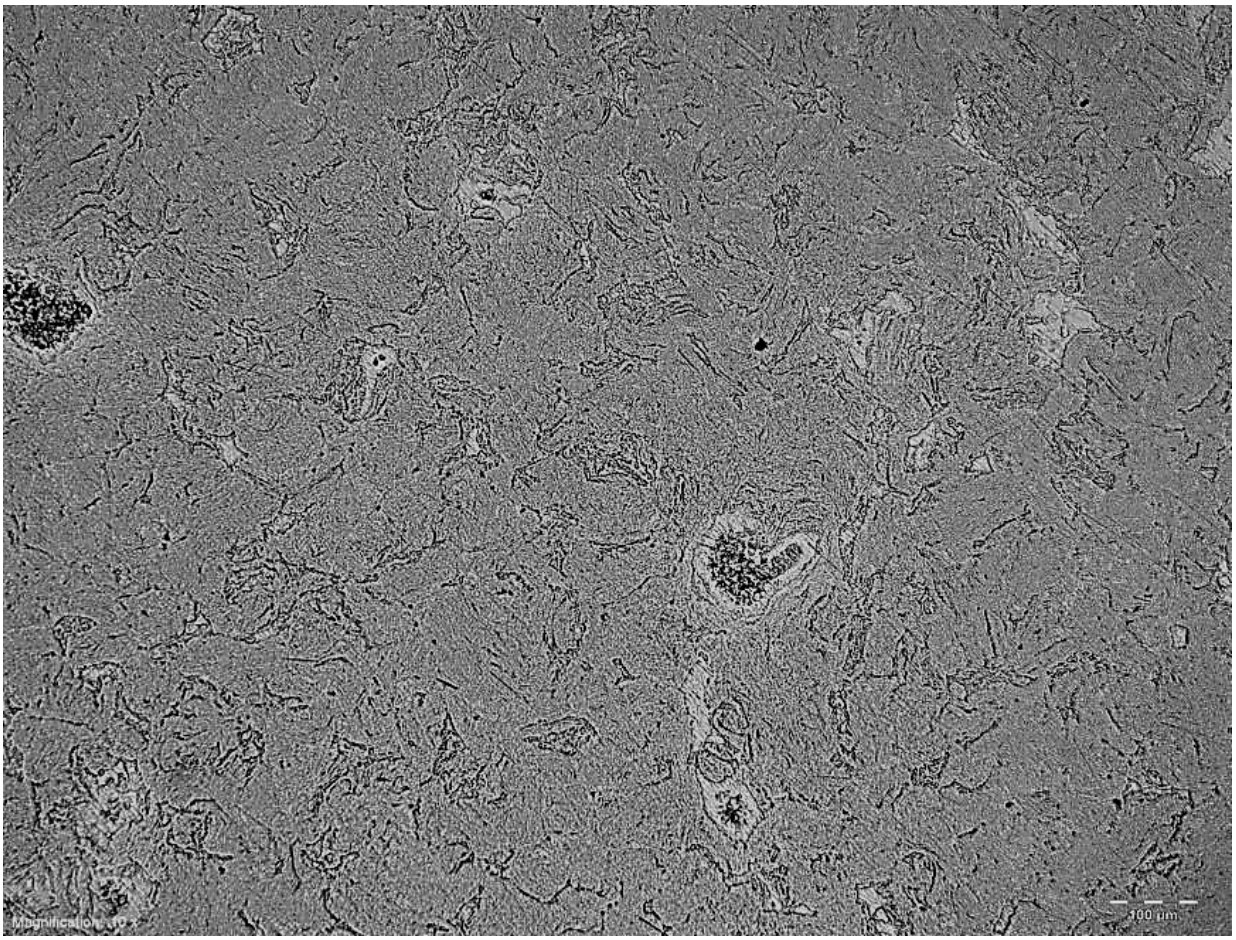

Figure 6.2. Annealed 14YWT 0.86" OD (Billet)

