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U Metal Oxidation with Steam (U)

R. A. Pierce

A. J. Duncan

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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) is evaluating a method to produce uranium dioxide (UO_2) from uranium metal using steam. The typical methods for making UO_2 yield powders with particle-size ranges of 1 to 50 micron. The goal of the steam oxidation is to produce a depleted uranium (DU) product with a larger particle size that can be used as a surrogate for testing physical and mechanical process techniques to refine uranium and/or plutonium bearing residues.

Several studies in the literature indicated that reactions between uranium metal and steam will yield UO_2 as fine powders in the 160 to 500 °C range, but granular materials form above 500 °C. Above 880 °C, a hard compact scale forms on the U metal. Furthermore, sintering of UO_2 occurs at about 1300 °C, but it has been stated that the presence of steam enhances the sintering characteristics of UO_2.

Initial studies into the oxidation of DU metal in an atmosphere of argon and steam have been completed. Oxidation of depleted uranium metal in Ar/steam at 600, 710, 770, and 830 °C yielded a UO_2 product with little or no impurity at each temperature. Although not enough sample was available to perform sieve analysis for particle size distribution, SEM images reveal that a significant volume fraction of the products at 710, 770, and 830 °C exceeds 100 microns. The material produced at 600 °C was too fine for programmatic needs.

Questions associated with the UO_2 include morphology of product, mechanical strength of the particles, and performance in milling operations. The size of the samples produced in these tests did not allow for quantitative assessment of particle size or mechanical strength. A qualitative assessment of the particles formed at 710, 770, and 830 °C was that the samples produced at 710 and 770 °C were friable, and the sample produced 830 °C had more mechanical strength. Sintering of the samples in Ar/steam at 970 °C appeared to increase the strength of the particles without changing their general particle size characteristics. Due to the small sample sizes, the increase in strength could not be quantified. If the sintering at 970 °C is inadequate, higher sintering temperatures in Ar/steam can be evaluated.

Future work will increase scale of the steam oxidation and refine metal oxidation conditions which yield the most-favorable particle-size distributions with sufficient mechanical strength for programmatic usage. Further consideration will be given to oxidation temperature, steam addition to the reaction vessel, U metal pretreatment, and sintering conditions after oxidation. The UO_2 production could involve a two-step process where particles are formed in steam at lower temperatures (700-850 °C) and then sintered in Ar/steam at elevated temperatures (950-1200 °C). Larger samples (50-100 g each) will provide sufficient UO_2 product for sieve analysis and assessment of mechanical strength relative to subsequent milling operations.

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LIST OF ABBREVIATIONS

CSEM	contained scanning electron microscope
DU	depleted uranium
OGE	off gas exhaust
SEM	scanning electron microscopy
SRNL	Savannah River National Laboratory
XRD	X-ray diffraction

1.0 Introduction

The Savannah River National Laboratory (SRNL) is exploring a method to produce uranium dioxide (UO_2) from uranium metal using steam. The typical method for making UO_2 involves either converting UF_6 gas to UO_2 or reducing UO_3 to UO_2 with hydrogen gas. However, the product UO_2 from both processes and other common synthesis methods is a powder with a particle-size range of 1 to 50 micron [1]. The powder is suitable for pressing into fuel pellets, but it is not satisfactory for an alternate programmatic need. The goal of the steam oxidation is to produce a depleted uranium (DU) product with a larger particle size that is needed as a surrogate for testing physical and mechanical process techniques being developed at ORNL for refining uranium and/or plutonium bearing residues [2].

2.0 Background

The literature offers several studies of U metal oxidation by steam. The articles provide similar perspectives on the nature of the oxide formed, but still with significant variation. Wilson, et. al report that the oxide formed at 600 °C and above was a glossy black UO_2 which did not flake off until the samples were cooled after exposure. Oxide formed at 400 °C was a brown colloidal material that continually released from the sample, with a transition occurring near 500 °C [3].

Hopkinson observed that from 200 to 500 °C the reaction product is in the form of a fine powder, but above 500 °C granular particles are formed [4]. Above 880 °C, a hard compact scale forms on the U metal. Uranium dioxide was the product formed over almost the entire temperature range from 200 to 1400 °C. He also mentions that normal sintering of UO_2 occurs at about 1300 °C, but it has been suggested that the presence of steam enhances the sintering characteristics of uranium dioxide. Unfortunately, the reference for the enhancing effects of steam was not in a public medium but in a private communication.

Hayward, et. al states that the oxide layers produced on foil samples below 400 °C had highly porous cellular structures while the layers formed at higher temperatures were significantly denser [5]. Non-adherent oxide layers were formed on bar samples below ~300 °C, and they become increasingly adherent at higher temperatures. No cellular porosity was observed in the oxide layers from sample heated above 300 °C. A tightly packed granular oxide layer with occasional cracks and fissures was formed at 400 °C while the product at 500 °C consisted of a heavily fissured oxide layer. The X-ray diffraction (XRD) peaks recorded for U-pellet samples became progressively narrower with increasing oxidation temperature, indicating increasing grain size and/or diminishing defect density in the oxide formed at higher temperatures.

Wilson, et. al noted that at 400 °C, the reaction rate is linear, but that a parabolic rate describes the oxidation rate between 600 °C and 1200 °C [3]. Hopkinson reports a linear rate law from 200 to 880 °C with maximum rates at ~300 °C and 750 °C [4]. Above 880 °C, a parabolic rate law applies while a protective oxide layer forms, after which the rate is linear. Hayward et. al indicate that below 450 °C, the thermogravimetric traces were always approximately linear [5]. Above 450 °C, the rates were quasi-linear for the first 10-15 minutes before becoming linear.

3.0 Experimental Procedure

Two steps were needed to perform U metal oxidation. First, the U metal pieces in storage were cleaned of surface oxide expected to be U_3O_8 and machining oils. Figure 3-1 shows the metal turnings inside of a plastic bag with a black oxide coating on them. Turnings that were cleaned are exhibited in Figure 3-2. The metal that went through the cleaning process was used for oxidation to UO_2. It should be noted, however, that some of the oxide coating grew back onto the metal between cleaning and steam oxidation.

3.1 Uranium Metal Cleaning

1) Lay out a towel for drying the turnings.
2) Using tongs, place the U metal turnings into the empty beaker labeled “cleaning”.
3) Pour 100 mL of deionized water into the beaker labeled “rinse”.
4) Add the 5 M HNO_3 to the “cleaning” beaker with the U metal turnings.
5) Allow the turnings to remain in the acid for about five minutes or until oxide scale is removed to a satisfactory degree.
6) Using the tongs, remove the turnings from the acid and add them to the “rinse” beaker.
7) Swirl the turnings in the water to rinse the residual 5 M HNO_3.
8) Using the tongs, remove the turnings from the “rinse” beaker and place them on the towel.
9) Rinse DU metal piece(s) with acetone or alcohol.
10) Dry with the towel.
11) Store in a container until used for testing (Section 3.2).

Figure 3-1. Metal Turnings from Mark-31 Slugs in a Bag

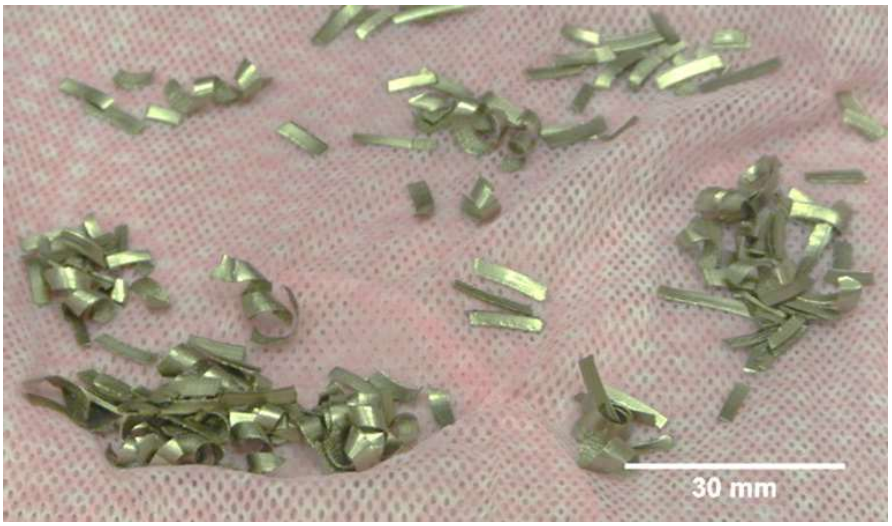

Figure 3-2. Cleaned Metal Turnings from Mark-31 Slugs

3.2 Uranium Metal Oxidation with Steam

1) Obtain cleaned DU metal samples from storage.
2) Weigh the ceramic sample boat and record its weight.
3) Weigh sample, record the weight, and load the sample into the ceramic boat.
4) Configure furnace setup as seen in Figure 3-3 and Figure 3-4.
5) Configure the furnace controller per guidance from the principal investigator (PI).
6) Load sample(s) from the exhaust (left) side of the furnace and connect flange (Figure 3-5).
7) Connect vacuum line and evacuate furnace tube for 30 minutes.
8) Turn on the pump that draws vapors from the furnace and delivers them to the exhaust.
9) Pull vacuum on reaction tube and backfill with argon. Do this three times.
10) Pull vacuum again and hold the system under vacuum.
11) Program and initiate ramp cycle on furnace.
    a. Heat the furnace to 450 °C at a ramp rate of 10 °C/min.
    b. Turn on the heating mantle for generating steam (Figure 3-6).
    c. Hold the furnace temperature at 450 °C for 30 minutes.
    d. Backfill the furnace process tube with argon such that bubbles are visible in exhaust bubbler.
    e. Ramp furnace to desired temperature (_____ °C) at 10 °C/min.
    f. Turn on pump to transfer water from the graduated cylinder to the glass vessel on the heating mantle at a feed rate of 5-10 mL/h.
    g. Hold the furnace at desired temperature (_____ °C) for 180 min (or as directed by PI).
12) Turn off the water feed pump but leave the heating mantle on.
13) Ramp furnace down to below 100 °C.
14) Turn off the heating mantle.
15) The following day, turn off the argon purge.
16) Remove the ceramic boat from the furnace.
17) Weigh the sample boat with the oxidized U metal and record the weight.
18) Carefully transfer the oxidized DU metal into a sample jar.
19) Weigh the empty sample boat and record the weight.
20) Weigh subsamples and transfer into plastic bottles (~0.3 g for XRD and ~1 g for scanning electron microscopy [SEM]).

