Pacific Northwest NATIONAL LABORATORY

PNNL-36964, Rev 0

Materials Characterization, Prediction, and Control Project

Summary Report on Material Characterization, Part 3

September 2024

DR Todd
K Nwe
M Pole
Y Guo
A Guzman
A Ortiz
MJ Olszta
O Linsuain

With contributions from:
DM Brown
C Minerich
JD Escobar Atehortua
D Garcia
T Wang
H Das
KA Ross
EI Barker
LE Smith

U.S. DEPARTMENT OF ENERGY

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Pacific Northwest National Laboratory
Richland, Washington 99354

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Authorship for this report reflects substantial contributions in collecting, curating, interpreting, and/or technical leadership of MCPC Material Characterization Vertex activities throughout the duration of the project. Those responsible for specific characterization activities are identified in report attachments that describe that data.

Acronyms and Abbreviations

DOI	Digital Object Identifier – also doi
EBSD	Electron Backscatter Diffraction
FOM	Figure-of-Merit
FSP	friction stir processing
MCPC	Materials Characterization, Prediction, and Control
PNNL	Pacific Northwest National Laboratory
SEM	scanning electron microscopy
SHA1	Secure Hash Algorithm 1
UT	ultrasonic testing

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1.0 Introduction and Background

The Pacific Northwest National Laboratory (PNNL) undertook the Materials Characterization, Prediction, and Control (MCPC) Laboratory Directed Research and Development Project to advance understanding of nuclear material processing and enable multifold acceleration in the development and qualification of new material systems in national security and advanced energy applications (Smith 2021). The MCPC Project executed research across three scientific vertices—material characterization, predictive modeling, and data analytics—with extensive support by a data curation and management team.

MCPC Project Logo

The central technical objective in the MCPC Project was to improve the prediction and characterization of the process-structure-property relationships within the microstructurally refined region of stainless-steel samples prepared utilizing friction stir processing (FSP). Application of the FSP technique is well established at PNNL within the Solid Phase Processing capability through many years of investment across a range of materials and applications (PNNL 2024).

Three distinct rounds of FSP experiments were performed by the experimental team, producing replicate samples across eleven different nominal processing conditions (Condition IDs) listed in Table 1. The starting material on which FSP was applied was commercially available unprocessed stainless-steel type 316L material. Chosen processing conditions were very diverse, and some were intentionally chosen to produce defects. Several samples experienced tool breakage during experimentation, so a full set of three replicates was not produced for every nominal processing condition.

Table 1. Nominal process conditions for FSP experiments.

Condition ID	Temperature (°C)	Tool traverse (in/min)	Tool traverse (mm/min)	Force (lbs)	Force (kN)
C00	N/A	N/A	N/A	N/A	N/A
C01	720	0.5	12.7	9000	40.0
C02	720	0.5	12.7	10500	46.7
C03	720	1.0	25.4	10500	46.7
C04	720	1.0	25.4	9000	40.0
C05	720	3.0	76.2	10500	46.7
C06	750	1.0	25.4	10500	46.7
C07	750	3.0	76.2	10500	46.7
C08	800	1.0	25.4	10500	46.7

Condition ID	Temperature (°C)	Tool traverse (in/min)	Tool traverse (mm/min)	Force (lbs)	Force (kN)
C09	800	3.0	76.2	10500	46.7
C10	850	1.0	25.4	10500	46.7
C11	850	3.0	76.2	10500	46.7

Condition ID “C00” represents the unprocessed starting material in the form of a control sample.
“Temperature” is the sensed value within the FSP tool and not the working temperature at the tool/steel interface.

The Material Characterization task within the MCPC Project was tasked with delivering results from two primary characterization approaches: 1) destructive characterization modalities utilizing traditional forms of microstructure and property evaluation, and 2) a nondestructive ultrasonic testing modality capable of full-volume interrogation for defects and microstructure characteristics. Material characterization data was obtained via five characterization modalities across the destructive and nondestructive approaches. The modalities are listed below alphabetically based on the names used with the curated datasets:

- Electron Backscatter Diffraction (EBSD): *Data for this modality is curated and archived with the identifying name “EBSD” in curated datasets*.
- Hardness testing via microscopic indentation method: *Data for this modality is curated and archived with the identifying name “HARDNESS” in curated datasets*.
- Optical Microscopy: *Data for this modality is curated and archived with the identifying name “OPTICAL” in curated datasets*.
- Scanning electron microscopy (SEM): *Data for this modality is curated and archived with the identifying name “SEM” in curated datasets*.
- Ultrasonic testing (UT) is referred to as nondestructive examination in some project documentation: *Data for this modality is curated and archived with the identifying name “ULTRASONIC” in curated datasets*.

Background information on these and other material characterization modalities relevant to FSP can be found in Glass et al. (2024).

As described in this report, material characterization data from each round and modality was carefully curated and key metadata collected to ensure traceability, reproducibility, and explainability of the MCPC datasets. Implementation of modern data management and curation was a central theme in MCPC, which facilitated efficient uptake and utilization, including in the calculation of example metrics via predictive modeling and data analytics methods for each nominal process condition. It is anticipated that the significant body of curated material characterization data developed under MCPC will be utilized for other research at PNNL and beyond in the future.

2.0 Scope

This report summarizes key data from material characterization Round 3 of the MCPC Project. It is one in a series of summary reports[1] that describe material characterization data obtained in the project. Each summary report captures data from a distinct round of experimentation and characterization and contains results for up to eleven different FSP nominal processing conditions for each of the five characterization modalities (see Table 1). Electronic versions of the materials described here, along with a large volume of similar data for the round, are made available via the PNNL DataHub (https://data.pnnl.gov) platform. Associated Digital Object Identifiers (doi) will be minted upon publication of the data. This report is structured as follows:

- Section 3: Sample identification and naming
- Section 4: Material characterization modalities
- Section 5: Example product metrics from material characterization
- Section 6: Structure of electronic modality data
- Attachment A: Material Characterization Results for Modality EBSD
- Attachment B: Material Characterization Results for Modality HARDNESS
- Attachment C: Material Characterization Results for Modality OPTICAL
- Attachment D: Material Characterization Results for Modality SEM
- Attachment E: Material Characterization Results for Modality ULTRASONIC

[1] A project bibliography is available in other MCPC Project publications. The report establishes a baseline reference for future reports, conference papers, and journal articles produced by the MCPC Project to build upon.

3.0 Sample Identification and Naming

Samples were produced from FSP experimentation at each nominal process condition[1], where processing conditions were programmed to be constant across the entire length of a sample. Samples were identified with a unique *Sample ID*, and replicate application of the same Condition ID on multiple sets of similar starting material produced distinct Sample IDs.

Several individual specimens were sectioned for measurement in the various modalities from each unique sample. Example specimens are shown in Figure 1. Each specimen was marked with a unique *Specimen ID* on the top surface, such as MCPC0079 (top) and MCPC0073 (bottom) for the specimens shown in Figure 1(a).[2] These specimens represented the actual test articles for testing in the material characterization rounds, and the Specimen ID provided traceability of a test article for a characterization modality to specimens from the same sample characterized by other modalities. The Specimen IDs also provided traceability to the nominal process conditions. Figure 1(b) shows the front surface of a microscopy specimen mounted in acrylic after etching in preparation for optical microscopy.

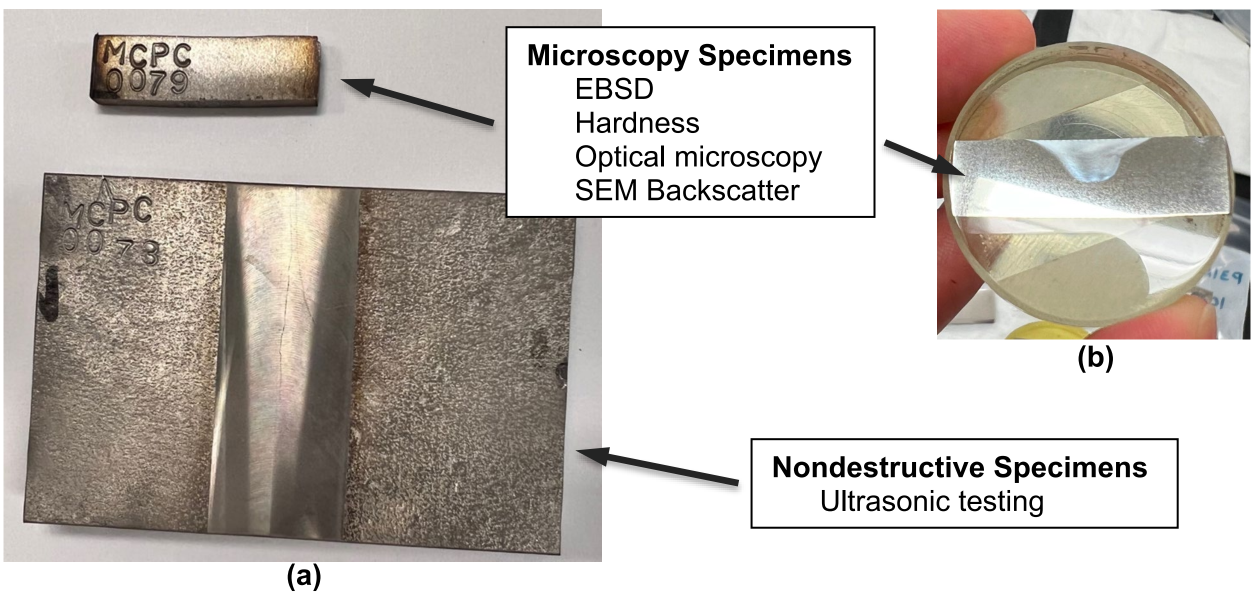

Figure 1. Example FSP specimens.

Table 2 provides a cross-reference table between Condition ID, Sample ID, and Specimen ID for each test article of the five material characterization modalities allocated to this round. The Sample ID and Specimen ID were directly used in file-naming structures as part of the traceability to individual microscopy and ultrasonic samples (see Figure 1), making this cross-reference table important for how to traverse datasets. Note that not all control specimens were measured in every modality.

[1] Actual process conditions from successful FSP experiments only deviated slightly from nominal (planned conditions).

[2] The top surface of UT specimens required light machining and sanding to smooth the surface for reliable penetration of the ultrasonic signal. The resulting specimens had a polished finish that is not present in Figure 1. The Specimen ID was reapplied after surface treatment.

Table 2. Condition, Sample, and Specimen ID matrix for each modality.

Condition ID	Sample ID	Hardness Specimen ID	Optical, SEM, and EBSD Specimen ID	UT Specimen ID
C00	SS36	MCPC0186	MCPC0187	N/A
C01	SS34	MCPC0166	MCPC0167	MCPC0168
C02	N/A	N/A	N/A	N/A
C03	SS23	MCPC0111	MCPC0112	MCPC0113
C04	N/A	N/A	N/A	N/A
C05	N/A	N/A	N/A	N/A
C06	SS21	MCPC0101	MCPC0102	MCPC0103
C07	SS22	MCPC0106	MCPC0107	MCPC0108
C08	SS17	MCPC0081	MCPC0082	MCPC0083
C09	SS18	MCPC0086	MCPC0087	MCPC0088
C10	SS19	MCPC0091	MCPC0092	MCPC0093
C11	SS20	MCPC0096	MCPC0097	MCPC0098

Condition ID “C00” represents the unprocessed control sample.

As noted earlier, samples were prepared by FSP for eleven different process conditions (plus control) to produce samples with different microstructural characteristics. The nominal process conditions and unique Condition IDs were previously listed in Table 1. Subject matter experts evaluated the resulting sample quality and time series data obtained from the FSP machinery during FSP experiments (including the actual process conditions that were compared to nominal conditions) to assess the quality of each experiment and identify if upsets occurred, such as a broken tool. Invalid samples were flagged, and samples were not tested for these FSP experiments. If this occurred, they are shown as N/A above in Table 2.