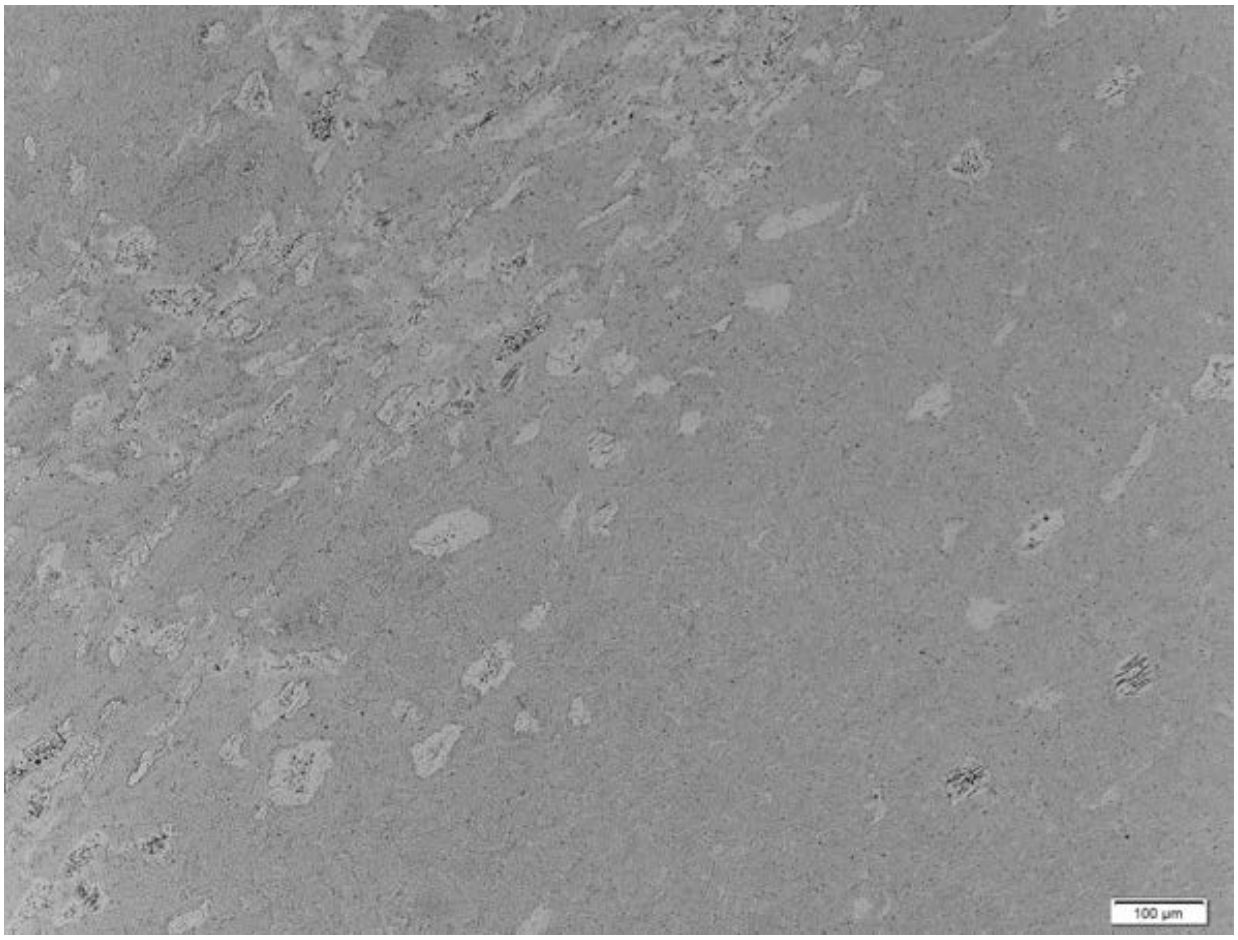

Figure 6.3. As-Extruded 14YWT 0.379" OD (Tube)