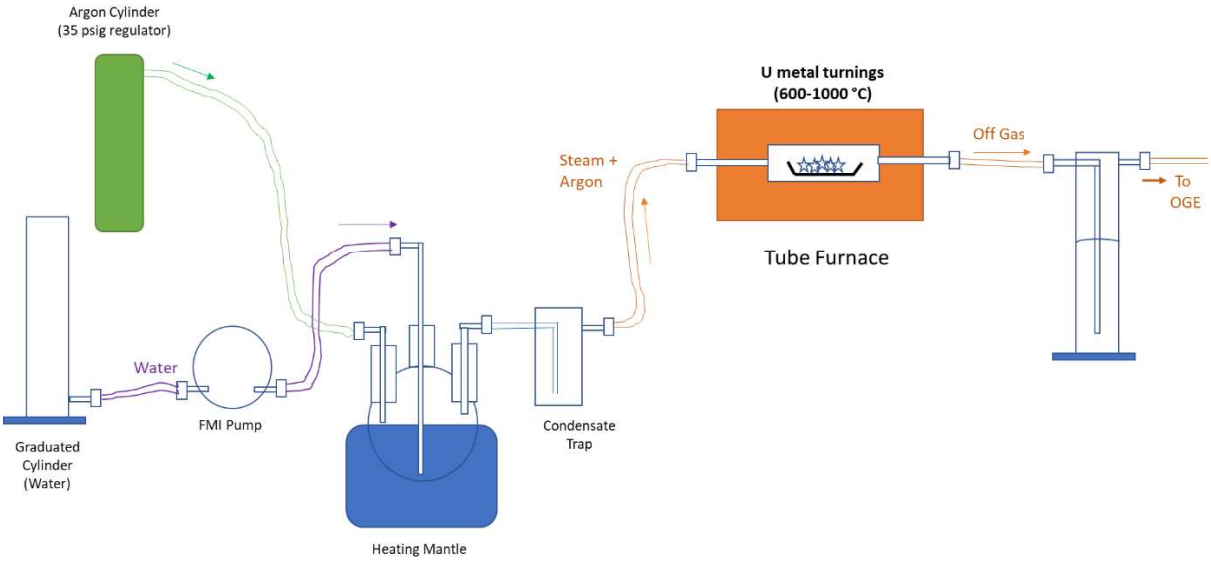

Figure 3-3. Equipment Configuration

Figure 3-4. Test Equipment in the Laboratory

Figure 3-5. Sample Being Removed from the Furnace

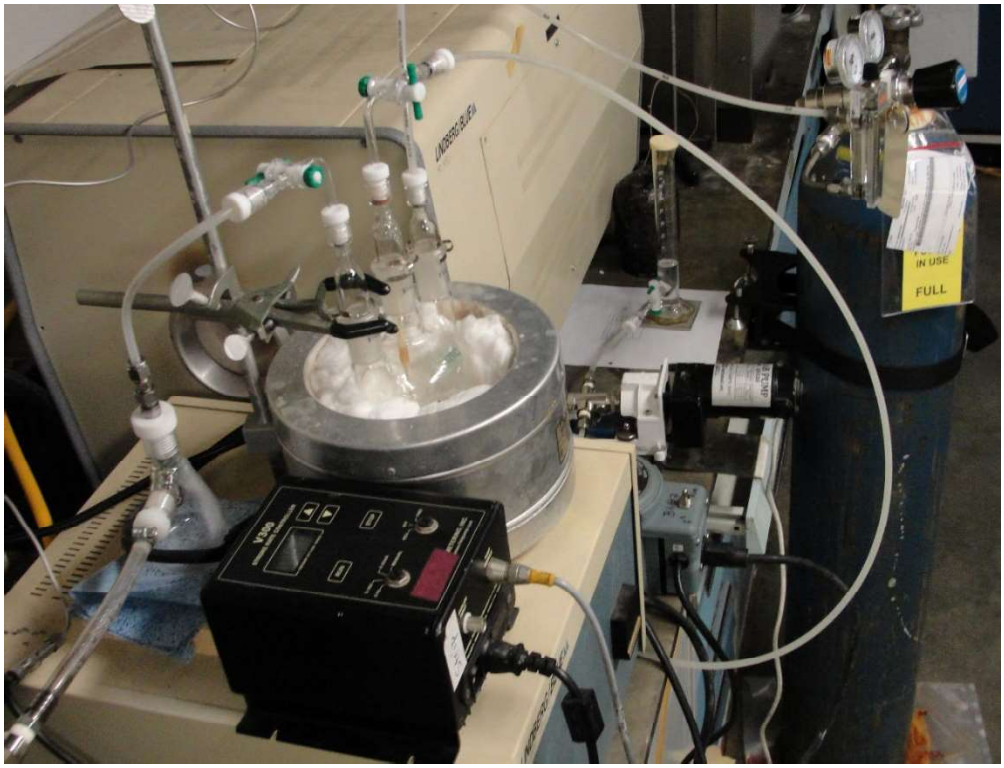

Figure 3-6. Steam / Argon Gas Delivery Equipment

3.3 Sample Analyses

Samples for SEM images were prepared by mounting on carbon sticky tape on an aluminum stub in a glovebox. The resulting sample was plasma coated with gold to decrease charging using a SPI plasma coater in the same glovebox. Images were taken using a LEO S440 contained scanning electron microscope (CSEM) completely enclosed in the same glovebox train as the plasma coater.

For XRD, samples were ground into a fine powder using an agate mortar and pestle. The powders were than affixed to individual plate glass slides using an adhesive and placed on a Bruker D8 Advance equipped with a copper x-ray source. Data was collected by step scanning over the 2theta range of 5-70° with a step size of 0.02° and a dwell time of 1 second. Search-match identification was performed with Jade software from Materials Data Inc and the PDF-4+ database from International Center for Diffraction Data.

4.0 Results and Discussion

The metal turnings for testing were cleaned on 7/11/22 per the instructions in Section 3.1. Oxidation of the metal turnings occurred on the dates listed in Table 4-1. Each oxidation experiment required a full work shift to complete. Consequently, only one sample could be oxidized per day. The process data for the tests are shown in Table 4-1. A picture of the 830 °C sample is provided in Figure 4-1.

Based on the literature, the formation of uranium dioxide was expected [3][4]. XRD Analyses completed on 8/10/22 of the four samples of Table 4-1 yielded UO_2 in all cases. The XRD spectra for the 600 °C sample is shown in Figure 4-2. Shown with the sample spectra are the spectral lines of U_3O_8 that any impurities are below the method detection limit. The stability of the UO_2 product is demonstrated in that although the sample was created on 7/14/22 and stored in air. Nonetheless, analysis shows a clean UO_2 product 27 days later.

Table 4-1. Sample Process Data

Temp (° C)	Test Date	Hold Time at Temp (min)	Argon Flow Rate (scfh)	Initial Mass (g)	Final Mass (g)	% Mass Gain
600	7/14/22	180	0.6	11.586	12.527	8.12
710	8/1/22	180	0.6	11.586	12.372	6.78
770	8/3/22	180	0.6	11.360	12.425	9.38
830	8/4/22	180	0.6	10.998	11.783	7.14
$U \rightarrow UO_2$, weight gain = 13.44% $U \rightarrow U_3O_8$, weight gain = 17.93% $U \rightarrow UO_3$, weight gain =20.17%						

Figure 4-1. 830 °C Sample in the Sample Boat

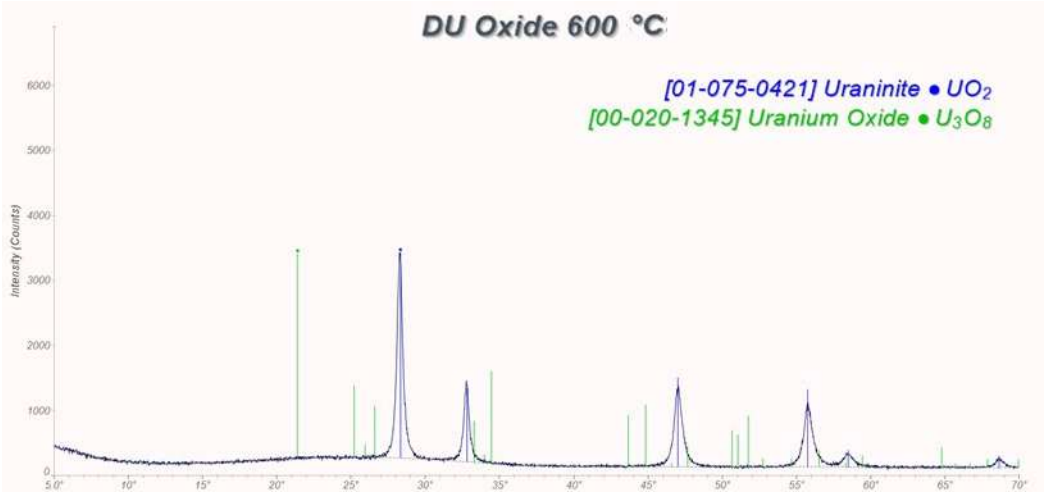

Figure 4-2. XRD of DUO_2 Formed at 600 °C (Plus Spectral Lines for U_3O_8)

Although the XRD analyses show only UO_2, it is expected that there was unoxidized DU metal in the larger sample that was not included in the subsample. The theoretical weight gain for a sample of U-238 metal being completely converted to UO_2 is 13.44%. The maximum weight gain measured was 9.38% for the sample at 770 °C. The relative percent weight gains observed at 710, 770, and 830 °C are consistent with those reported by Hopkinson, but the value for 600 °C is higher than expected based on the literature [4]. However, the 600 °C sample was generated only three days after the metal was cleaned, compared to the tests at 710, 770, and 830 °C, which were made 18-20 days after the cleaning process. Consequently, the

oxidation rates for the tests at 710, 770, and 830 °C may have been suppressed by the accumulation of a room temperature and atmospheric pressure generated oxide layer.

The characteristics of the U metal oxidation products at each temperature are displayed in Figure 4-3. The brightness of the pictures has been adjusted to show the granularity of the samples. There is a distinct increase in general particle size with increasing temperature. The differences are more evident in the SEM images at 30X magnification (Figure 4-4 and Figure 4-5). The 600 °C sample was a fine particle size. The materials made at 710 and 770 °C had larger particle sizes but were friable. The 830 °C product was coarse and more resistant to breakage.

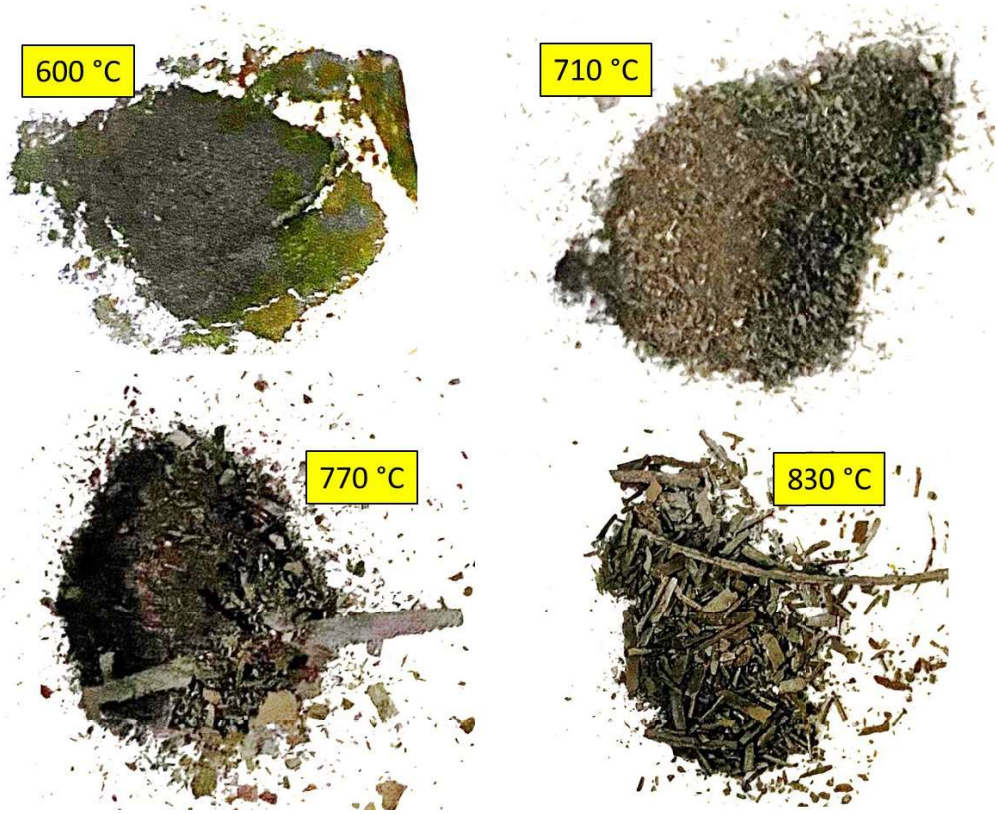

Figure 4-3. Post-Oxidation Samples – Brightness Adjusted to Show Sample Granularity