At least one successful sample was produced for each nominal process condition. The MCPC Project elected not to perform additional experimentation to replace failed experiments, as the failed experiments were for challenging conditions that risked the tool and equipment. Assignment of samples to a characterization round was performed independently of the characterization effort, and samples were traceable to a particular date/time of experimentation and the original unprocessed material.

4.0 Material Characterization Modalities

Material characterization measurements were performed for each sample. The characterization approach evolved over the rounds as lessons were learned and the desired and most useful data came into focus. This mainly involved refining the spatial sampling approach and refinement in the choice of magnification levels for the microscopy. At the operational level (not affecting data quality), preparation of samples for EBSD was refined over the rounds to reduce polishing times and to facilitate easier orientation of samples within the instrument. The characterization approach is summarized in this section for the current round to facilitate comparison to other rounds of data.

The characterization plan for Round 3 was based on obtaining coincident EBSD and SEM data[1] at five locations across the stir region where prior experience indicated variations in results could be detected. Hardness and ultrasonic testing were performed consistent with past practice. The approach for each modality is summarized below, roughly following the order of the data that was collected. See the attachments for each modality for more information, where available.

- The optical microscopy result was obtained for etched samples using a combined ocular and magnification level of 100x that focused on the FSP-affected region. Other magnification and contrast methods were not performed.
- Microhardness testing was performed for all available samples. Measurements were performed on a 0.5 × 0.5 mm grid for all samples.
- A total of five different collection sites, labeled as L01-L05, were interrogated for SEM. An example of the grid of locations is shown in Figure 2. Measurements were performed at multiple magnifications and acceleration voltages at each location with one panel per magnitude, voltage, and location. The magnification levels provided a range of data for use in machine learning applications. The typical magnifications were 3500 and 8000, but others were also used depending on the size of grains.
- A total of five different collection sites, labeled as L01-L05, were interrogated for EBSD in coincidence[1] with the SEM collection sites. An example of the grid of locations is shown in Figure 2 with hardness data from a previous round shown in the background to inform how much spatial variation might be expected. Spatial variation in grain sizes was detected and all results are listed in Attachment A. But Location L02, being proximate to the reported data in earlier rounds, was only reported in the tabulations found in the next section.
- The approach for UT was invariant across all measurements and rounds in the MCPC Project in terms of spatial extent (i.e., full volume of each UT sample), spatial resolution, transducer frequency, etc. See archived electronic files along with technical references describing this work for details.

[1] A platinum fiducial was laid down at the upper left corner of each location so the SEM and EBSD collections covered identical locations, and the same grains could be observed by both modalities.

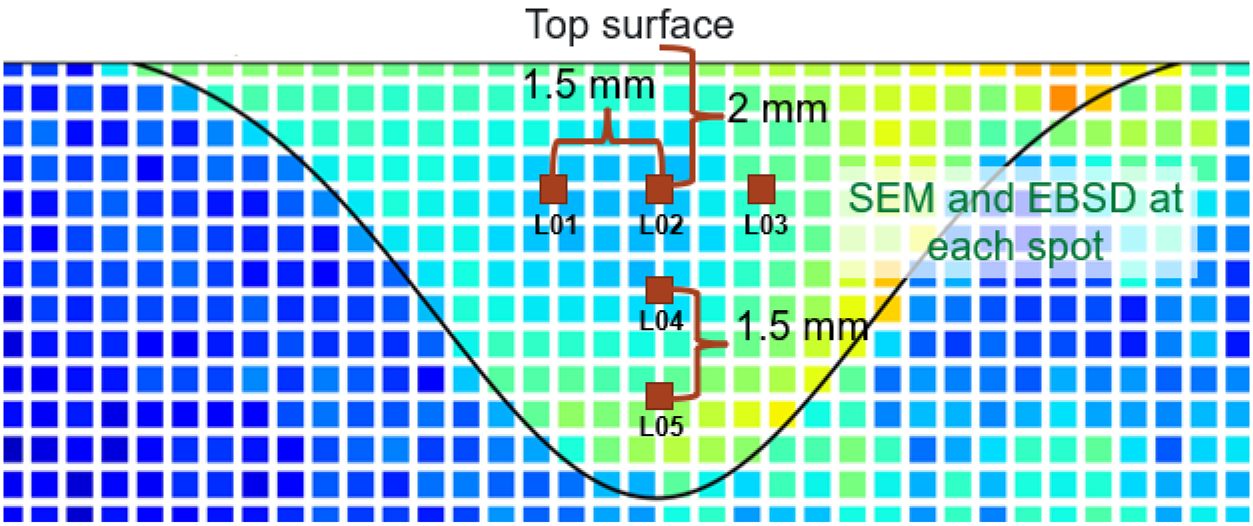

Figure 2. Example SEM and EBSD measurement locations.

5.0 Example Product Metrics from Material Characterization

The modality data (i.e., from microscopy and UT instruments) described in the prior sections was analyzed, individually and in combination, to produce a wide range of metrics for characterizing the FSP product corresponding to each nominal condition. Example product metrics typically used in production environments include grain-size distribution, grain-orientation distribution, hardness, and tensile strength. The MCPC Project elected to focus on a selected subset of these metrics. This section provides tabular and graphical representations of the product metrics that were of primary interest during the MCPC Project, but they represent just a few examples of the product metrics that can be generated from the destructive and nondestructive approaches.

5.1 Tabulations of Example Product Metrics

This section provides a tabulation of selected metric results drawn from across the modalities. First, Table 3 is a re-listing of the processing-parameter data found in Table 1, but sorted by Sample[1] instead of Condition ID. Table 4 provides a tabulation of the example product metrics. Brief descriptions of each example product metric are given below. Additional information can be found in the associated appendices and references.

- The “circle equivalent” mean grain diameter[2] was obtained from data described in Attachment A at location L02. The data was obtained using the AZtecCrystal EBSD analysis software package (Nanoanalysis 2024)
- The intercept-based grain diameter was obtained based on the application of linear paths through the grain boundaries obtained from EBSD data in general conformance with ASTM Standard E112-13 (ASTM E112-13 2021). This metric aligns with the way in which the UT method measures grain characteristics. See technical references associated with UT data analysis for more details.
- The hardness value was obtained in conformance with the Vickers hardness test method, and the listed data was from a small window near the L02 location associated with EBSD.
- The UT Figure-of-Merit (FOM) value was extracted from analysis of UT data using a frequency-domain analysis of sheer wave UT data with special treatments for transducer effects. See Guo et al. (2024) describing this work for details.

[1] Data maintained in electronic repositories is sorted by Sample ID. Therefore, data in this section is displayed in the same order.
[2] The circle equivalent diameter gives a value for area that is equivalent to the area of a segmented grain obtained by calculating the number of steps or pixels within the grain.

Table 3. FSP conditions per sample (sorted by Sample ID).

Condition ID	Sample ID	Temperature (°C)	Tool Traverse (in/min)	Tool Traverse (mm/min)	Force (lbs)	Force (kN)
C08	SS17	800	1.0	25.4	10500	46.7
C09	SS18	800	3.0	76.2	10500	46.7
C10	**SS19**	**850**	**1.0**	**25.4**	**10500**	**46.7**
C11	SS20	850	3.0	76.2	10500	46.7
C06	SS21	750	1.0	25.4	10500	46.7
C07	SS22	750	3.0	76.2	10500	46.7
C03	SS23	720	1.0	25.4	10500	46.7
C01	SS34	720	0.5	12.7	9000	40.0
C02	N/A	720	0.5	12.7	10500	46.7
C04	N/A	720	1.0	25.4	9000	40.0
C05	N/A	720	3.0	76.2	10500	46.7
C00	SS36	N/A	N/A	N/A	N/A	N/A

Table 4. Tabulated modality data per sample.

Condition ID	Sample ID	Mean Grain Diameter (μm)	Intercept-based Grain Diameter (μm)	Hardness Value	UT FOM Value
C08	SS17	2.3	3.94	229.06	0.2824
C09	SS18	1.7	3.04	233.41	0.2136
C10	**SS19**	**4.3**	**9.62**	**228.04**	**0.5772**
C11	SS20	2.7	5.94	217.99	0.4249
C06	SS21	1.3	1.92	260.67	0.0855
C07	SS22	1.6	2.50	249.47	0.1099
C03	SS23	1.0	1.38	282.53	0.0504
C01	SS34	0.8	1.04	288.96	0.0443
C02	N/A	N/A	N/A	N/A	N/A
C04	N/A	N/A	N/A	N/A	N/A
C05	N/A	N/A	N/A	N/A	N/A
C00	SS36	34.5	N/A	N/A	N/A

- Sample ID SS19 is highlighted in **Bold**. In figures shown in the next section suggests the results are anomalous compared to trends with other data.
- A tool break was noted for Condition ID C02, C04, C05 by FSP experts after the experiment.
- Data was only collected for hardness modality from the control sample, C00.
- The listed hardness value for the control sample is obtained from unprocessed regions of other samples.

5.2 Graphical Representations of Product Metrics

Graphical representations of example product metrics are provided in this section to visually illustrate trends and to identify outlier and/or suspect data from the instrument modalities. Interpretation of the trends is not provided in this report; please consult other MCPC Project publications for that information.

Figure 3 through Figure 5 illustrate the trend of mean grain diameter, hardness, and UT FOM as a function of nominal processing temperature for the process conditions. With one exception, subject matter expert assessment judges the illustrated trends reflect valid data with respect to the nominal processing conditions. As noted in the last section, the exception is related to Sample ID SS19 that is highlighted in bold in Table 3. This sample should be used with caution as a tool break may have occurred, resulting in different processing conditions than the nominal (planned) values.

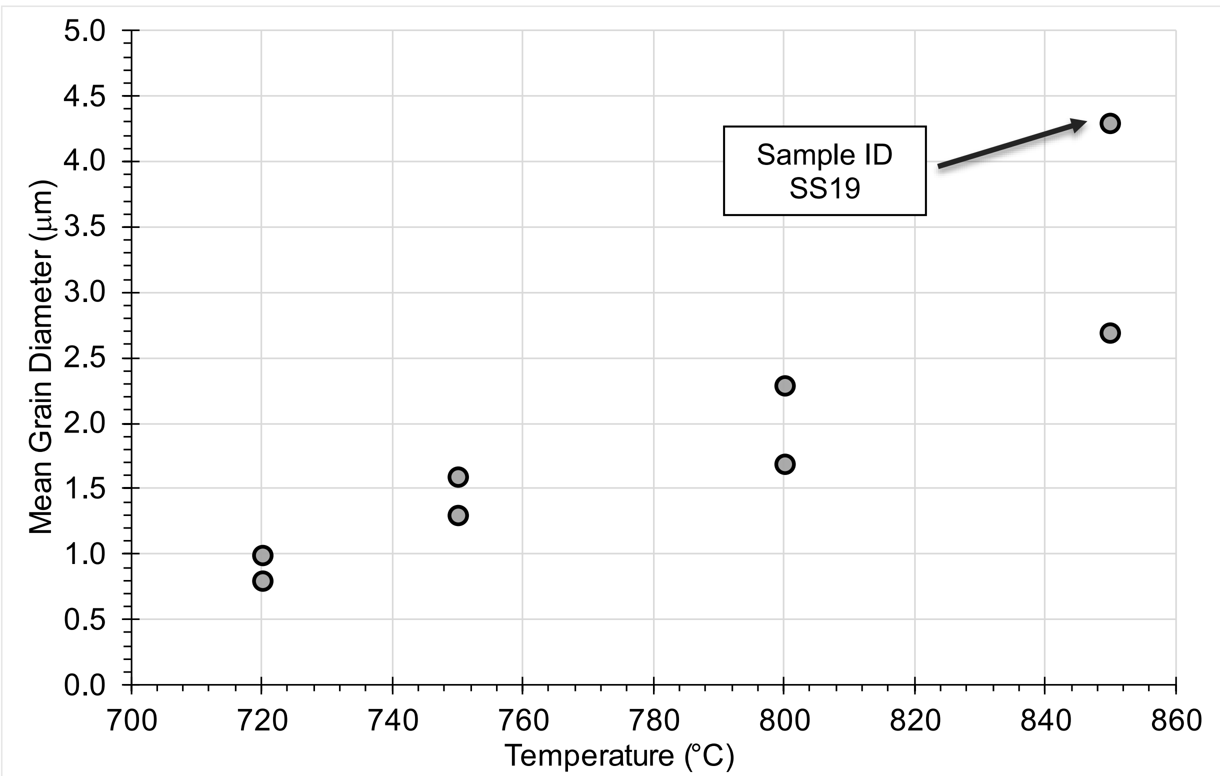

Figure 3. Circle-equivalent mean grain diameter versus nominal processing temperature.