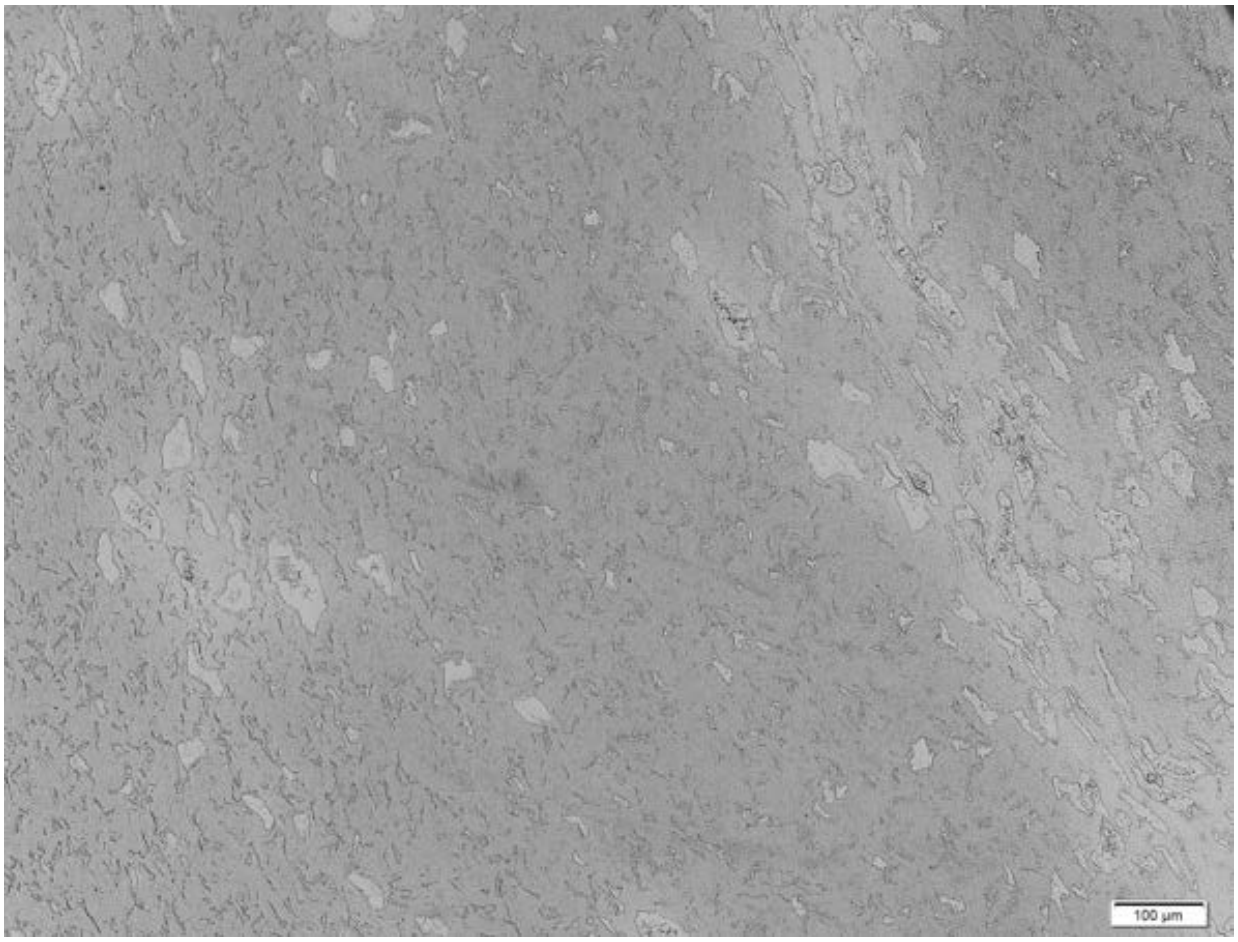

Figure 6.4. Heat Treated 14YWT 0.379" OD (Tube)