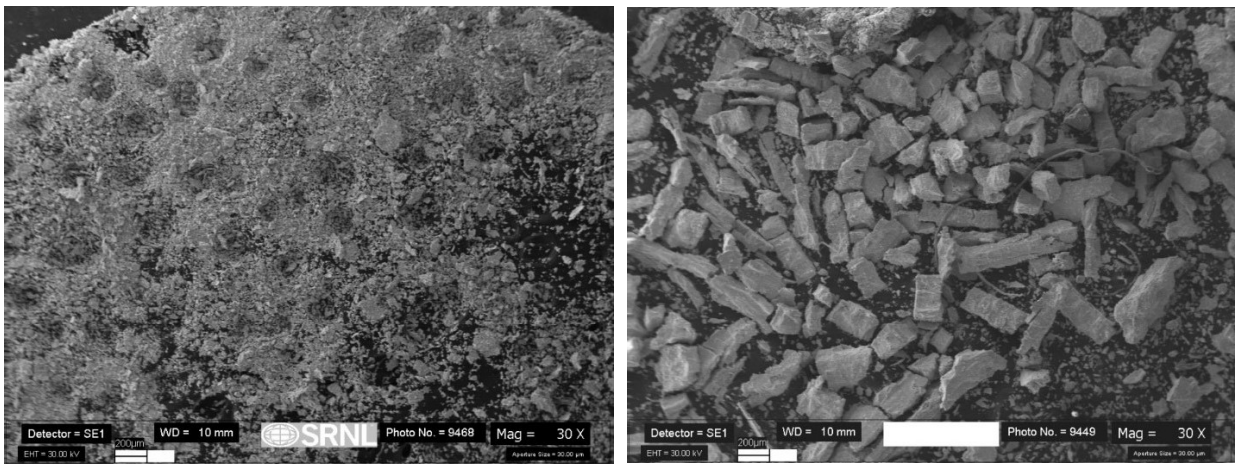

Figure 4-4. SEM of 600 °C and 710 °C Samples at 30X Magnification

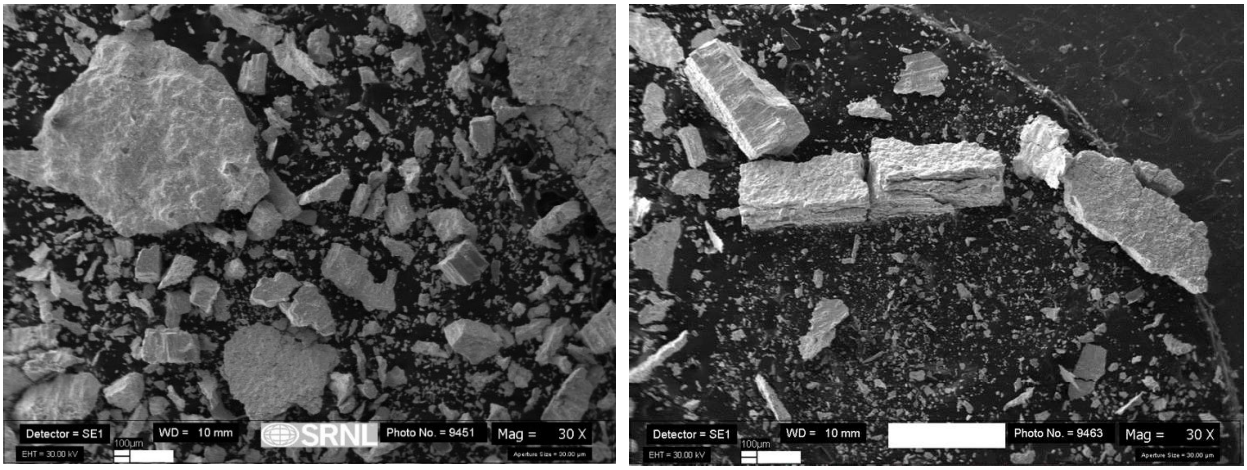

Figure 4-5. SEM of 770 °C and 830 °C Samples at 30X Magnification

Because both particle size and strength are important, portions of the products from each temperature were subjected to further heat treatment to sinter the products and increase their strength. Sample sizes ranging from 3.2 to 4.6 g were heated in the furnace with a flow of Ar/steam for three hours at 970 °C. The process conditions are shown in Table 4-2. A comparison of the initial and final weights of the samples indicates negligible weight gain or loss. While this result is consistent with the XRD analyses which identified only UO_2 (Figure 4-2), it is not consistent with the data of Table 4-1which indicate incomplete conversion of DU metal to UO_2. If the conversion of DU metal to UO_2 was incomplete, and the samples provided for the secondary calcination are representative, they all should have experienced distinct weight gains unless the UO_2 exists as a layer which is impervious to steam penetration. However, that would be in contradiction to the data in the literature [4] which reports steady weight gains of U metal in steam at elevated temperatures.

Table 4-2. Secondary Calcination Process Data

Initial Oxidation Temperature (° C)	Hold Time at 970 °C (min)	Argon Flow Rate (scfh)	Initial Mass (g)	Final Mass (g)	% Mass Gain
600	180	0.6	3.694	3.664	-0.81
710	180	0.6	3.454	3.441	-0.38
770	180	0.6	4.580	4.581	0.02
830	180	0.6	3.190	3.177	-0.41

Figure 4-6 provides pictures of the samples after the oxidation of the samples produced at the four different temperatures. The brightness of the pictures has been adjusted to reveal the granularity of the products. An increase in sample clumping was observed at 970 °C for the sample initially produced at 600, 710, and 770 °C. The sample initially produced at 600 °C stuck together to form a monolith, but sample agglomeration was easily overcome by a small amount of force. Qualitatively, the particles making up the samples seemed to have increased strength (i.e., reduced friability) after the secondary calcination. However, due to the available sample quantities, a quantitative comparison of material strength could not be obtained. That comparison will be the subject of future studies.

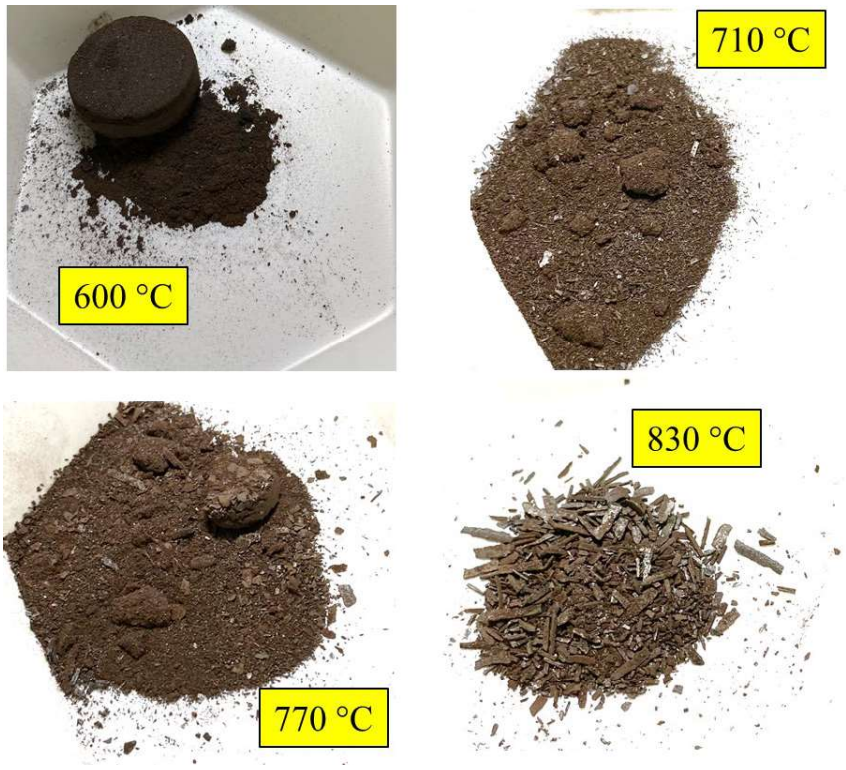

Figure 4-6. Samples after Secondary Oxidation – Brightness Adjusted

5.0 Conclusions

Initial studies into the oxidation of DU metal in an atmosphere of argon and steam have been completed. The objective of the studies was to produce UO_2 powders with larger particle sizes than is formed by typical UO_2 syntheses which have particle size distributions in the range of 5-50 microns [1]. Oxidation of DU metal in Ar/steam at 600, 710, 770, and 830 °C yielded a UO_2 product with little or no impurity at each temperature, within the detection limits of XRD. Although not enough sample was available for analysis of the particle size distribution, SEM images reveal that a significant volume fraction of the products at 710, 770, and 830 ° C exceed 100 microns. The material produced at 600 °C is too fine of a particle size for program needs.

A remaining question associated with the UO_2 synthesis is the mechanical strength of the particles formed. The size of the samples produced in these tests did not allow for quantitative assessment of particle size or mechanical strength. A qualitative assessment of the particles formed at 710, 770, and 830 °C was that the samples produced at 710 and 770 °C were friable, and the sample produced 830 °C had more mechanical strength. Sintering the samples in Ar/steam at 970 °C appeared to increase the strength of the particles without changing their general particle size characteristics. If the sintering at 970 °C is inadequate for program needs, higher sintering temperatures can be evaluated. The literature indicates that UO_2 sintering occurs at 1300 °C, but that the presence of steam enhances it [3], which implies that it will sinter at a lower temperature in an atmosphere of Ar/steam.

6.0 Future Work

Testing in FY22 evaluated DU metal oxidation with Ar/steam at four temperatures – 600, 710, 770, and 830 °C. The particle sizes of UO_2 formed at 600 °C were too small to meet the programmatic need. The particle sizes of UO_2 formed at 710, 770, and 830 °C show promise for being of the required particle sizes. However, material strength remains a question and warranted additional testing at higher temperatures.

In FY23, SRNL will advance toward identifying DU metal oxidation conditions which yield the most-favorable particle-size distributions with sufficient mechanical strength for programmatic usage. Further consideration will be given to oxidation temperature, steam addition to the reaction vessel, U metal pretreatment, and sintering conditions after oxidation. The UO_2 production could involve a two-step process where particles are formed in steam at lower temperatures (700-850 °C) and then sintered in steam at elevated temperatures (950-1200 °C). Larger samples (50-100 g each) will be produced and characterized with a sieve analysis. The set-up of the sieve analysis in a radioactive hood would be an activity of this work scope. The work scope will include other analyses for sample characterization, as necessary.