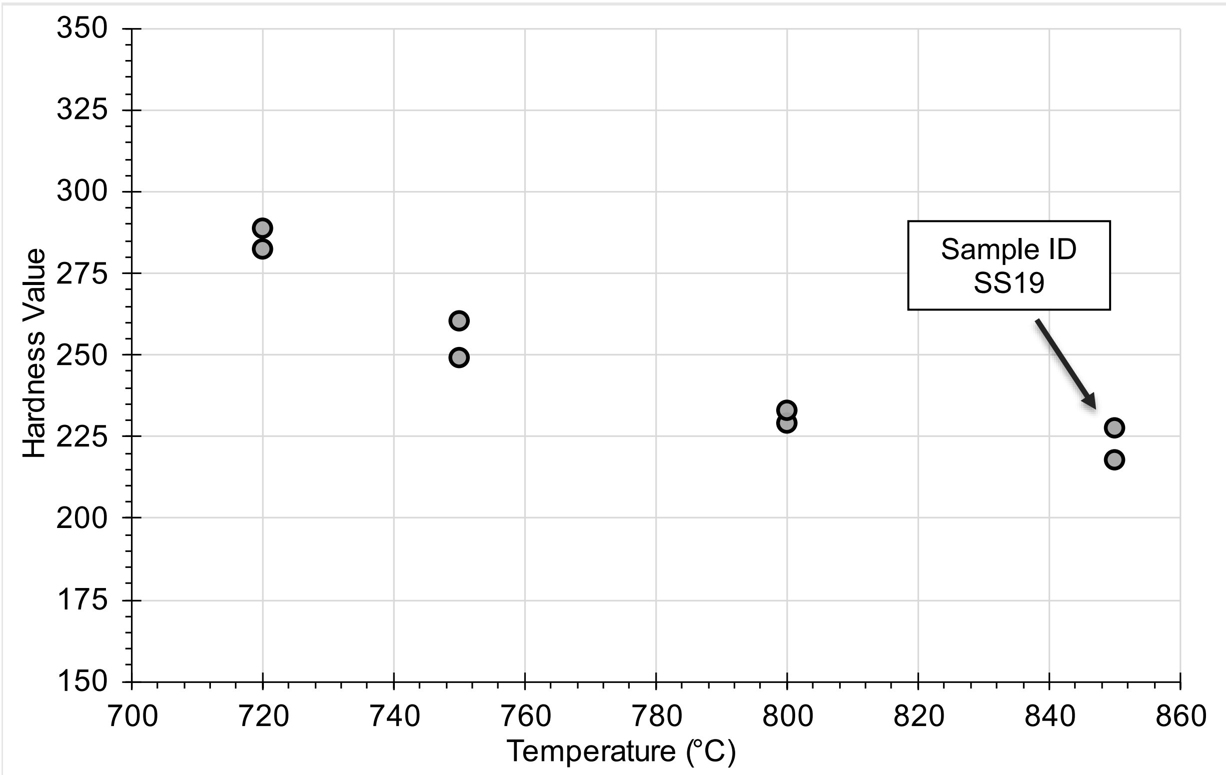

Figure 4. Hardness versus nominal processing temperature.

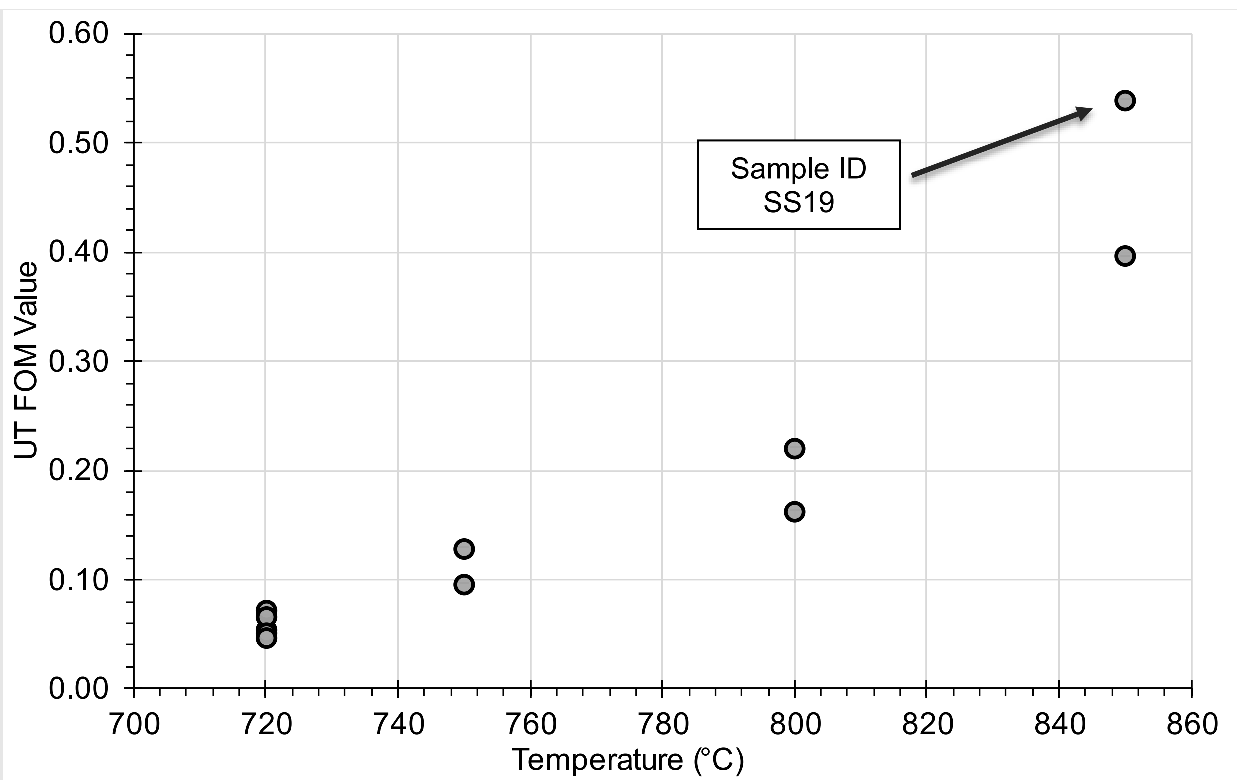

Figure 5. UT FOM nominal processing temperature.

A classical means for evaluating the structure-property relationship is to compare hardness versus the inverse square root of mean grain diameter (i.e., 1/√d). This relationship is shown in Figure 6 and validates the overall consistency of the mean grain size versus hardness data. Note that Sample ID SS28 is not an outlier in this relationship, suggesting that the hardness and grain size for that sample are consistent with each other, but as shown in prior figures, neither metric is consistent with the *nominal* (i.e., as-planned but not actual) processing condition.

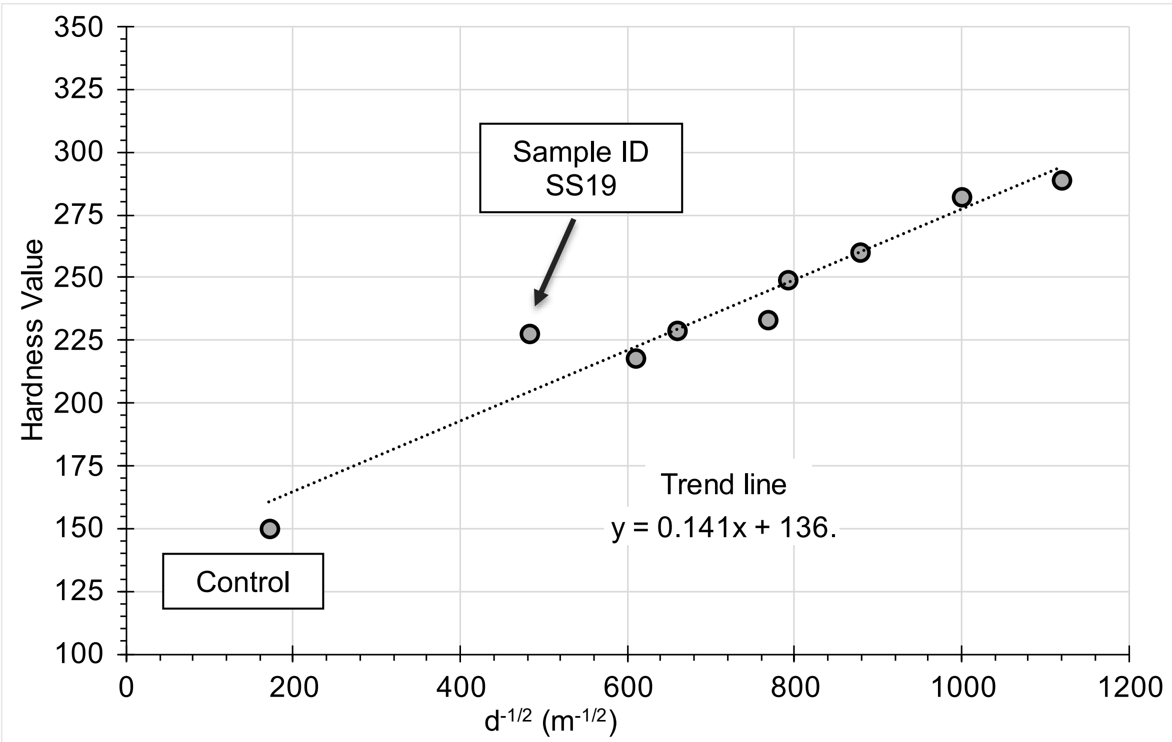

Figure 6. Hardness and mean grain size relationship.

Finally, the relationship between the UT FOM and intercept-based mean grain diameter is shown in Figure 7, fitted with an exponential trend line. The graphical trend is consistent with published work (Guo et al. 2003) for the expected theoretical shape, thus demonstrating validity of the EBSD data obtained by analyzing grain imagery using an intercept method in comparison to the UT FOM data.