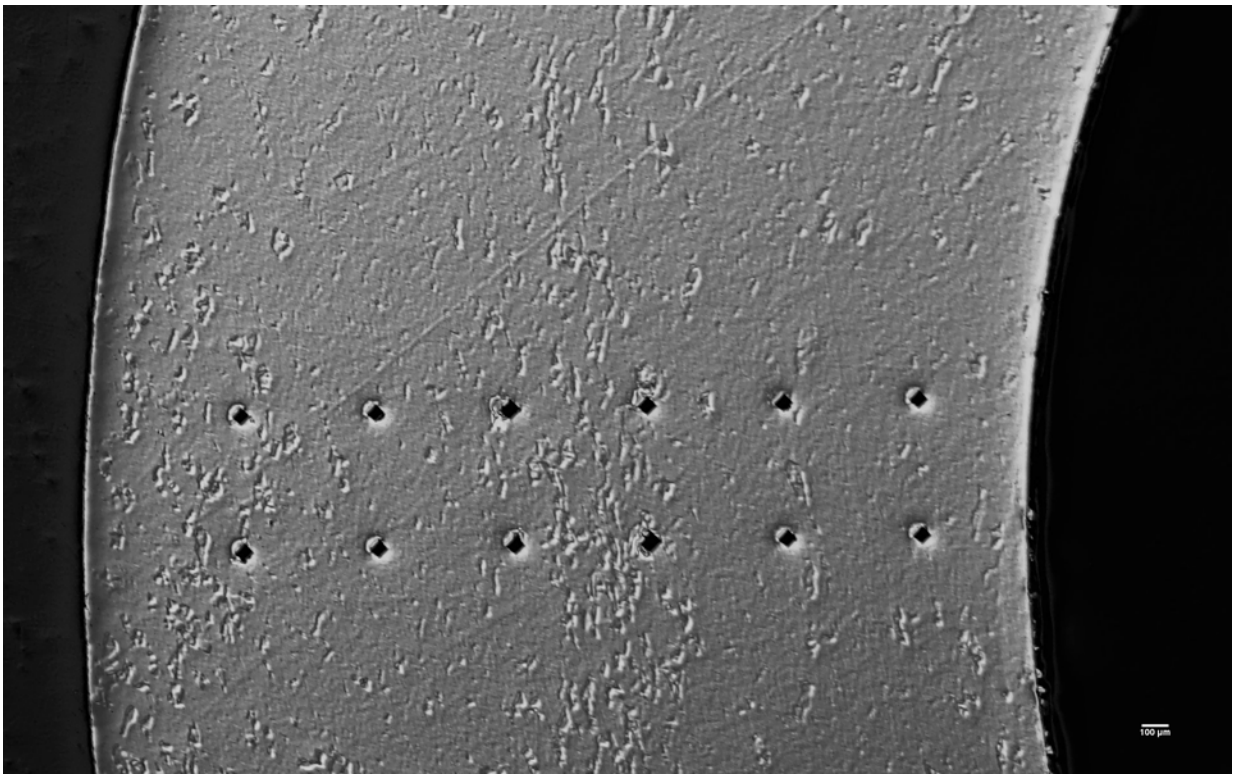

Figure 6.5. As-Pilgered 14YWT 0.340" OD (Tube)

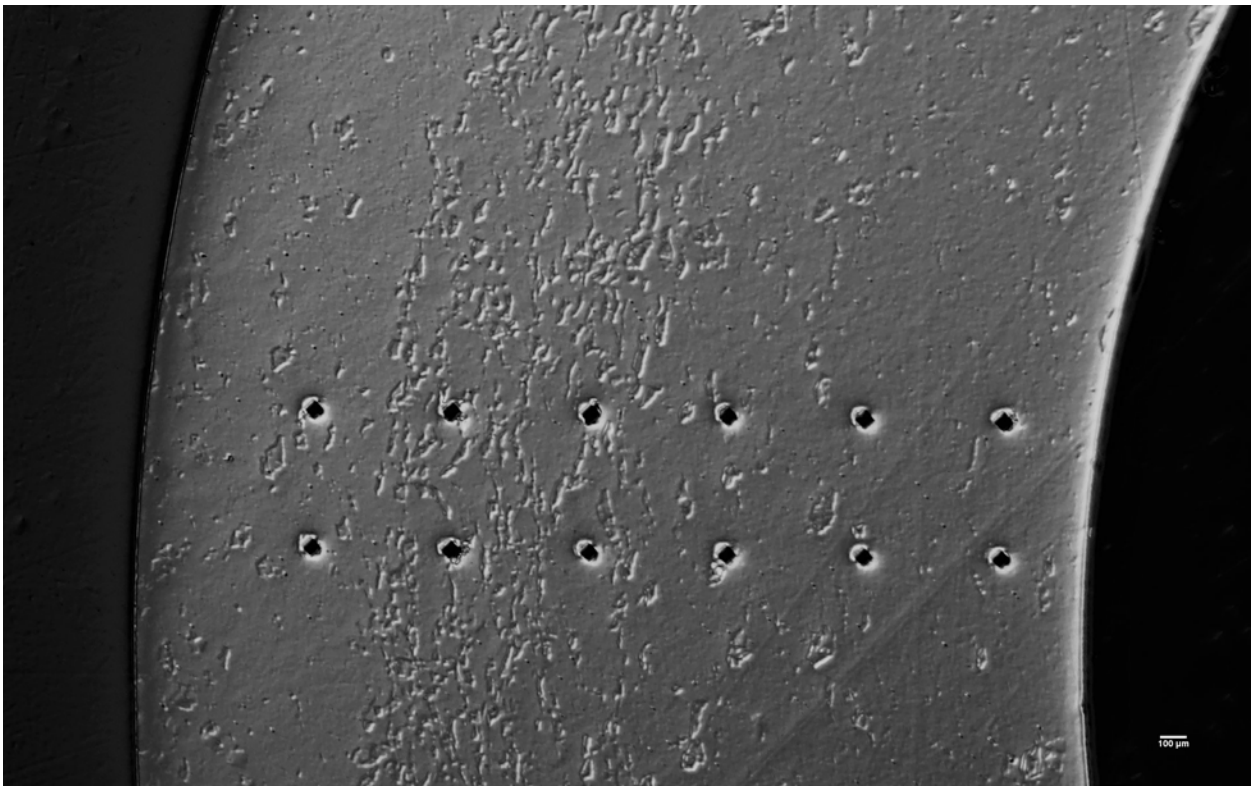

Figure 6.6. Stress Relieved 14YWT 0.340" OD (Tube)