7.0 References

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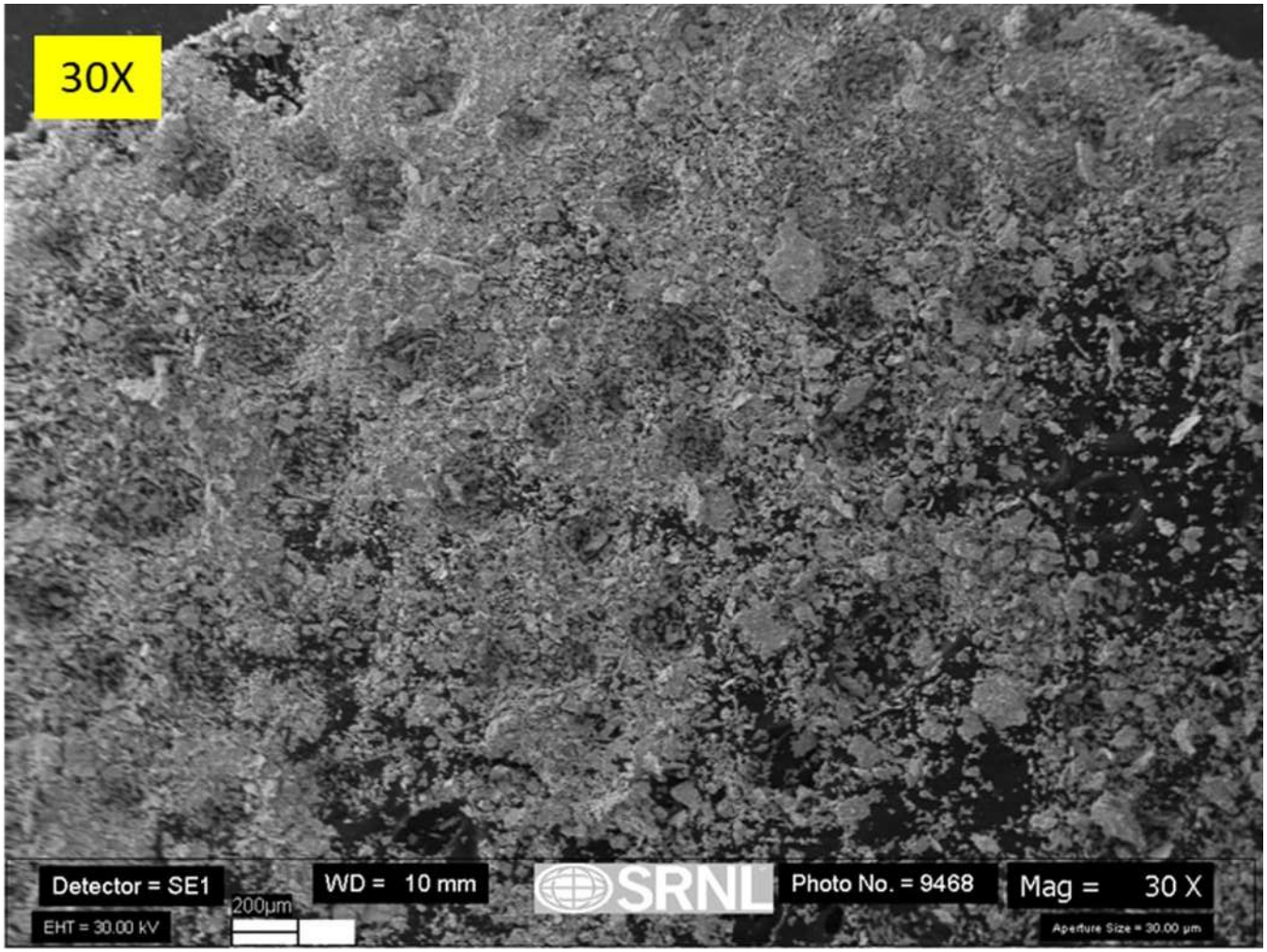
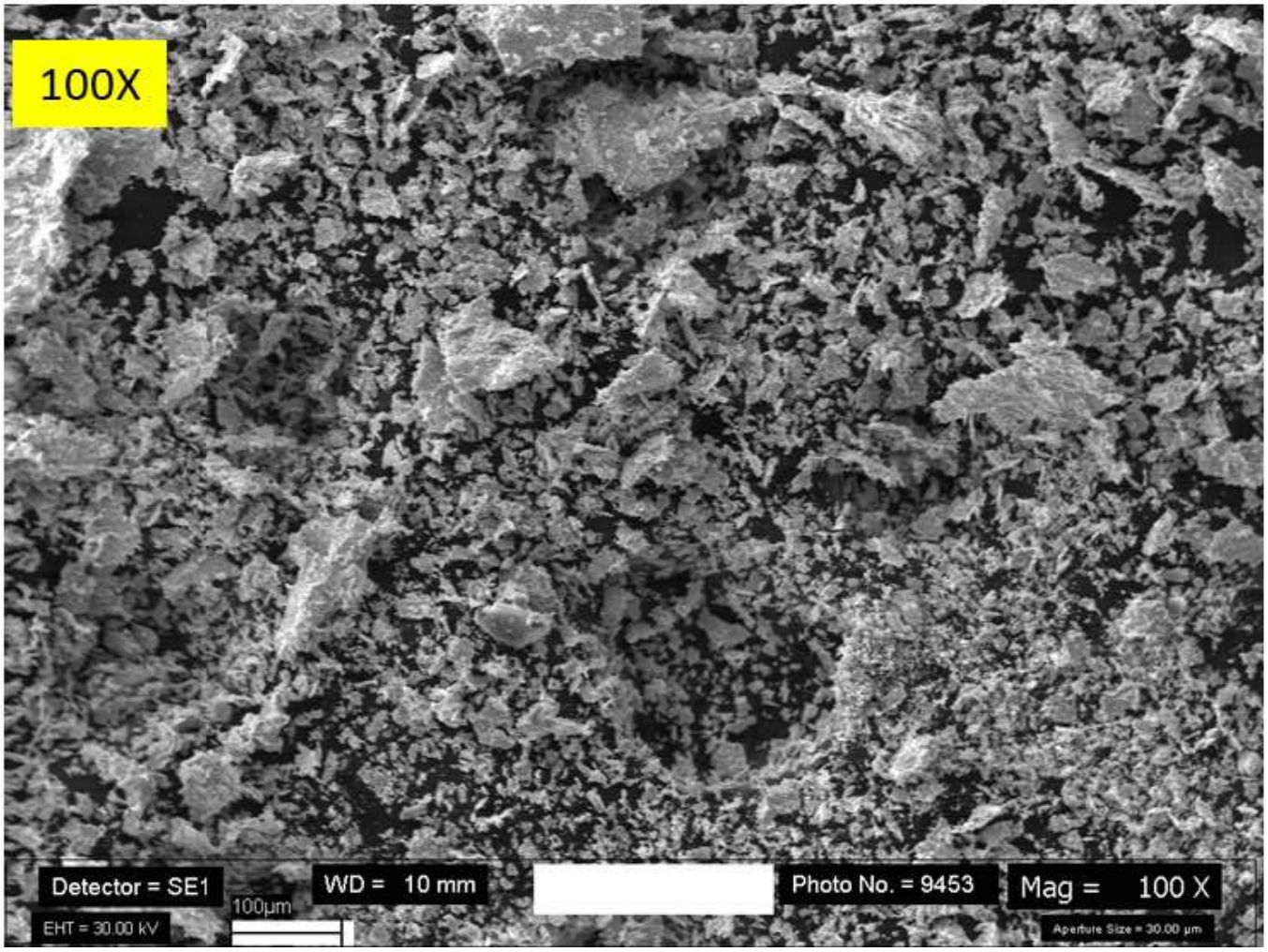
4. B. E. Hopkinson, Kinetics of the Uranium-Steam Reaction, *J. Electrochem Soc,* 1959, 106:2, 102-106.
5. P. J. Hayward, D. G. Evans, P. Taylor, I. M. George, and A. M. Duclos, Oxidation of Uranium in Steam, *J Nucl Mat*, 1994, 217, 82-92.