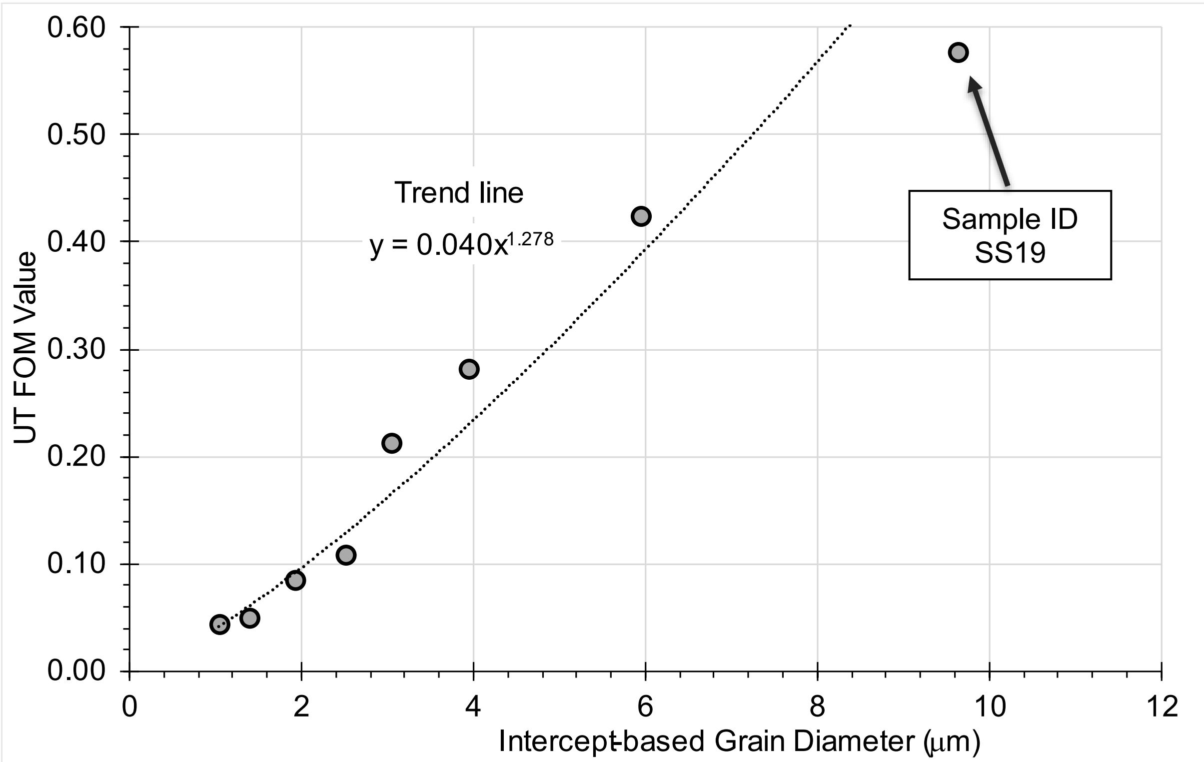

Figure 7. UT FOM versus intercept-based grain diameter.

6.0 Structure of Electronic Modality Data

6.1 Approach

The MCPC Project undertook a robust approach to curation and management of scientific data since the inception of the project. All material characterization data described in this report was managed through workflows and version control processes that are common to software development activities. The final end-product of the curation and management effort is made available via the PNNL DataHub platform.

A key objective in the overall curation and management process was to deliver systematically organized information that is human and computer readable in format and structure. To this end, a naming hierarchy and file structure that was compatible with the project workflows was used. The fundamental element of data was a dataset assigned a Dataset ID that is unique within a modality and round. The Dataset ID for microscopy data was drawn from the unique number assigned during the preparation and mounting within the metallographic preparation lab (i.e., the mount number), as this number was highly visible to the microscopist at the time of data collection. Note that a letter may be added to the end of the mount number in creating the Dataset ID if the same specimen was examined multiple times. For other data, a unique scan or log number was assigned by the data collector. This unique number differs for repeated scans of the same sample.

All information about a dataset was designed to be self-contained within an assigned directory, including raw data, analysis, visualizations, and a summary parameter file containing useful information and metadata. Interpreted results, collages of imagery, and summary data listings found outside a dataset directory were sourced to the original dataset. In case of a discrepancy or inconsistency, refer to information within a dataset directory for the primary source. In using this approach, addition of new datasets and distribution of individual datasets to collaborators were easily achieved by adding or sharing a single directory containing all pertinent information for the dataset.

6.2 Structure of Data

The top file structure of electronic data (and the attachments of this report) reflects the five modalities described at the end of Section 1.0: `EBSD`, `HARDNESS`, `OPTICAL`, `SEM`, and `ULTRASONIC`. Data from individual datasets for each modality was placed in a dedicated subdirectory named by a Sample ID and Dataset ID. Furthermore, as noted in the last section, datasets associated with a re-measurement, if present, had a unique letter appended at the end of the name or other means to ensure uniqueness within the modality files.

The key “raw” files from each measurement were placed in the `DATA` directory of the dataset. These file(s) were typically obtained directly from the instrument. A digital fingerprint was captured in metadata for key files for traceability purposes. Derived visualizations (rescaling or cropping of an optical image, for example), production of numerical data (such as grain size data from EBSD), and interpretation are obtained from these files. The derived information is organized in `ANALYSIS` or `VISUALIZATION` directories. See discussion for each modality in the attachments regarding contents.

A metadata parameter file in the human and computer readable YAML[1] file format was created for each dataset. This parameter file contained important information about the individual dataset. It also captured comments related to the validity of the dataset. A dataset may be determined to be invalid because the underlying FSP experiment was determined invalid—see the curated FSP data for the source of which experiments were determined to be invalid. Or the data collection was affected in some manner that affected data quality.

A file named `{modality}_METADATA_LISTING` was produced in several file formats at the root of each modality directory. This file contained a convenient collation of all data in the metadata parameter files described above for the modality and permitted easy inter-comparison and lookup of information across all datasets. In case of discrepancy between the summary file and individual dataset parameter file—data in the individual dataset files takes precedence.

Finally, useful collections of information from across multiple datasets of a modality and relevant summary results files were placed in the `ENSEMBLE_DATA` directory (if present). These files were designed to collect key results from all the datasets into tabular listings for convenience.

An example file structure is illustrated in Figure 8 using example Sample ID, Specimen ID, and Dataset ID values. The arrangement of file naming was carefully designed to assist data generators and data consumers in finding information while reducing the likelihood of unintentional use related to the Condition ID, Sample ID, Specimen ID identification scheme listed in Table 2.

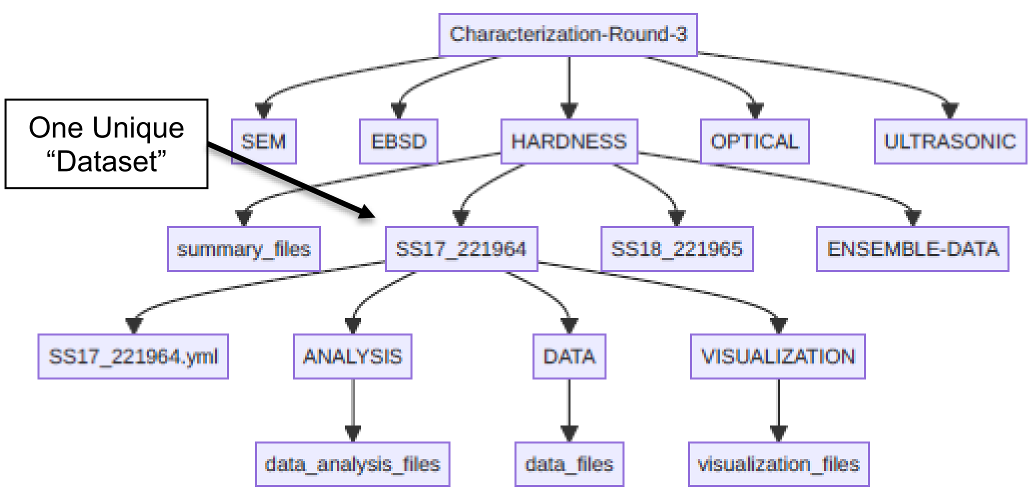

Figure 8. Illustration of typical file structure.

[1] YAML is a widely used text-based file format supported in most modern programming languages for saving and reading data. The YAML name is not considered an acronym, though it originated as one.

As shown in the figure, an individual dataset is placed into a directory named `{Sample ID}_{Dataset ID}` (for example SS17_221964). The parameter file for the dataset used this same naming. The use of `{Sample ID}` in the name immediately tied a dataset to the same sample found in other modalities—it also yielded a consistent sort order of datasets across modalities. The `{Dataset ID}` was a unique value, but it has no connection to the processing conditions. The modality data files in directory `{Sample ID}_{Dataset ID}/DATA` are given the name `{Specimen ID}_{Dataset ID}`. This naming was used to reduce human-performance mistakes because `{Specimen ID}` is physically stamped on a specimen and is the only visual association with the original experiment. Recall that two specimens from the same sample were expected to be metallurgically identical, and these identical specimens were typically characterized by different modalities to account for different sample preparation requirements. As noted earlier, a unique Dataset ID was defined within the modality if the same specimen is measured multiple times.

A readme in each modality subdirectory provided additional detail and discussion for the data and results for that modality. Much of this readme text is reproduced in the attachments to this report.

An example of a metadata parameter file in YAML file format is shown in Figure 9. As seen in the example, this file contained metadata, the status of the dataset, a listing of key "raw" files that included the first 10 digits of an SHA1[1] digital fingerprint for the file, spatial and geodetic information, and other notes about the individual dataset. This file type was chosen because it is easily loaded into a "dictionary" data type by tools such as Python while also being human readable.

---

[1] SHA1, or "Secure Hash Algorithm 1", is a cryptographic hash function that produces a 160-bit message digest from an input that can be used to verify the authenticity of electronic information.

```yaml
Operator: Anthony Guzman
Date: '2022-05-25'
Time: '13:41:51'
Dataset ID: '22742'
Specimen ID: MCPC0006
Sample ID: SS02
Condition ID: C05
Modality: OPTICAL
Instrument ID: 'Olympus, Model: DP74'
Dataset status: Normal
Dataset status notes: Tool pin was chipped after this run. It is unclear
exactly where the chipping occurred from processing conditions
Key data files:
    MCPC0006_22742_10X_Nugget-Region-Etched.jpg: 1aa04623ff
    MCPC0006_22742_5X_Montage-DIC.jpg: 5b2fb1b468
    MCPC0006_22742_5X_Montage-Etched.jpg: eb221906c8
Spatial:
    Stir center:
        Width: '18387.0'
        Height: '5698.0'
    Rotation: '2.1'
    Scale: 1704.5
    Scale units: pix/mm
    Notes: For 10x etched data. Spatial location & rotation data to align the
center of the stir region is only approximate at this time.
Notes: NONE
```


Figure 9. Example YAML metadata parameter file.

As noted earlier in this section, “raw” data, interpreted data, and derived visualizations of the data were placed into separate directories. The general principle was that all interpreted data and visualizations could be re-derived and are traceable to the raw data listed in the parameter files. All interpreted results were located within the dataset directory and a comingling of results across datasets or modalities could trace the data source to the individual dataset directory.

7.0 References

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Pacific Northwest National Laboratory

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99354

1-888-375-PNNL (7665)

www.pnnl.gov

Attachment A: MCPC Round 3 Material Characterization Results for Modality EBSD

M Poole DR Todd
K Nwe A Guzman

Attachment A contains 40 pages

Acknowledgments

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Contents

Figures

Tables

1.0 Introduction

Electron Back Scatter Diffraction (EBSD) is a microscopy technique that can measure microstructural properties such as grain sizes, crystallographic orientations, misorientations, texture, and others. The primary property of interest within the scope of the MCPC Project is circle equivalent mean grain size. Additionally, the grain boundary imagery is utilized for some elements of the project. Phase discrimination, analysis of texture, and other properties is not undertaken even though the information is contained in the results.

The following sections provide a summary of results for this modality to enhance dissemination of the large volume of similar data that are made available via the PNNL DataHub (https://data.pnnl.gov) platform.

2.0 Key Information and Results

The main portion of this report provides a summary of key information related to the modality, including processing conditions, sample identification information, and the numerical results retrieved from EBSD data analysis in the project. Where to find the information and results in the main report is described below (Section, Table, and Figure numbers listed below are found in the main report):

- Nominal process conditions for the FSP experiments are defined in Table 1 and Table 3 (data is in two different sorting orders).
- The Condition, Sample, and Specimen ID matrix is defined in Table 2 that is necessary to decode the nominal conditions applied to a particular specimen.
- Details about the modality data collection approach is defined in Section 4.0. This includes information about sites where EBSD is performed.
- Tabulation of the modality results considered most important (mean grain diameter) per Sample ID is listed in Table 3 with discussion of what the particular result means introduced earlier in the section.
- Mean grain diameter value versus nominal processing temperature listed in Table 3 and Table 4 is displayed graphically in Figure 3.
- A classical means for evaluating the structure-property relationship is to compare hardness versus the inverse square root of mean grain diameter. This is shown in Figure 6 to highlight consistency of results across a range of conditions.