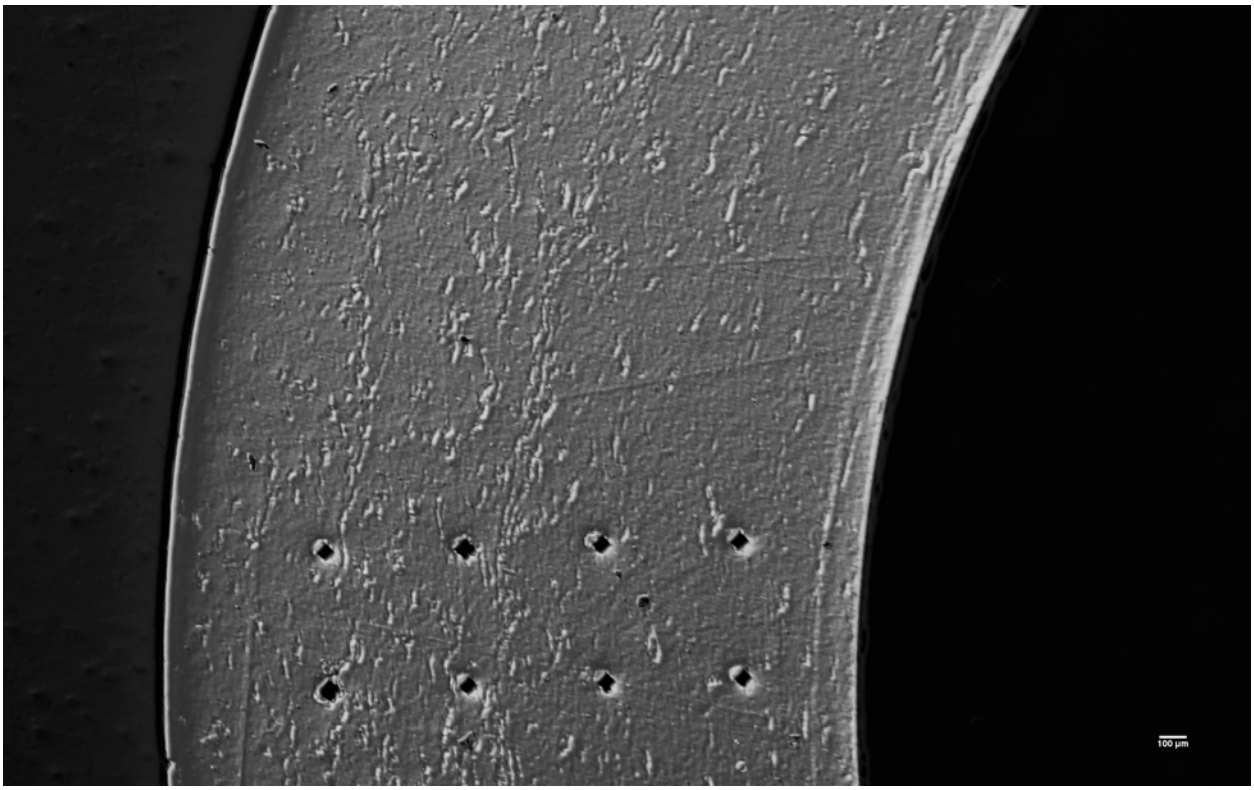

Figure 6.7. As-Pilgered 14YWT 0.300" OD (Tube)