8.0 Appendices

8.1 Appendix A. SEM Images

8.1.1 Sample from Steam Oxidation at 600 °C

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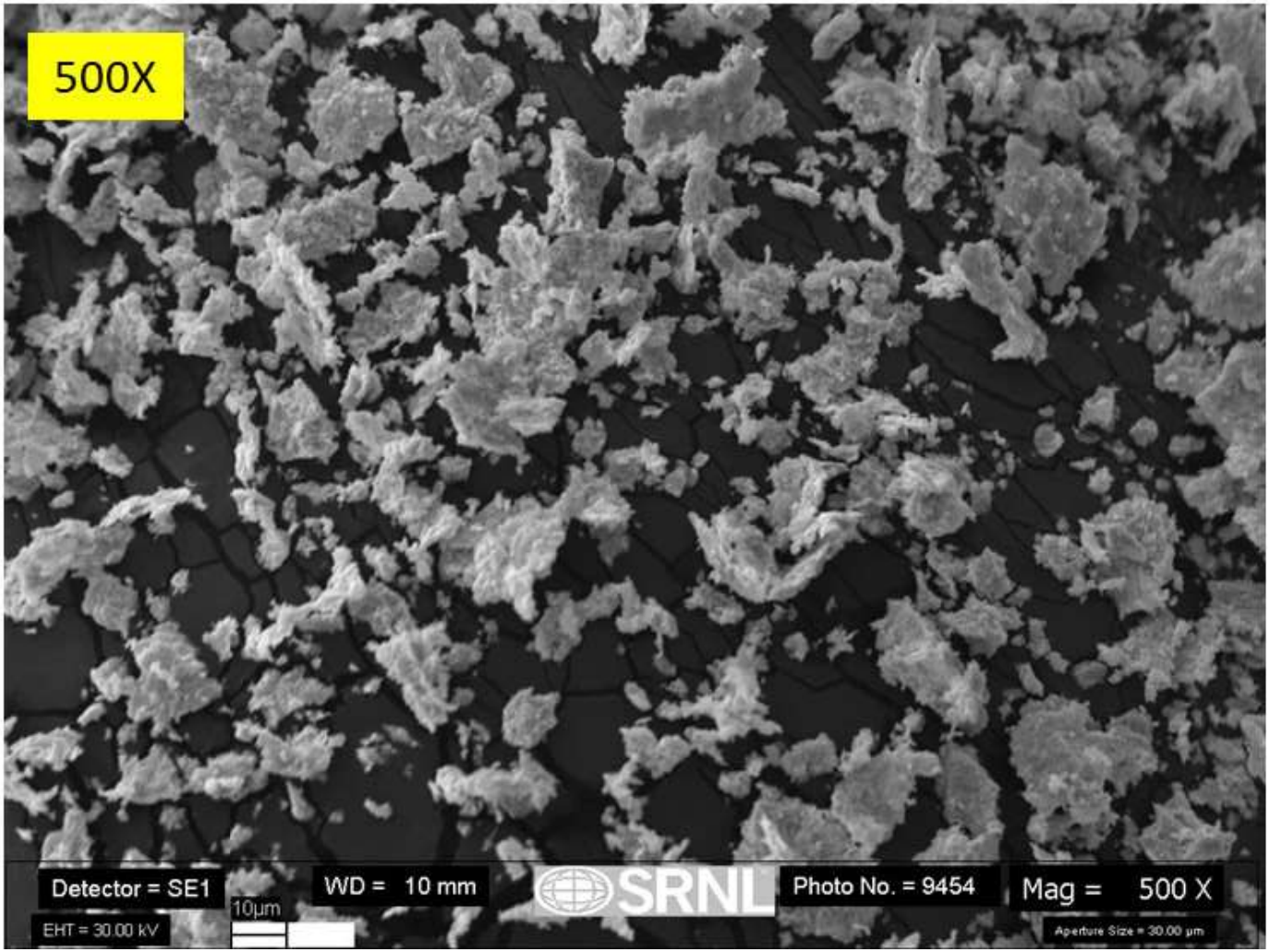
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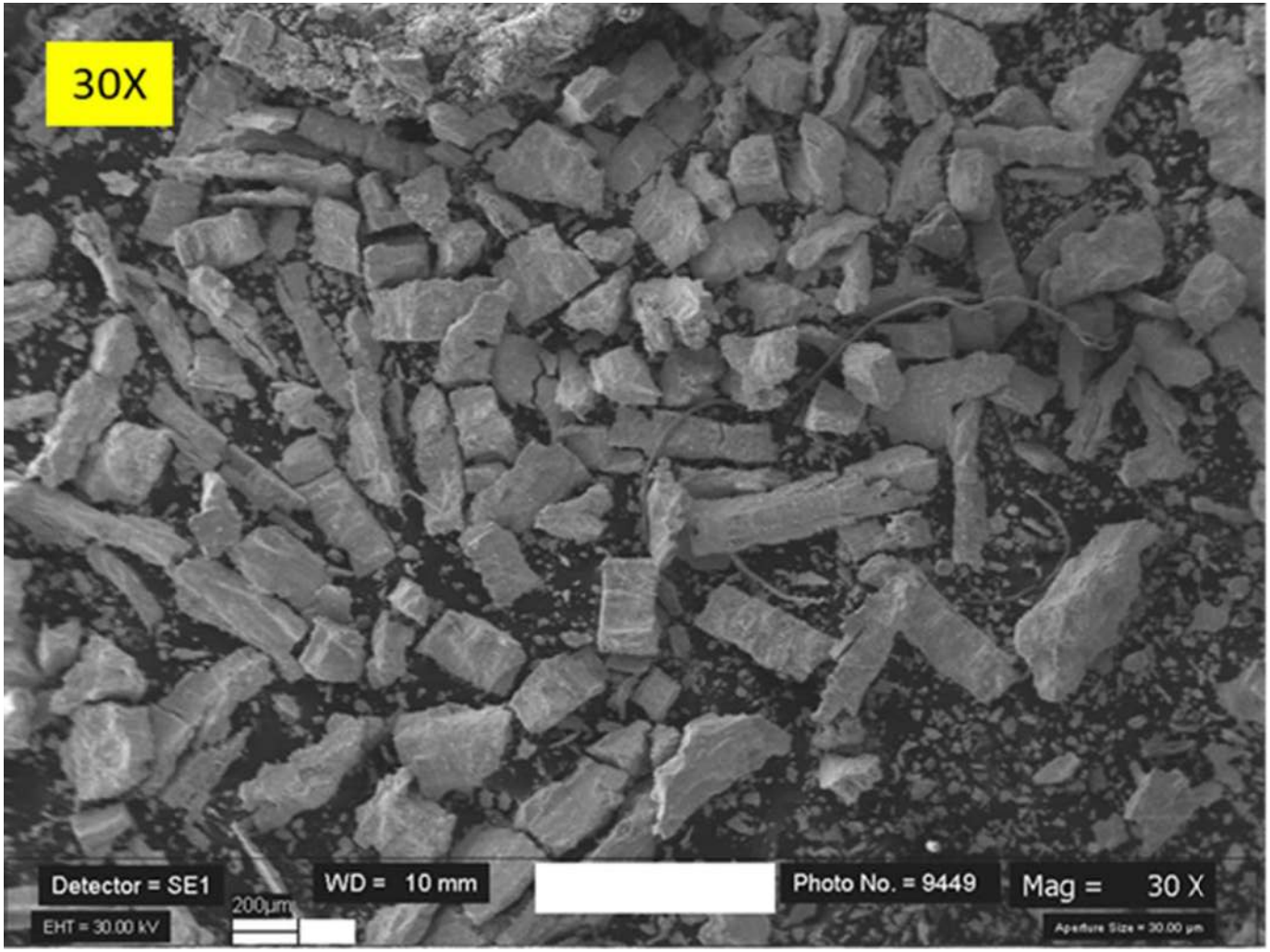
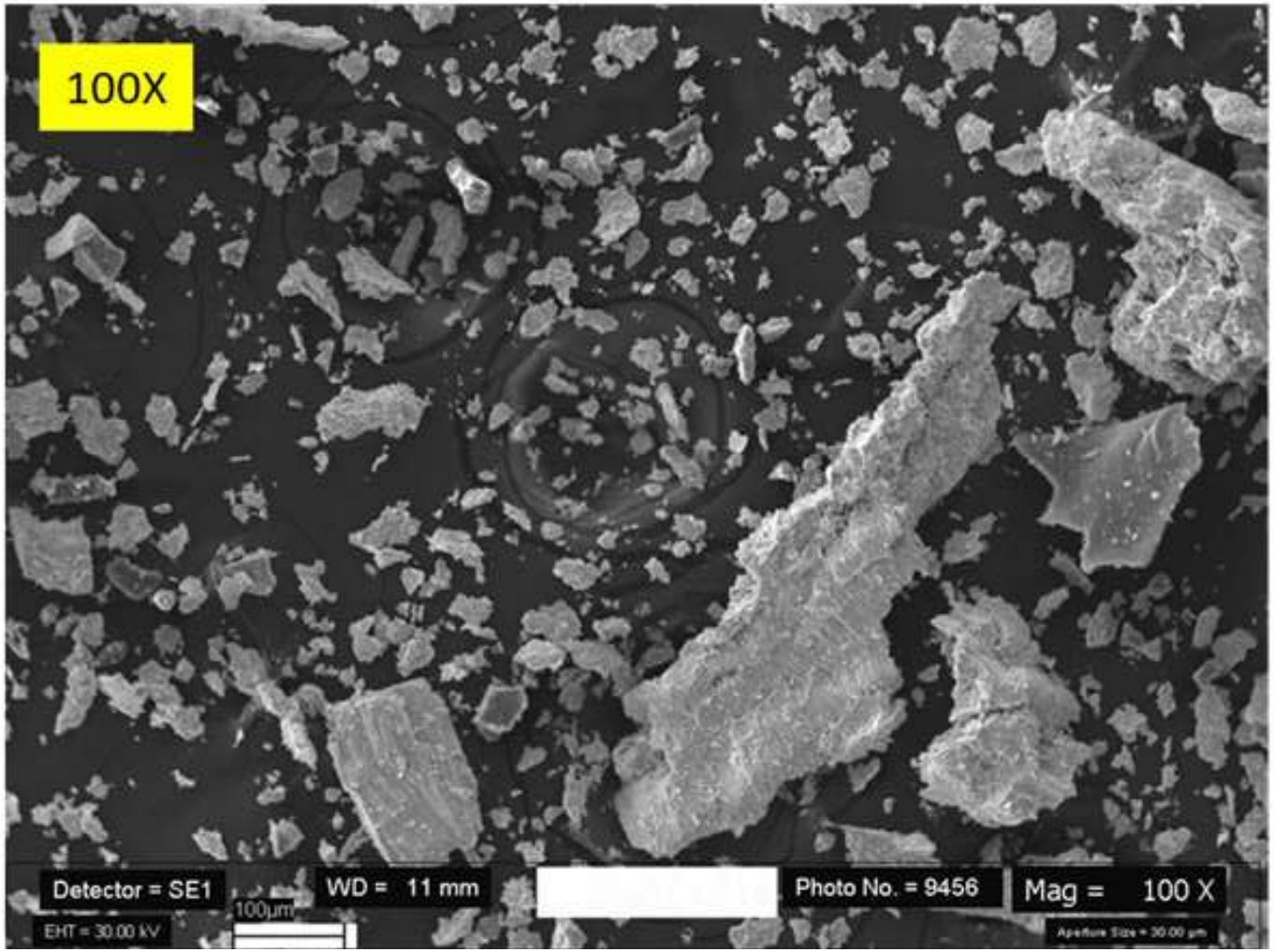
8.1.2 Sample from Steam Oxidation at 710 °C