AZtecCrystal https://nano.oxinst.com/azteccrystal is a comprehensive software package distributed by Oxford Instruments for processing data collected using EBSD. This software package generates numerous visualizations and tabulated results based on user specification. Tabulated results include various grain size statistics, via circle equivalent diameter, which was listed in the main report for location L02. The source of tabulated data can be found in the right-hand side of the ”Grain Charts” shown in Section 7.1, below.

3.0 Instrumentation and Measurement Locations (Sites)

The following instrument was utilized in testing of specimens:

Thermoscientific Helios Hydra UX, Plamsa Focused Ion Beam microscope

EBSD results were obtained at multiple locations (sites) for each specimen as described in Section 4.0 and illustrated in Figure 2 in the main report. The distinct locations were labeled L01 through L05 as illustrated in the figure. Each interrogation produced results files labeled with the related location code.

4.0 Results Files

Below is a summary of the unique results files produced for each location where measurements were made for each specimen. Items after the first entry describe derived data files that are provided, where the indicated string is found in the file name.

- h5oina (DATA Directory, Extension): The ”HDF5 Oxford Instruments NanoAnalysis” file format contains all data captured during an EBSD measurement session. All subsequent visualizations, data analysis, and information is obtained from this key file. Generally, proprietary software (AZtecCrystal) is needed to extract visualizations and perform data analysis. Data is contained in the HDF5 format. The specification for this file format can be found here: https://github.com/oinanoanalysis/h5oina.

- GB+BC_HAGB (VISUALIZATION Directory, TIF Image): EBSD results image showing the band contrast image overlaid with grain boundaries identified through segmentation. The overlays only indicate high angle grain boundaries.

- GB+BC_LAGB+HAGB (VISUALIZATION Directory, TIF Image): EBSD results image showing the band contrast image overlaid with grain boundaries identified through segmentation. The overlays indicate both low angle (in red) and high angle grain boundaries.

- IPF-X+GB (VISUALIZATION Directory, TIF Image): EBSD Inverse pole figure (IPF) map in the X-direction with grain boundaries.

- IPF-Y+GB (VISUALIZATION Directory, TIF Image): EBSD IPF map in the Y-direction with grain boundaries.

- IPF-Z+GB (VISUALIZATION Directory, TIF Image): EBSD IPF map in the x-direction with grain boundaries.

- Grain-Chart (ANALYSIS Directory, TIF Image): Distribution chart of grain sizes.

- Grain-Chart-Data (ANALYSIS Directory, CSV file): Text listing of the distribution chart of grain size data.

Note that all visualizations and grain chart analysis were produced with a grain misorientation angle threshold of 10 degrees. A subdirectory in the ANALYSIS directory may contain grain chart data with other threshold settings as indicated in the directory name.

5.0 Notes and Comments

The following observations are made about data and files for this modality:

1. None

6.0 Tabular Results

Mean grain diameter for locations L01-L05 is listed Table 6.1 in units of microns. This data is obtained from the EBSD_Grain-Data-Results.xlsx file described earlier. The file contains additional tabular data transcribed from the right side of each Grain Chart shown in Section 7.1.

Table 6.1. Listing of mean grain diameter.

Sample ID	L01	L02	L03	L04	L05
SS17	2.1	2.3	2.5	2.1	1.5
SS18	1.7	1.7	1.8	2.0	1.6
SS19	3.4	4.3	5.1	3.3	2.2
SS20	2.6	2.7	2.8	2.1	1.3
SS21	1.3	1.3	1.3	1.2	1.0
SS22	1.5	1.6	1.5	1.2	0.8
SS23	0.9	1.0	1.0	0.9	0.7
SS34	0.9	0.8	0.9	0.8	0.7
SS36	34.5				

7.0 Graphics

The following sub sections display key graphics obtained from EBSD analysis.

7.1 Grain Charts

The following figures show graphical results for all datasets associated with location L02 in the round. These graphs indicate area-weighted fraction of grains measured for the specimen as a function of equivalent circle diameter in the form of histograms. Grain sizing settings and numerical results are displayed on the right of the figure for grain count (number of grains), mean (circle equivalent) grain diameter, area weighted mean, minimum, maximum, and standard deviation of the grain size distribution, and misorientation angle threshold. The mean grain diameter displayed above in Table 6.1 is transcribed from this figure. The other results are available in file EBSD_Grain-Data-Results.xlsx.