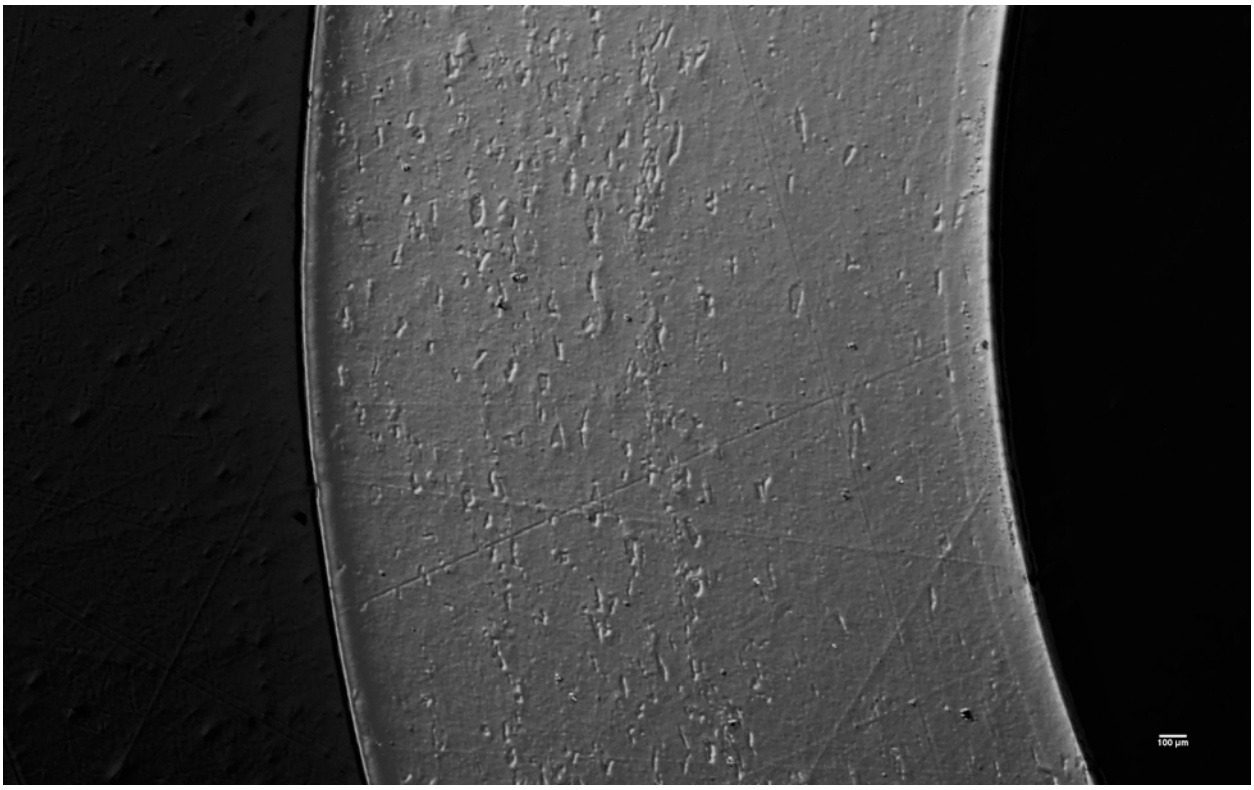

Figure 6.8. Stress Relieved 14YWT 0.300" OD (Tube)

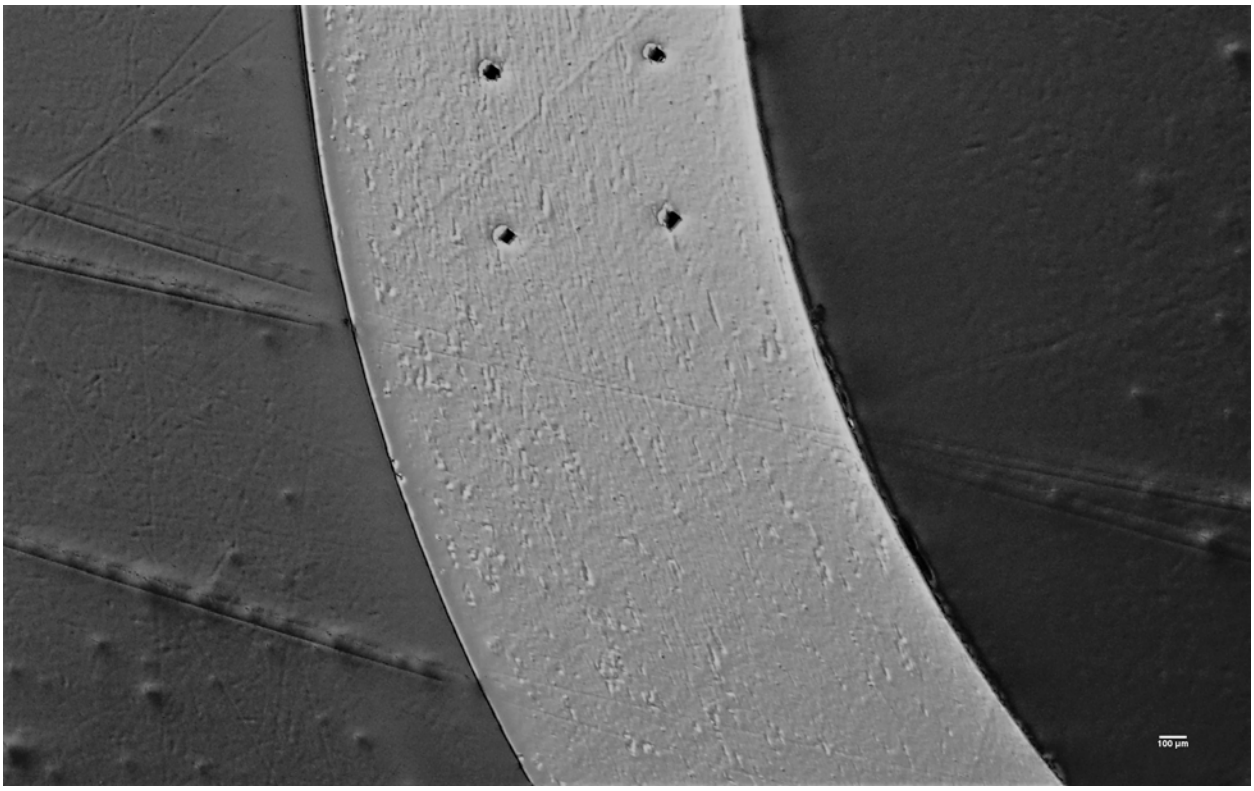

Figure 6.9. As-Pilgered 14YWT 0.265" OD (Tube)