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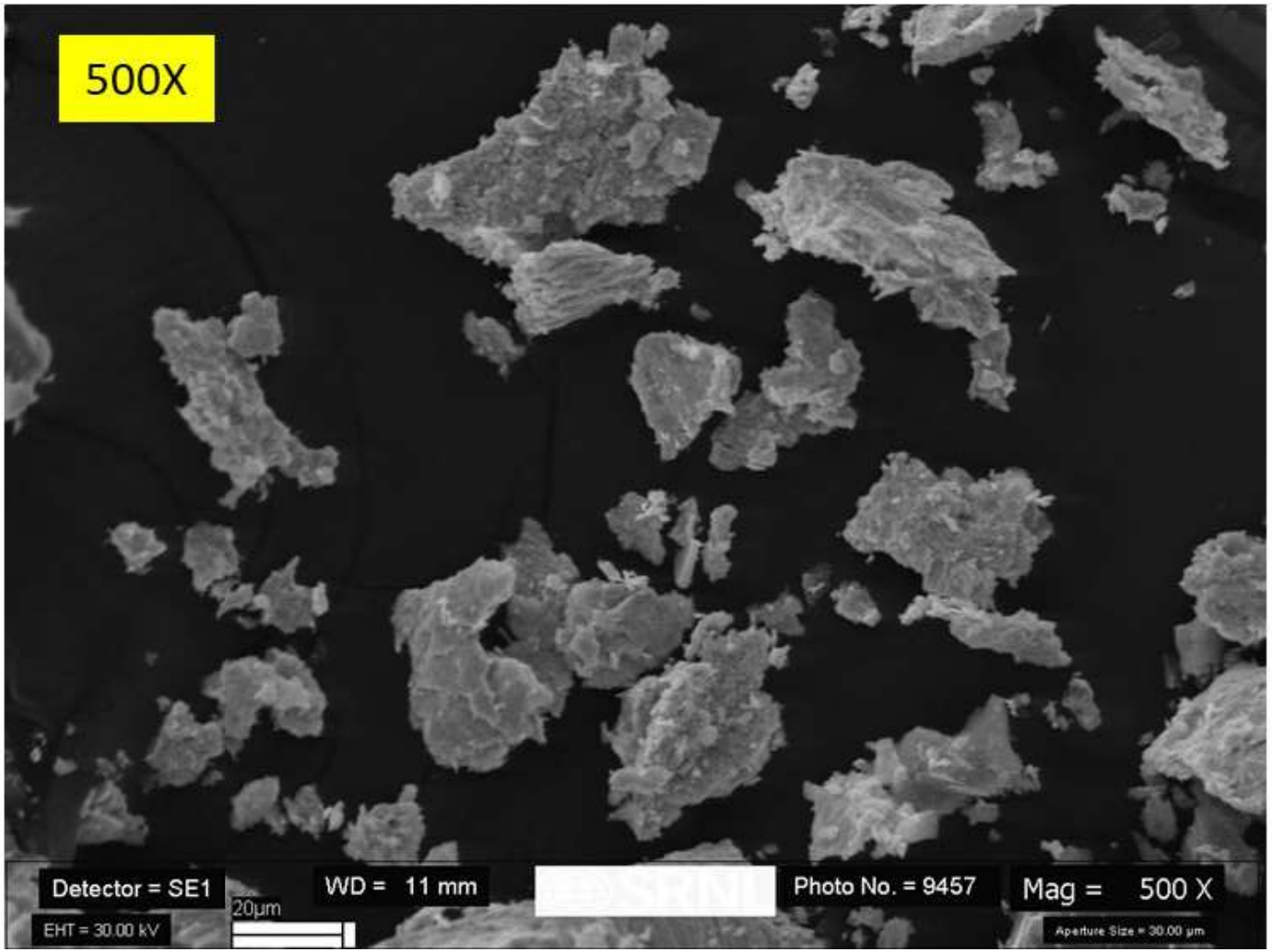
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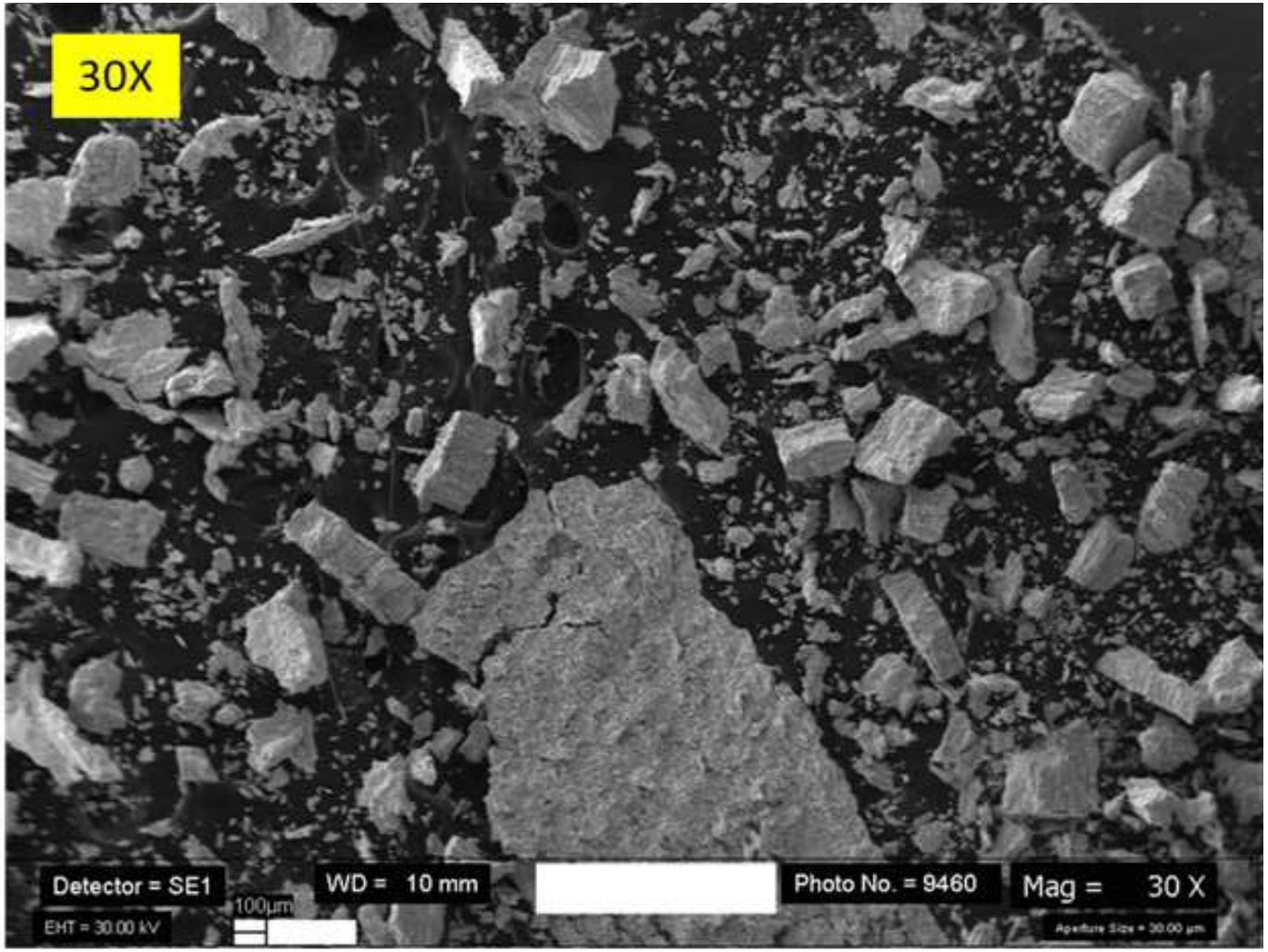
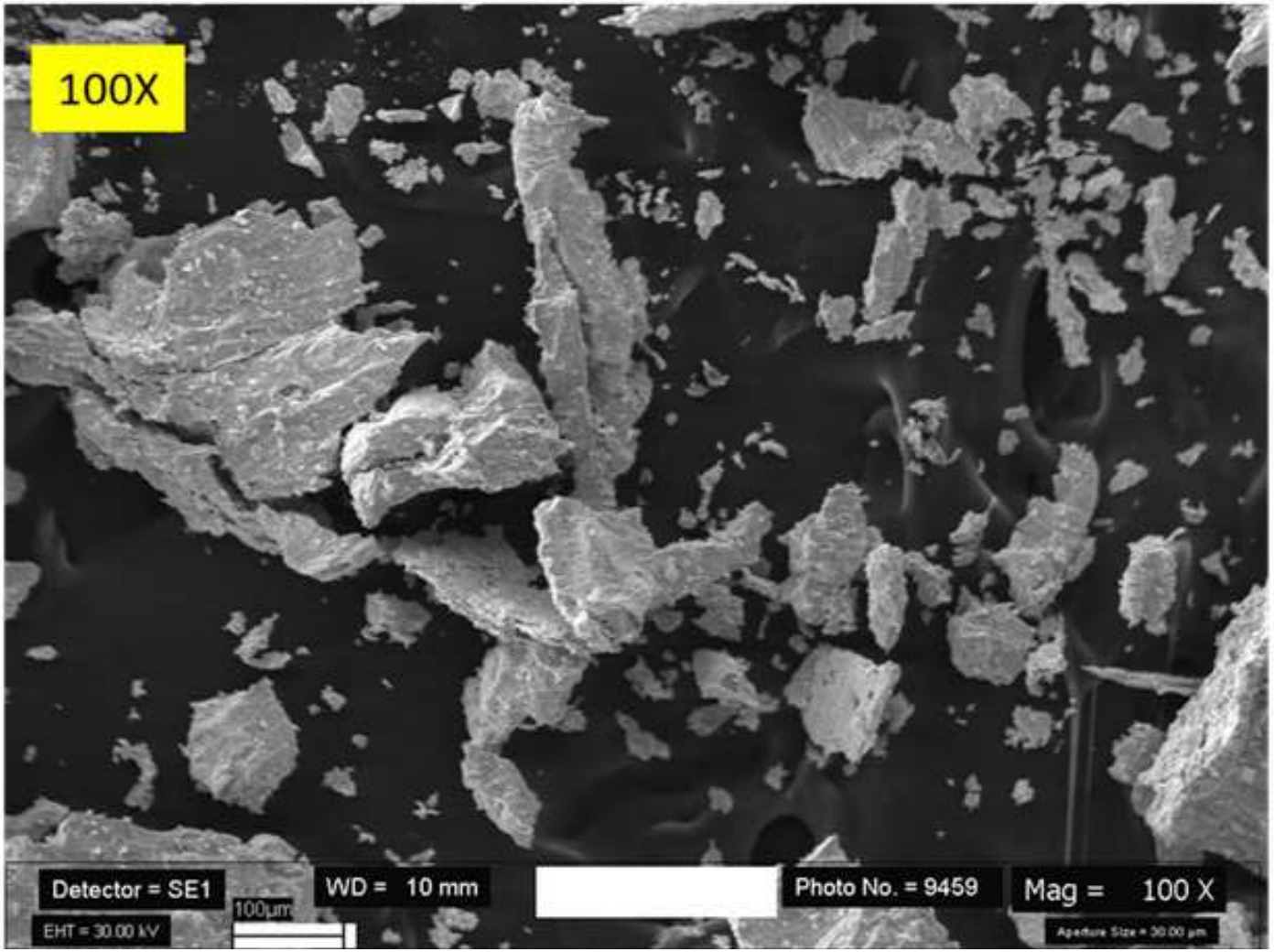
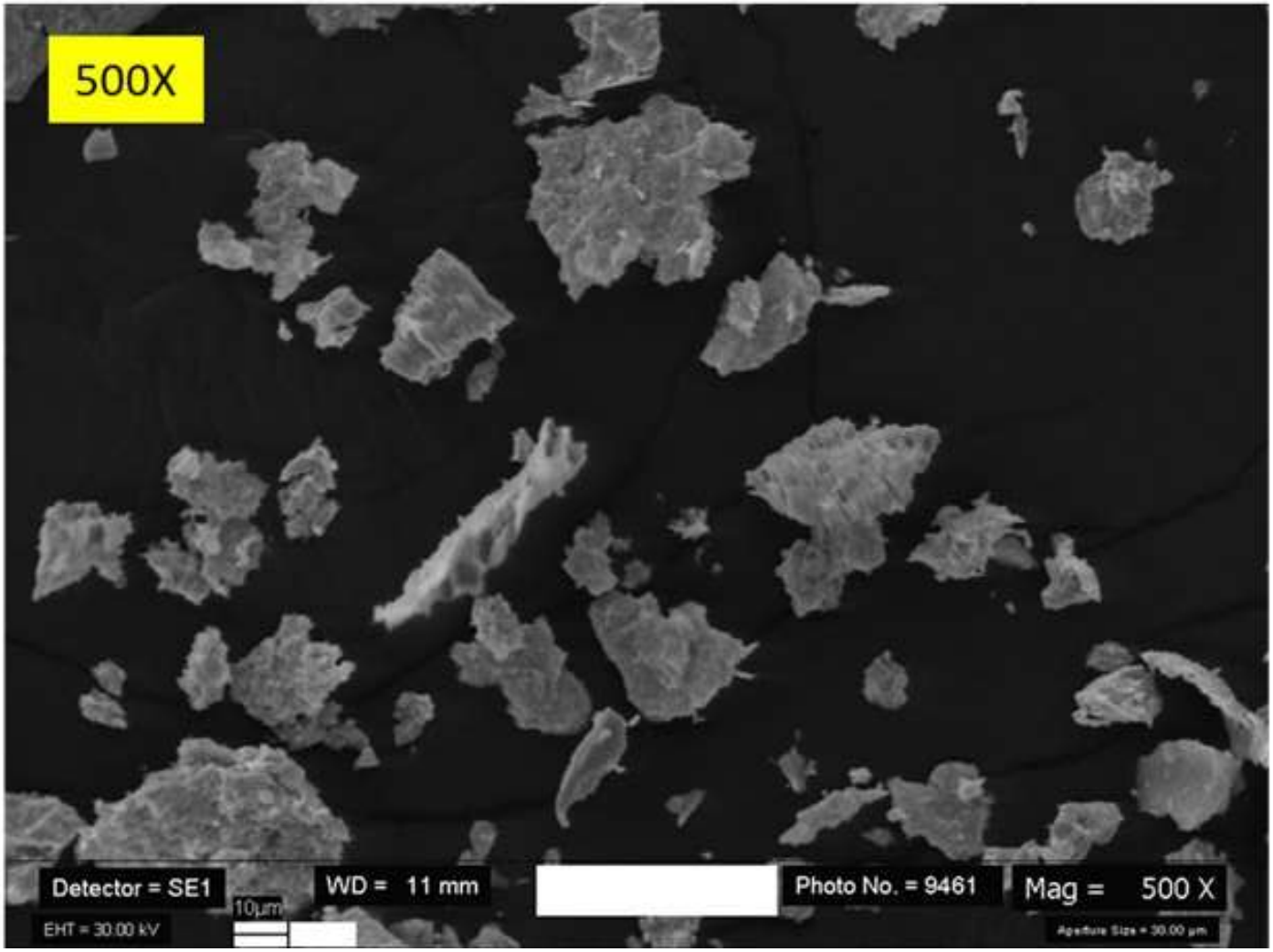
Photo No. = 9457
Mag = 500 X
Aperture Size = 30.00 µm