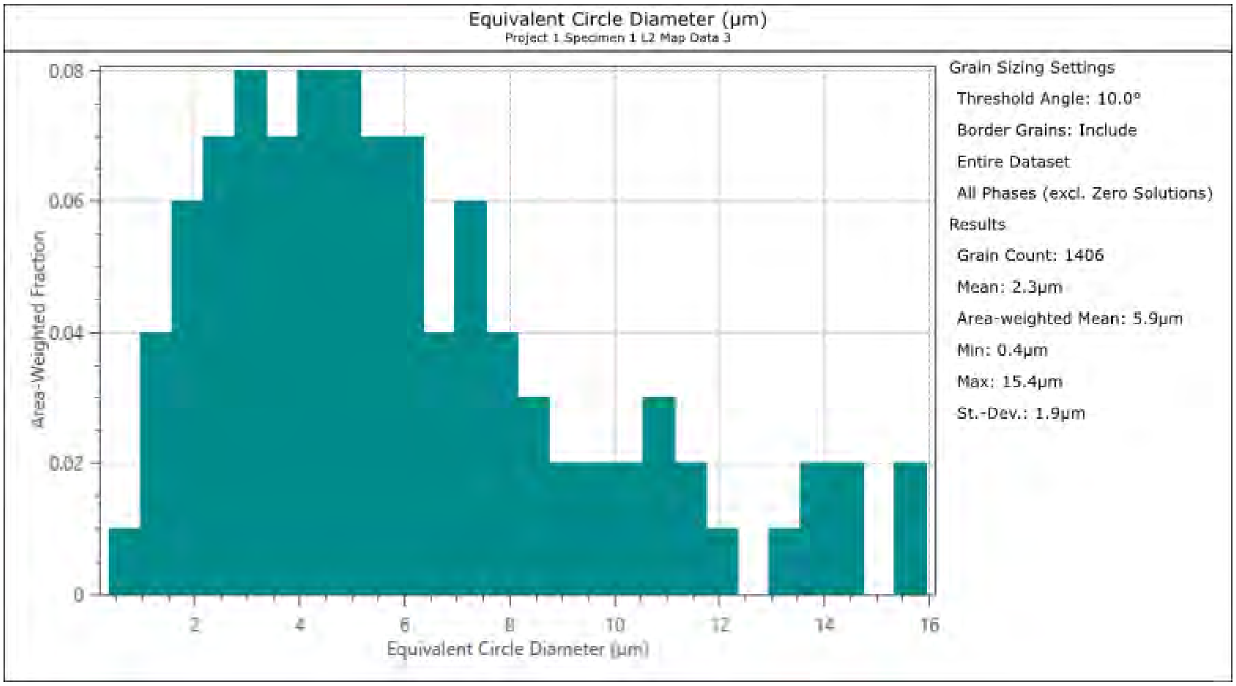

Figure 7.1. CED Grain Size and Other Data for L02: Condition C08, Sample SS17

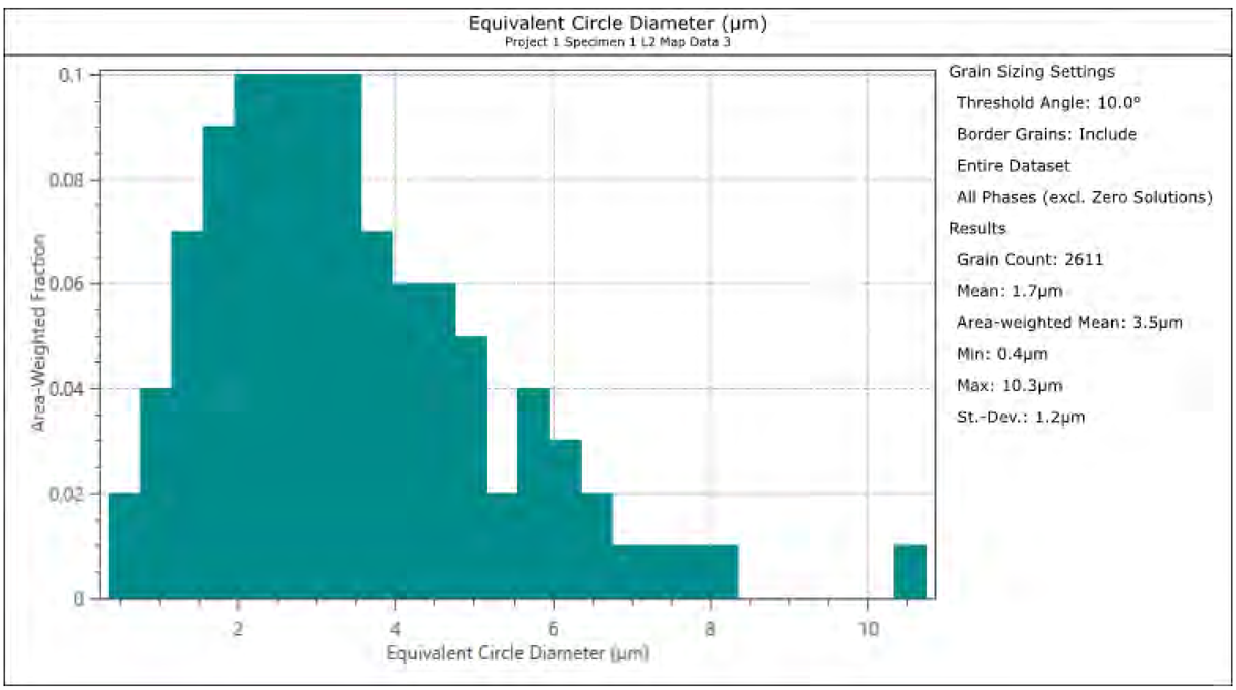

Figure 7.2. CED Grain Size and Other Data for L02: Condition C09, Sample SS18

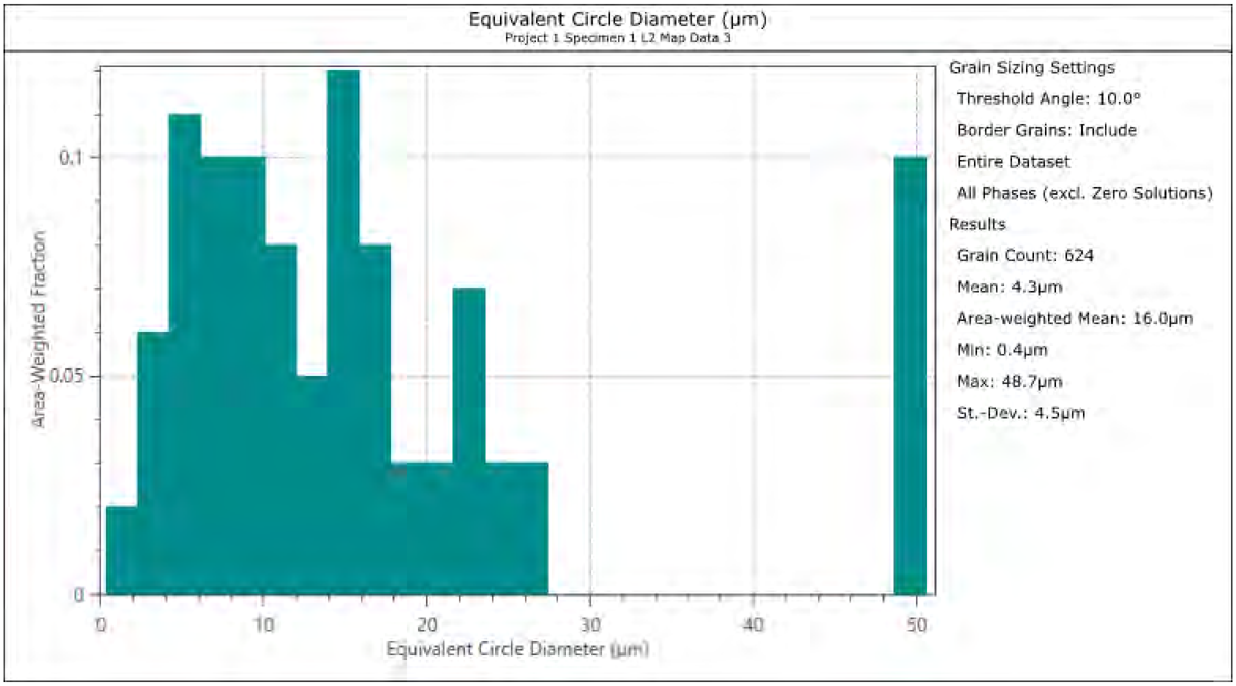

Figure 7.3. CED Grain Size and Other Data for L02: Condition C10, Sample SS19

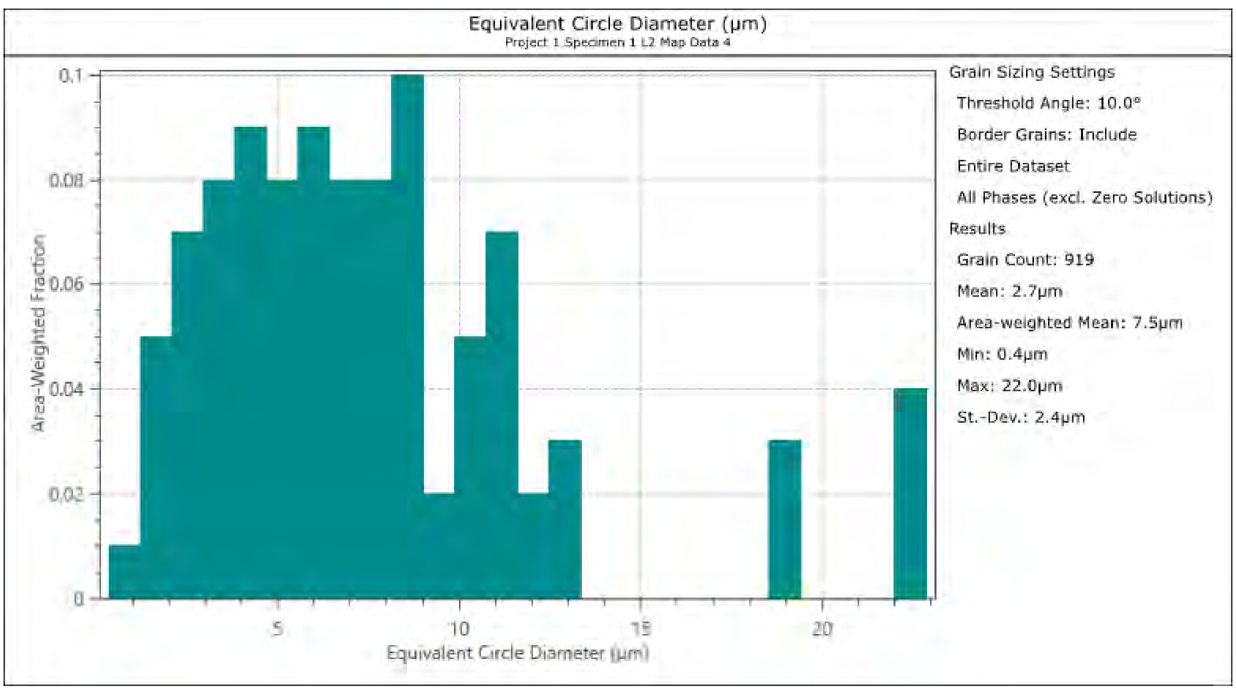

Figure 7.4. CED Grain Size and Other Data for L02: Condition C11, Sample SS20

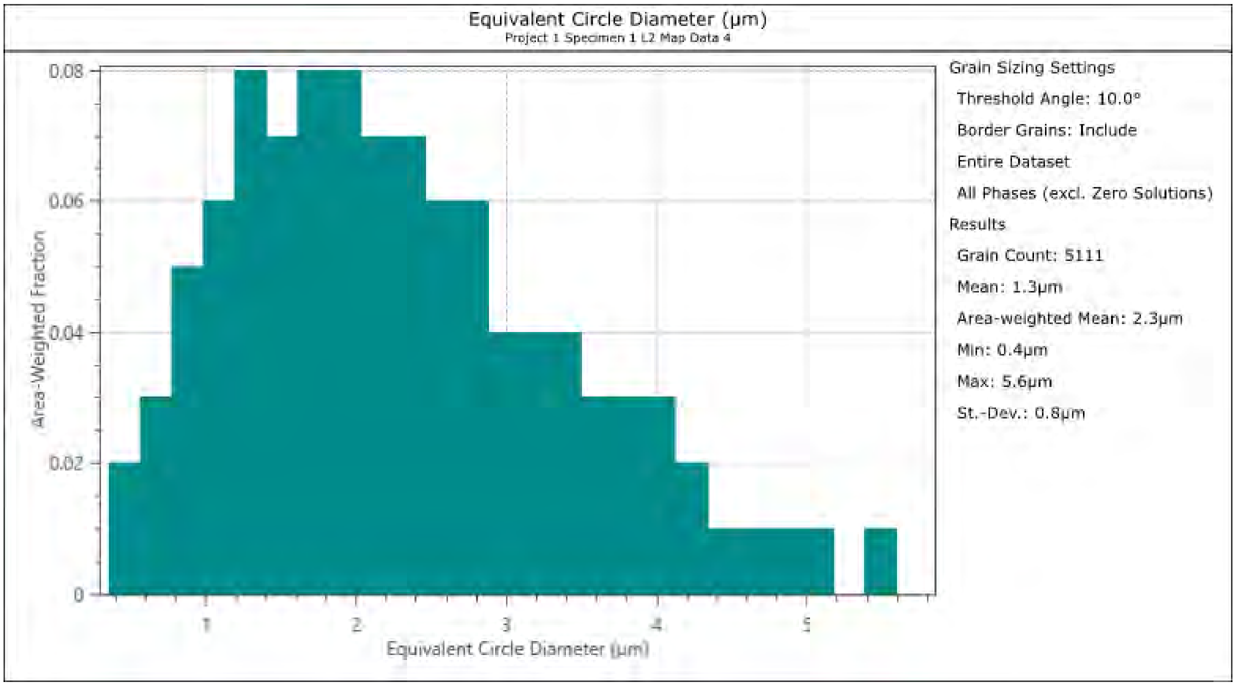

Figure 7.5. CED Grain Size and Other Data for L02: Condition C06, Sample SS21

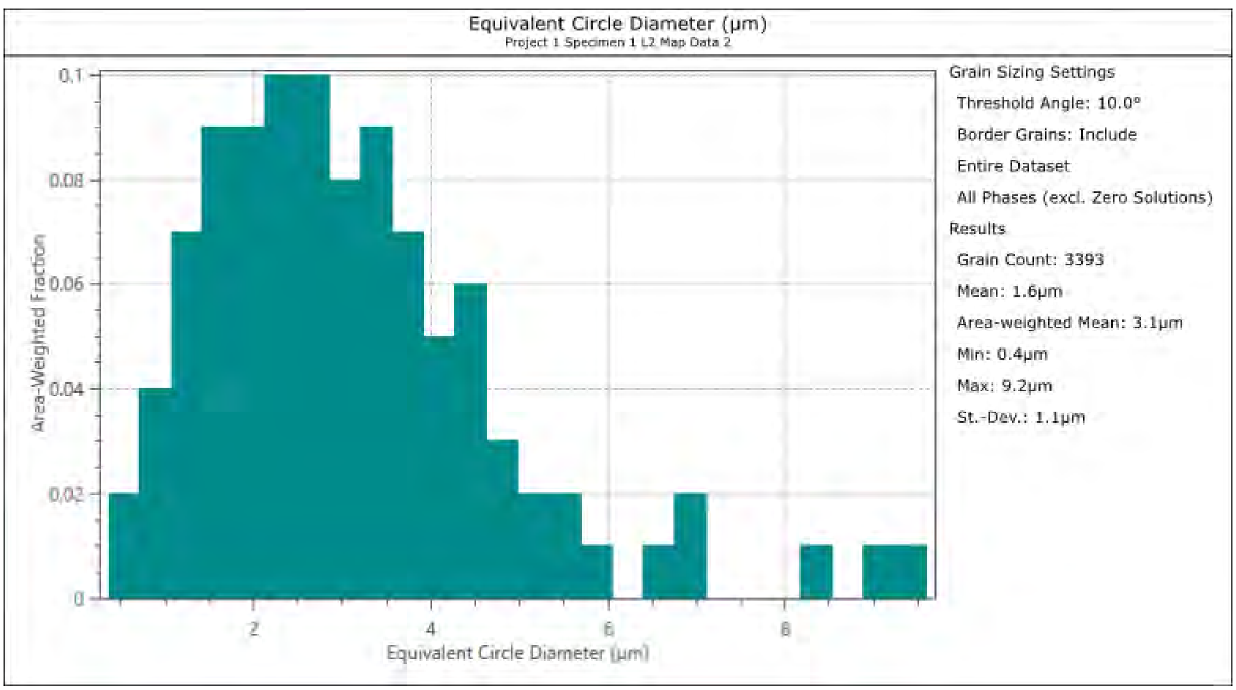

Figure 7.6. CED Grain Size and Other Data for L02: Condition C07, Sample SS22

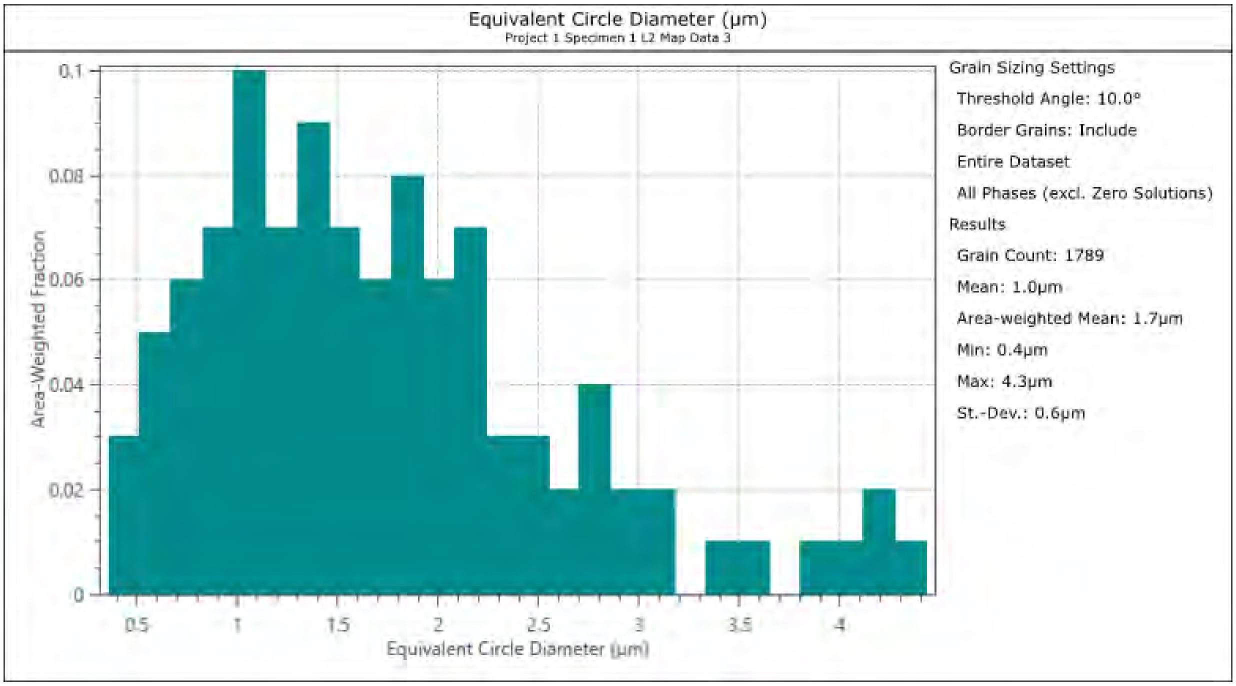