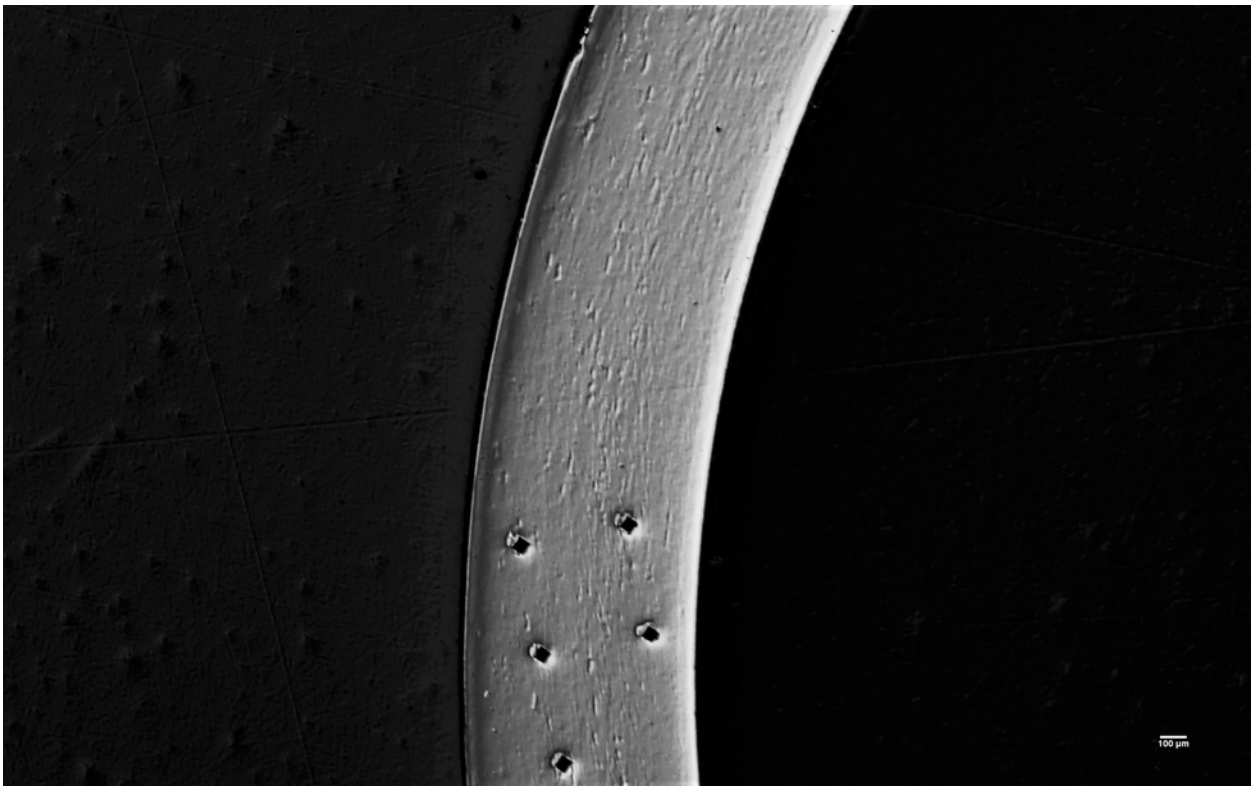

Figure 6.10. As-Pilgered 14YWT 0.242" OD (Tube)