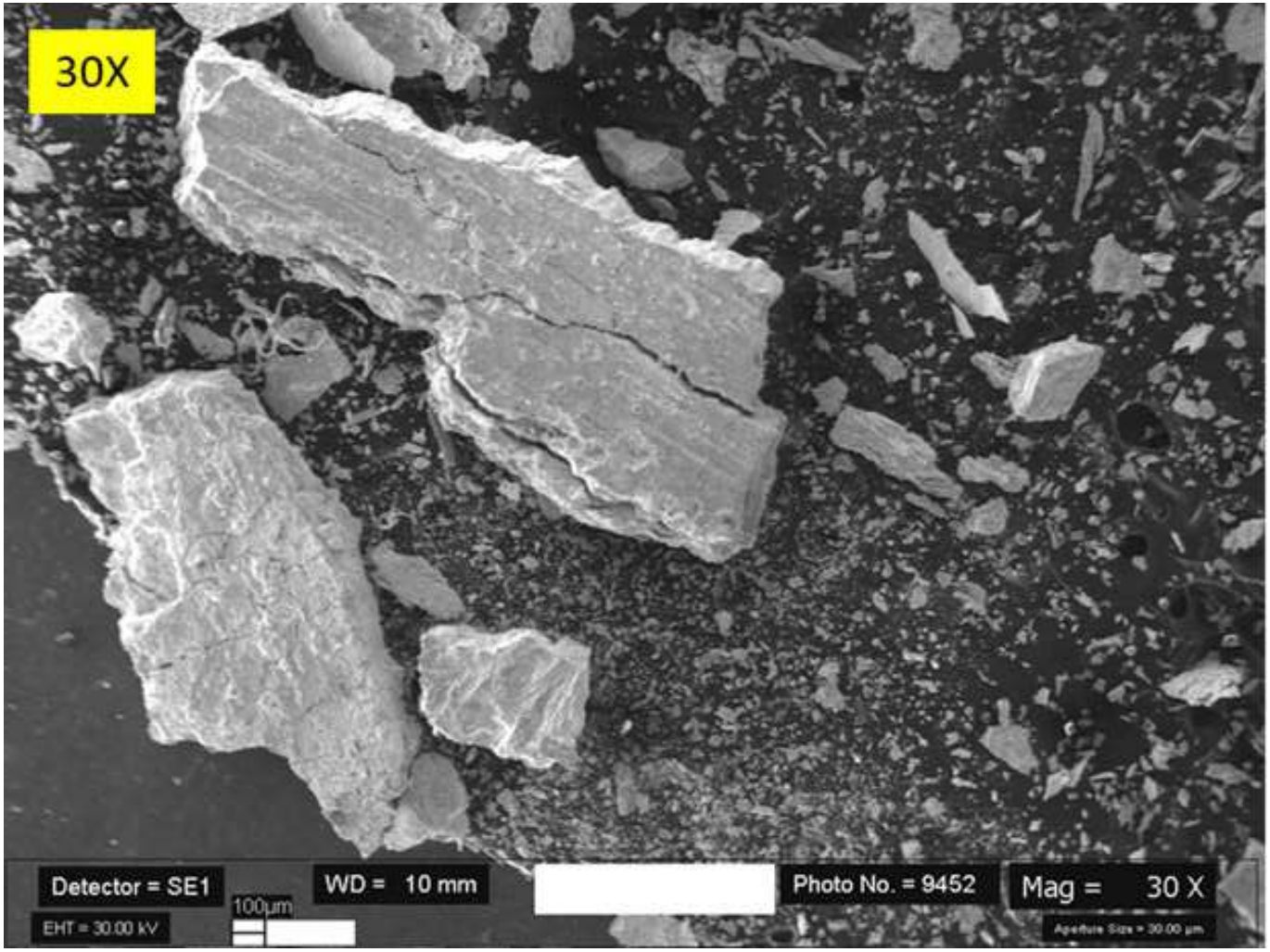
8.1.3 Sample from Steam Oxidation at 770 °C