Figure 7.7. CED Grain Size and Other Data for L02: Condition C03, Sample SS23

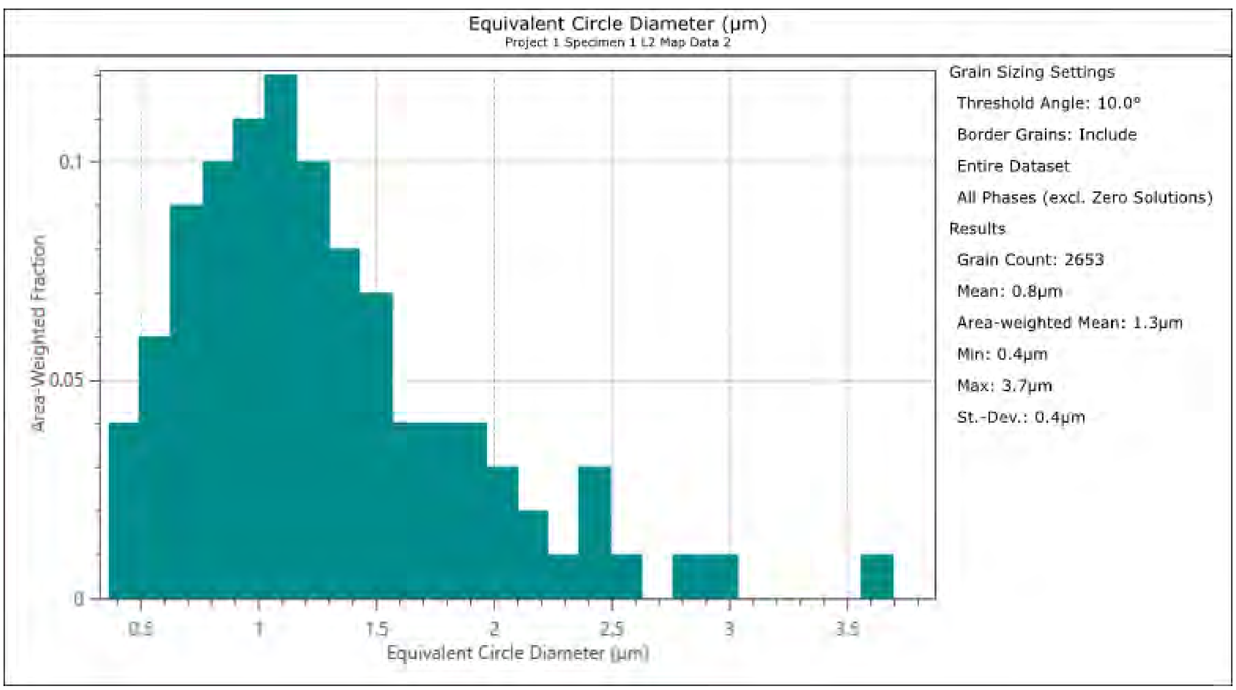

Figure 7.8. CED Grain Size and Other Data for L02: Condition C01, Sample SS34

7.2 Band Contrast + High Angle Grain Boundaries

The following figures show the grain boundaries identified through segmentation with a setting of 10° the misorientation angle, overlaid on the band contrast image of the microstructure. These images are the ones utilized to calculate the intercept-based grain diameter as briefly discussed in the main report.

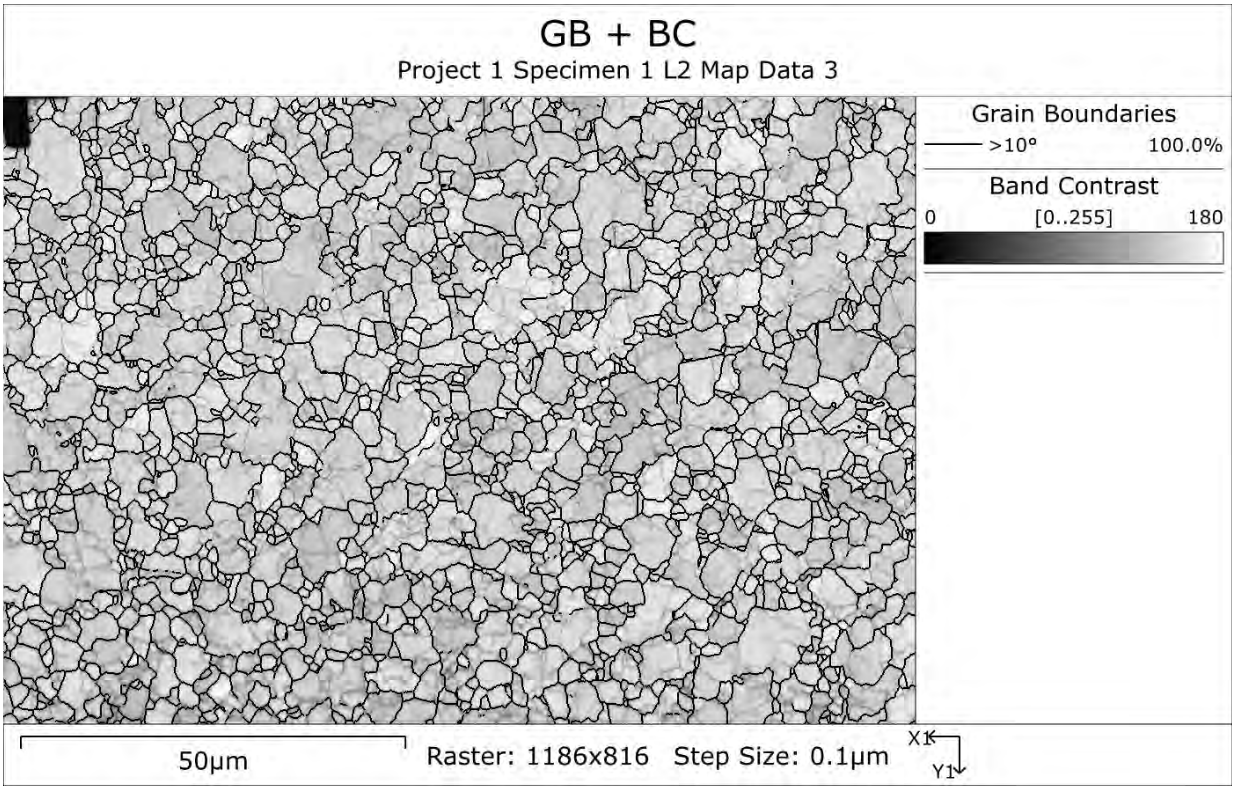

Figure 7.9. High Angle Grain Boundary + Band Contrast for L02: Condition C08, Sample SS17

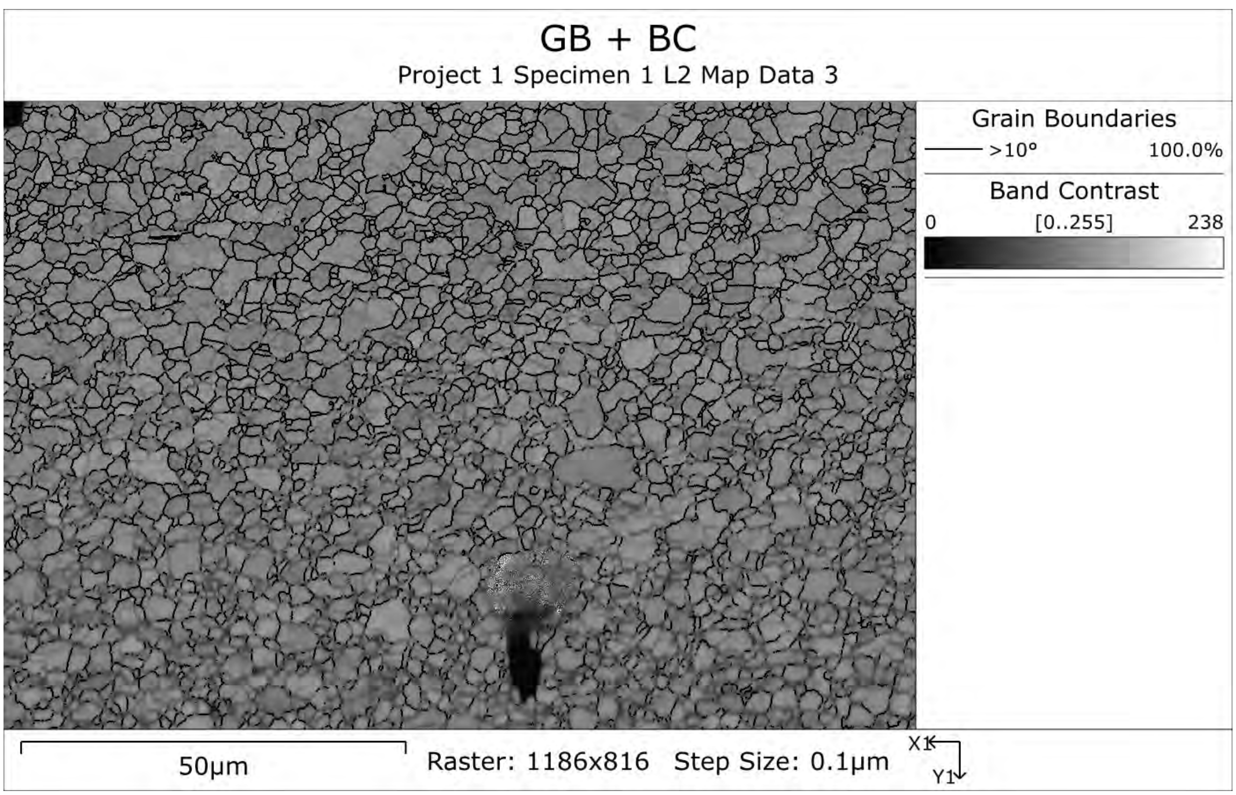

Figure 7.10. High Angle Grain Boundary + Band Contrast for L02: Condition C09, Sample SS18