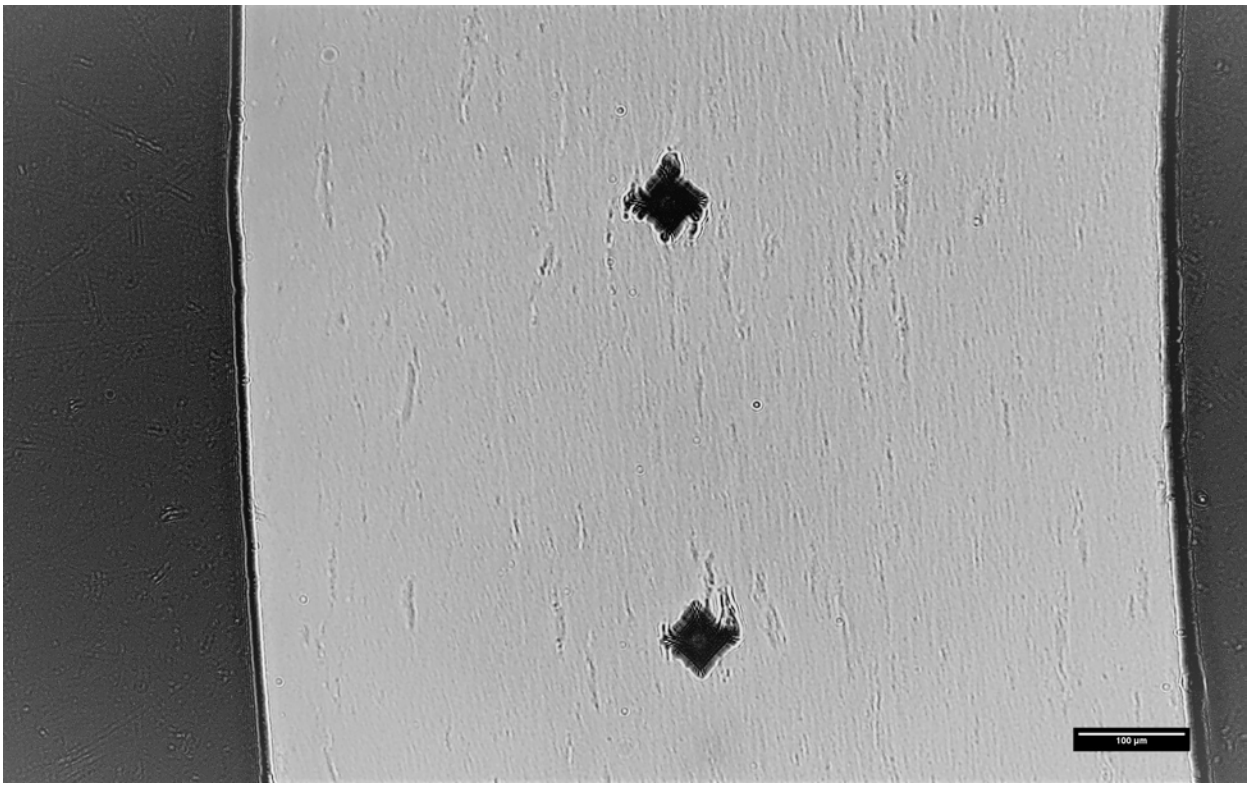

Figure 6.11. As-Pilgered 14YWT 0.242" OD (Tube)

7. MICROHARDNESS CHARACTERIZATION OF PILGERED 14YWT TUBING.

Microhardness (Vickers) measurements compliment the micrographs from Section 6.0. Table 7.1 provides the hardness results. As expected, the hardness increased from mid-300s to mid-400s Vickers with increased ROA. Furthermore, the intermediate stress relief treatments during the pilger process had a slight reduction in hardness; while the post-extrusion heat treatment had a slight increase in hardness. However, the hardness results are from a small data set.

Table 7.1. 14YWT Vickers Hardness

Condition	Approximate OD (inch)	Average Vickers Hardness
As-Received[3]	0.86”	368
As-Extruded	0.443	355
Heat Treated[4]	0.379	383
As-Pilgered	0.340	388
Heat Treated	0.340	397
As-Pilgered	0.300	386
Heat Treated	0.300	361
As-Pilgered	0.265	434
0.265 heat treatment sample was not analyzed		
As-Pilgered	0.242	445

[3] The as-received billet analyzed was not the billet that produced the final thin-wall tube.
[4] The extruded thick-wall tube used in the pilger process was not heat treated. It was pilgered as-extruded.

8. REFERENCES

(1) Pal, S.; Alam, M. E.; Odette, G. R.; Maloy, S. A.; Hoelzer, D. T.; Lewandowski, J. J. Microstructure, Texture and Mechanical Properties of the 14YWT Nanostructured Ferritic Alloy NFA-1. In *Mechanical and Creep Behavior of Advanced Materials*; Charit, I., Zhu, Y. T., Maloy, S. A., Liaw, P. K., Eds.; Springer International Publishing: Cham, 2017; pp 43–54.

(2) Alam, M. E.; Pal, S.; Fields, K.; Maloy, S. A.; Hoelzer, D. T.; Odette, G. R. Tensile Deformation and Fracture Properties of a 14YWT Nanostructured Ferritic Alloy. *Mater. Sci. Eng. A* **2016**, *675*, 437–448. https://doi.org/10.1016/j.msea.2016.08.051.

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