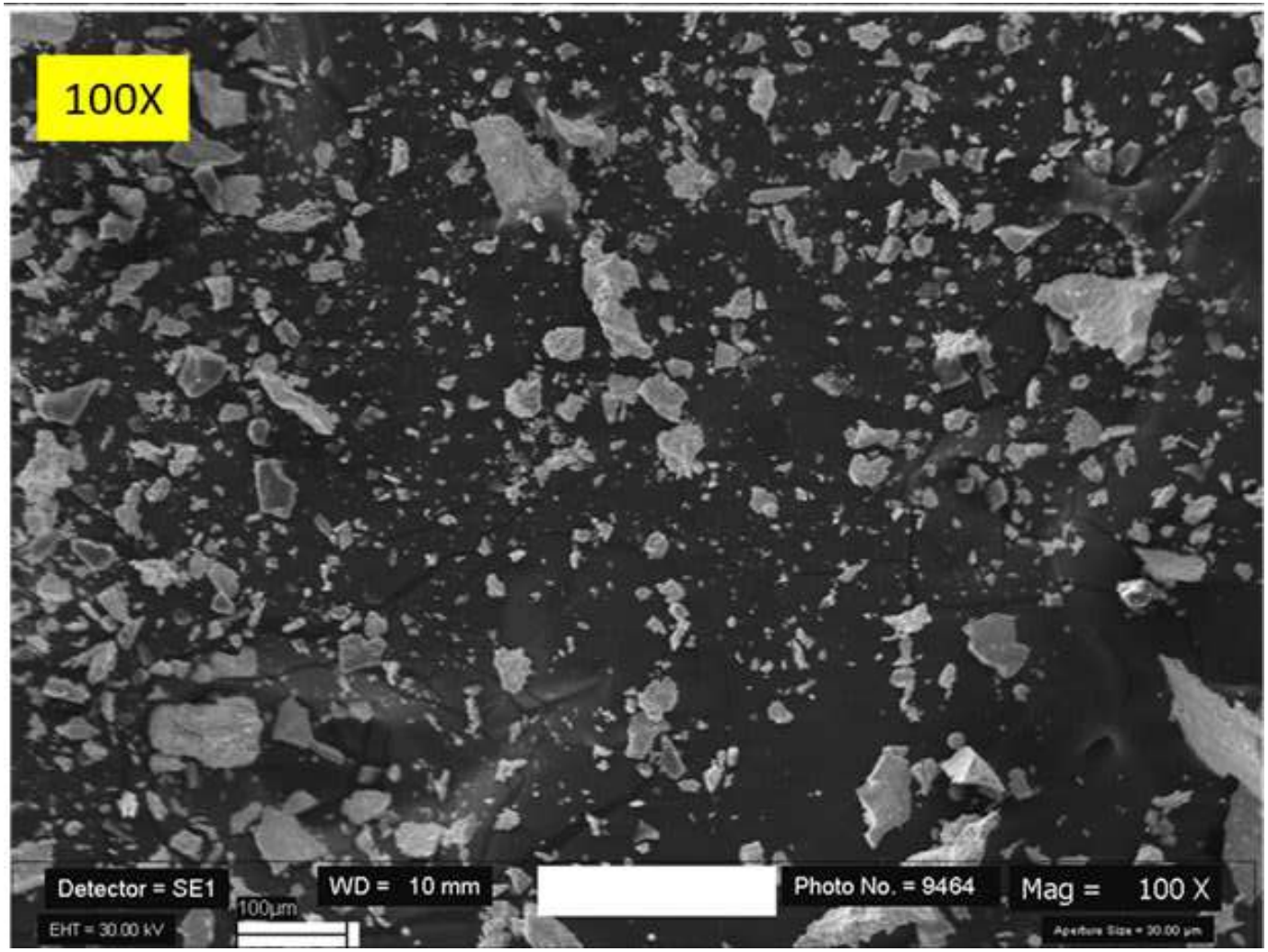


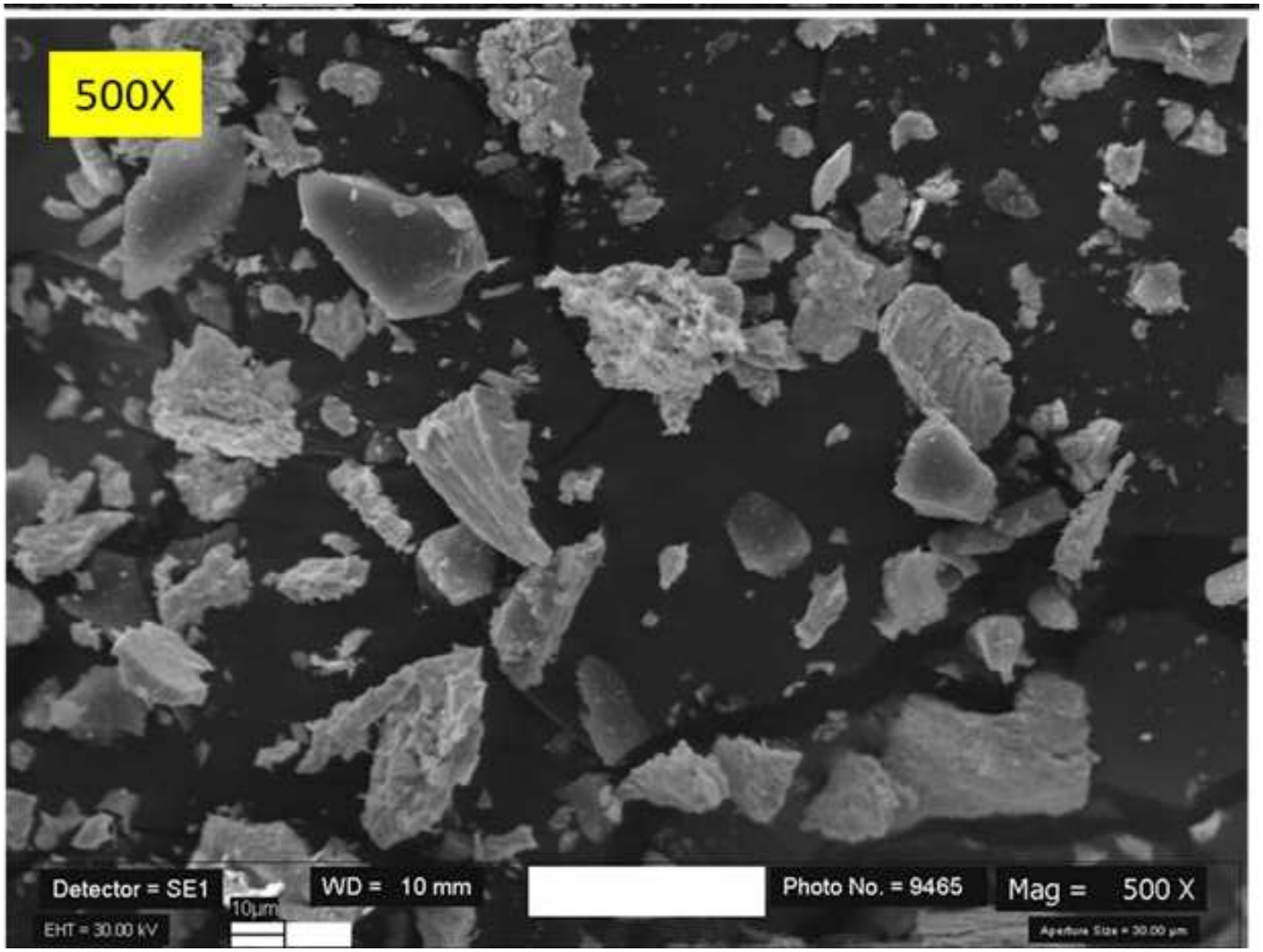

100X
Detector = SE1
EHT = 30.00 kV
100µm
WD = 10 mm
Photo No. = 9459
Mag = 100 X


500X
Detector = SE1
WD = 11 mm
Photo No. = 9461
Mag = 500 X
10µm
EHT = 30.00 kV

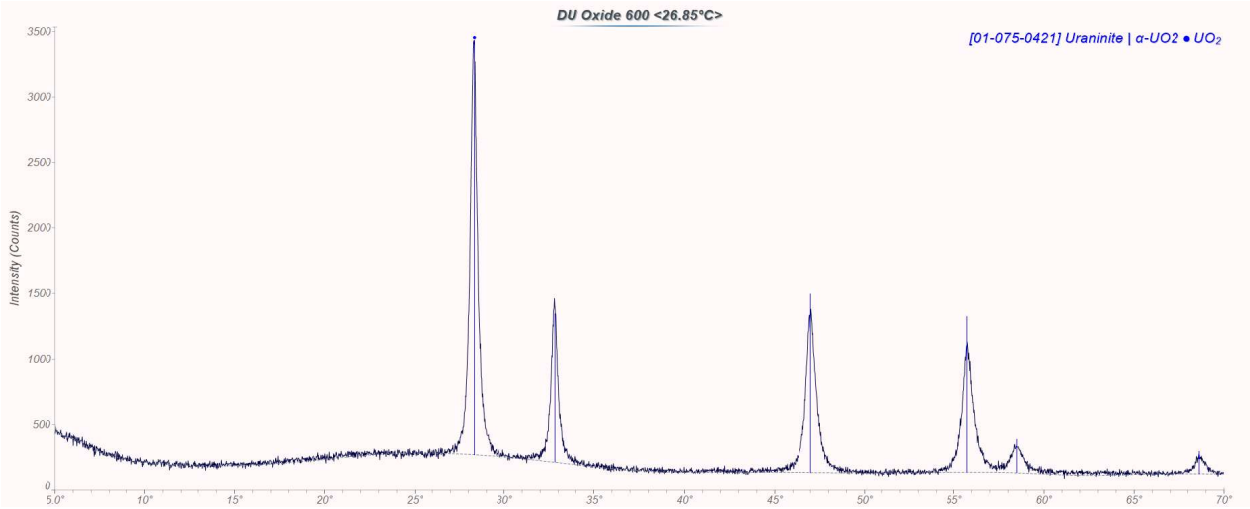
8.1.4 Sample from Steam Oxidation at 830 °C

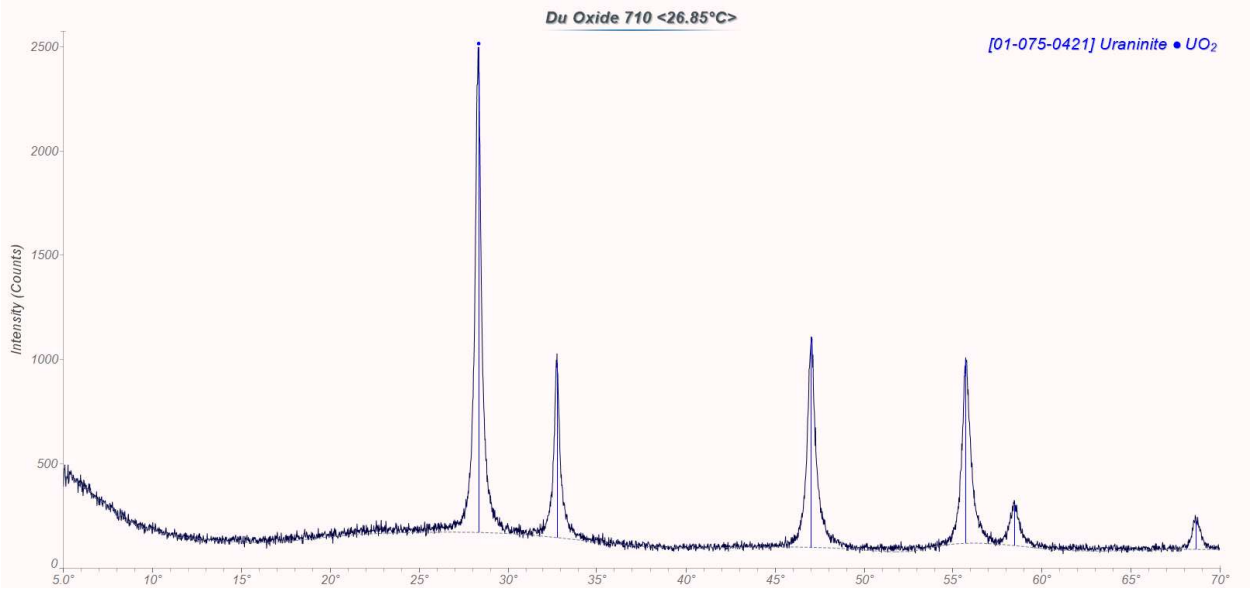


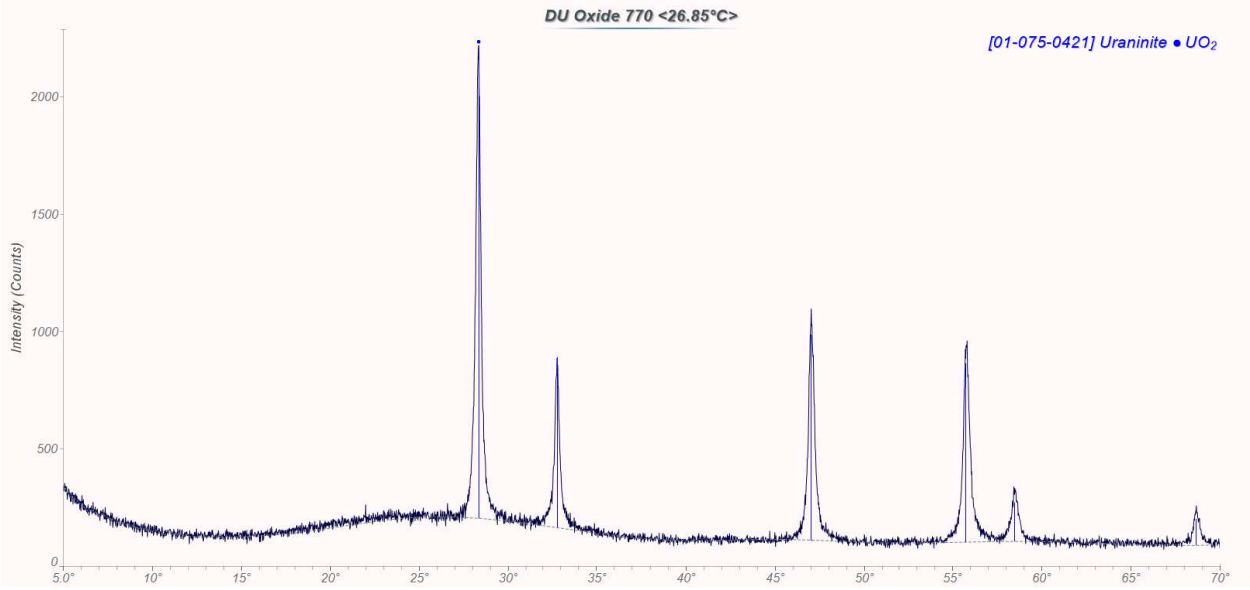

100X
Detector = SE1
EHT = 30.00 kV
100µm
WD = 10 mm
Photo No. = 9464
Mag = 100 X

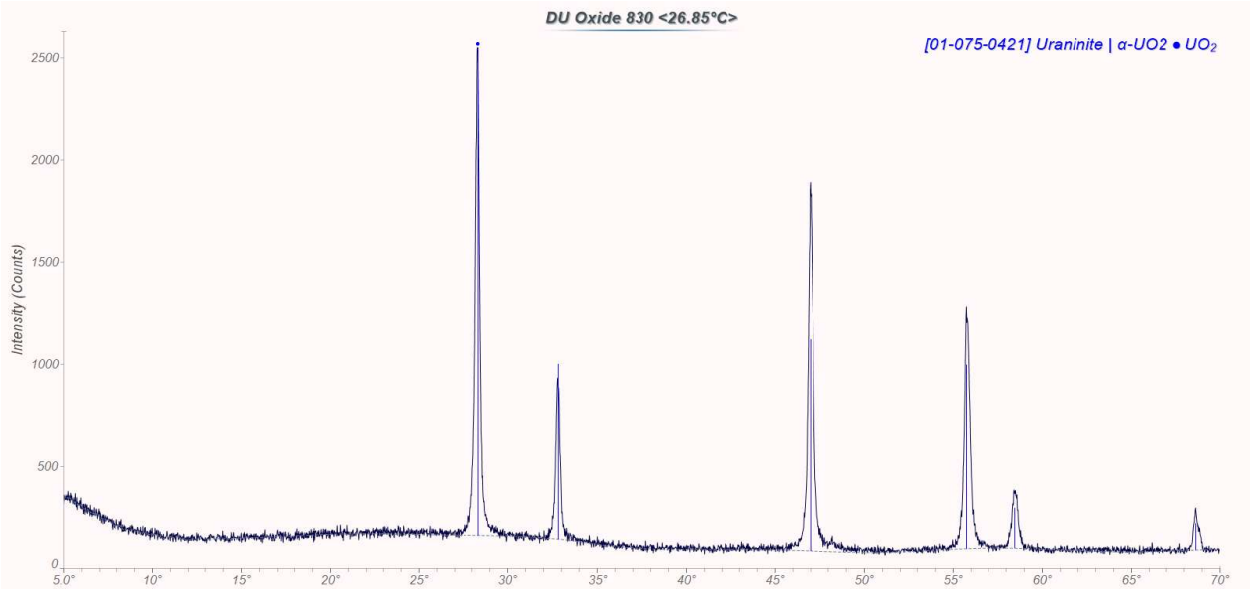

500X
Detector = SE1
EHT = 30.00 kV
10µm
WD = 10 mm
Photo No. = 9465
Mag = 500 X

8.2 Appendix B. XRD Analyses of Oxidation Products






DU Oxide 770 <26.85°C>
[01-075-0421] Uraninite ● UO2
Intensity (Counts)
2000
1500
1000
500
0
5.0°
10°
15°
20°
25°
30°
35°
40°
45°
50°
55°
60°
65°
70°


DU Oxide 830 <26.85°C>
[01-075-0421] Uraninite | α-UO2 ● UO2
Intensity (Counts)
2500
2000
1500
1000
500
0
5.0°
10°
15°
20°
25°
30°
35°
40°
45°
50°
55°
60°
65°
70°

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