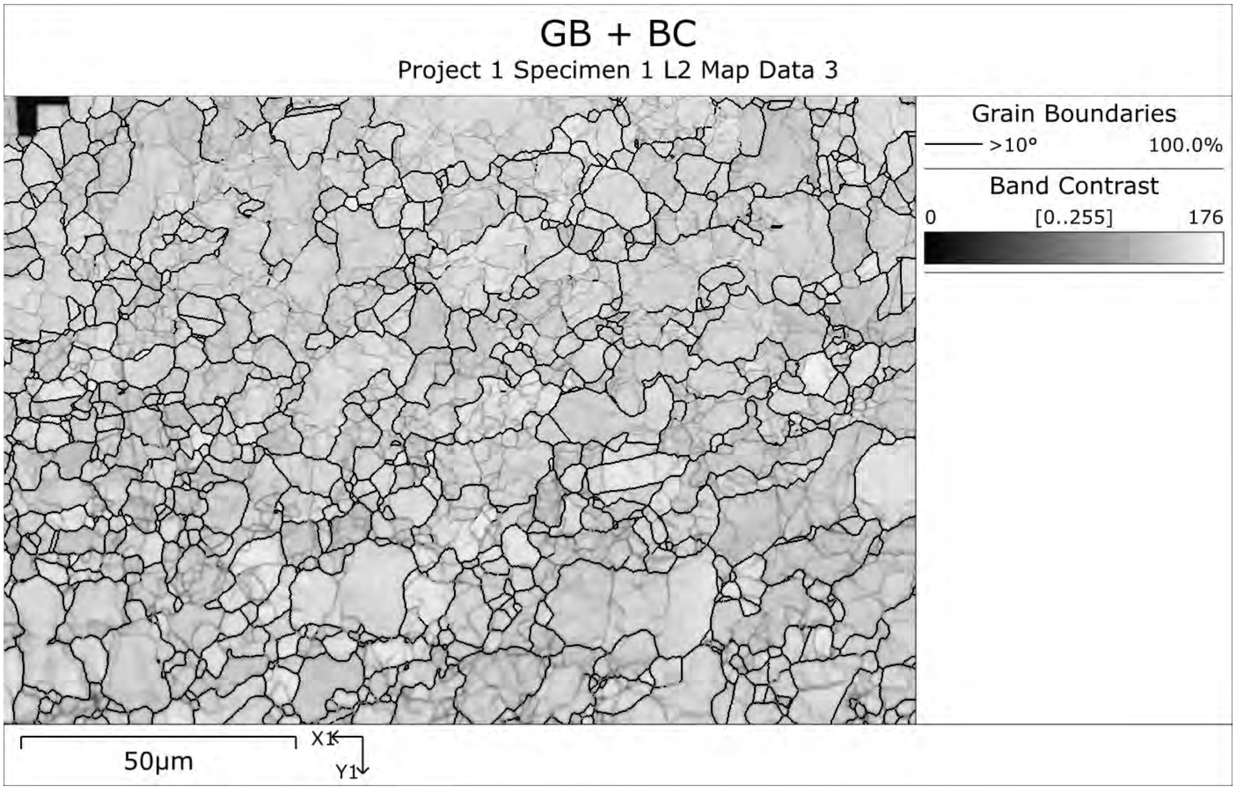

Figure 7.11. High Angle Grain Boundary + Band Contrast for L02: Condition C10, Sample SS19

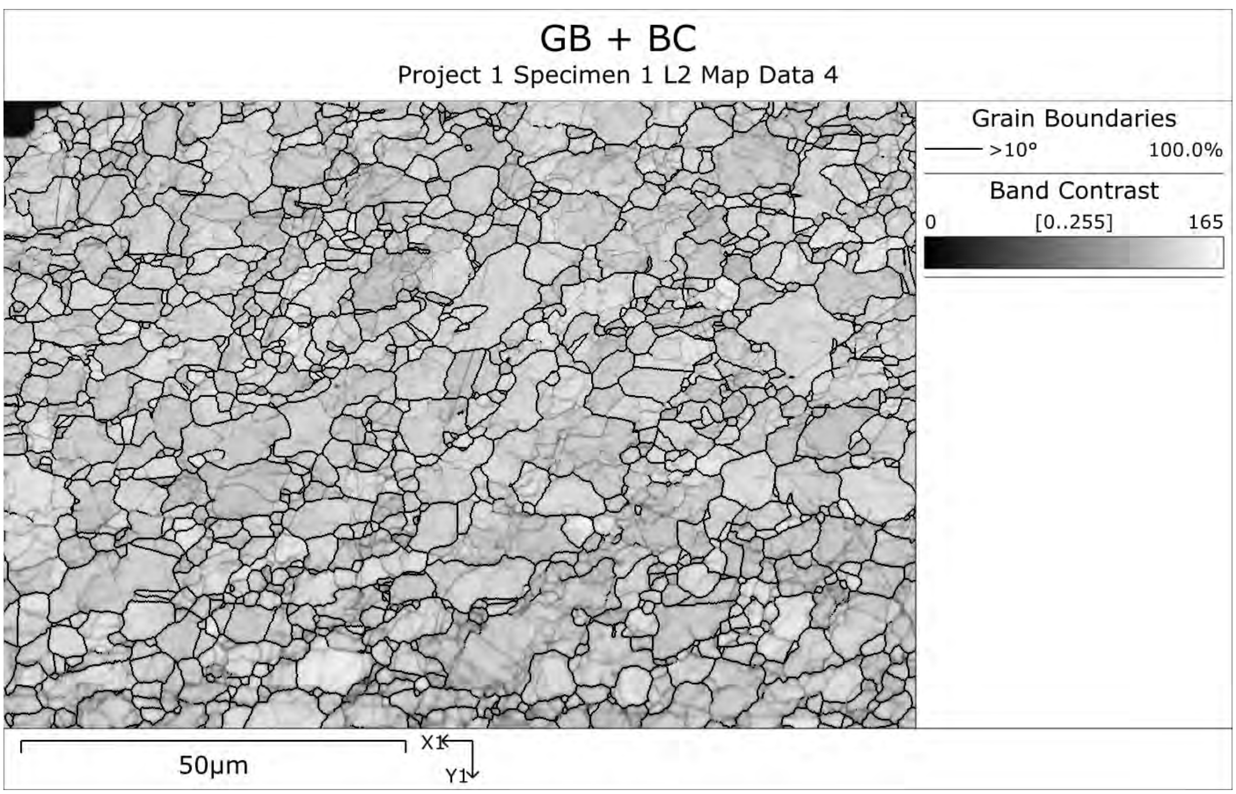

Figure 7.12. High Angle Grain Boundary + Band Contrast for L02: Condition C11, Sample SS20

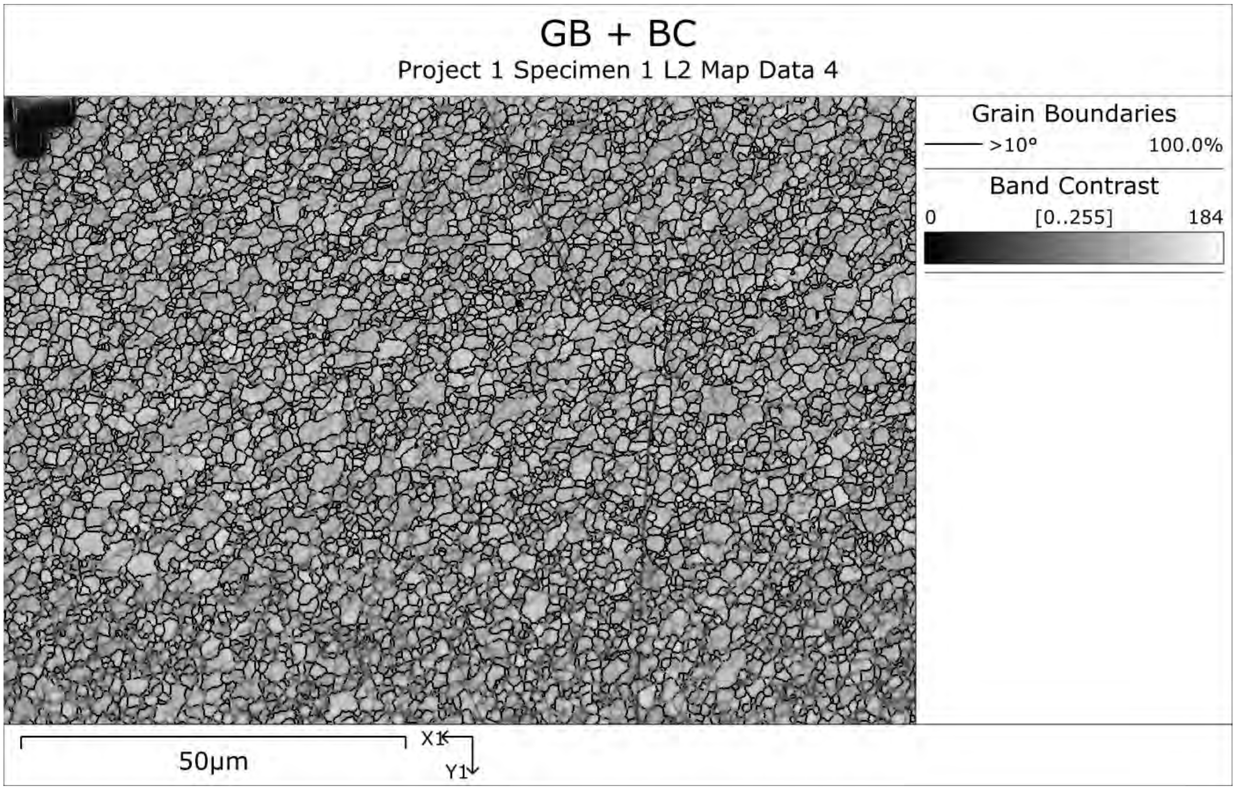

Figure 7.13. High Angle Grain Boundary + Band Contrast for L02: Condition C06, Sample SS21

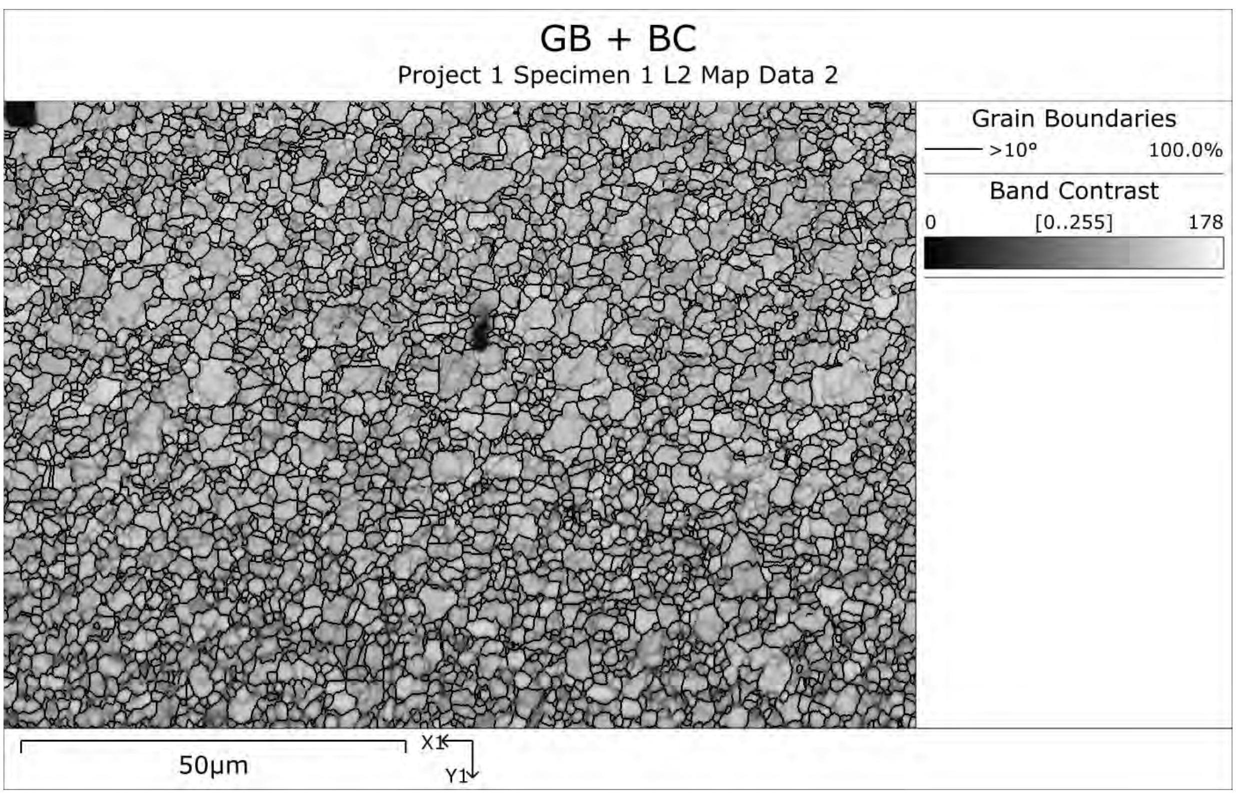

Figure 7.14. High Angle Grain Boundary + Band Contrast for L02: Condition C07, Sample SS22

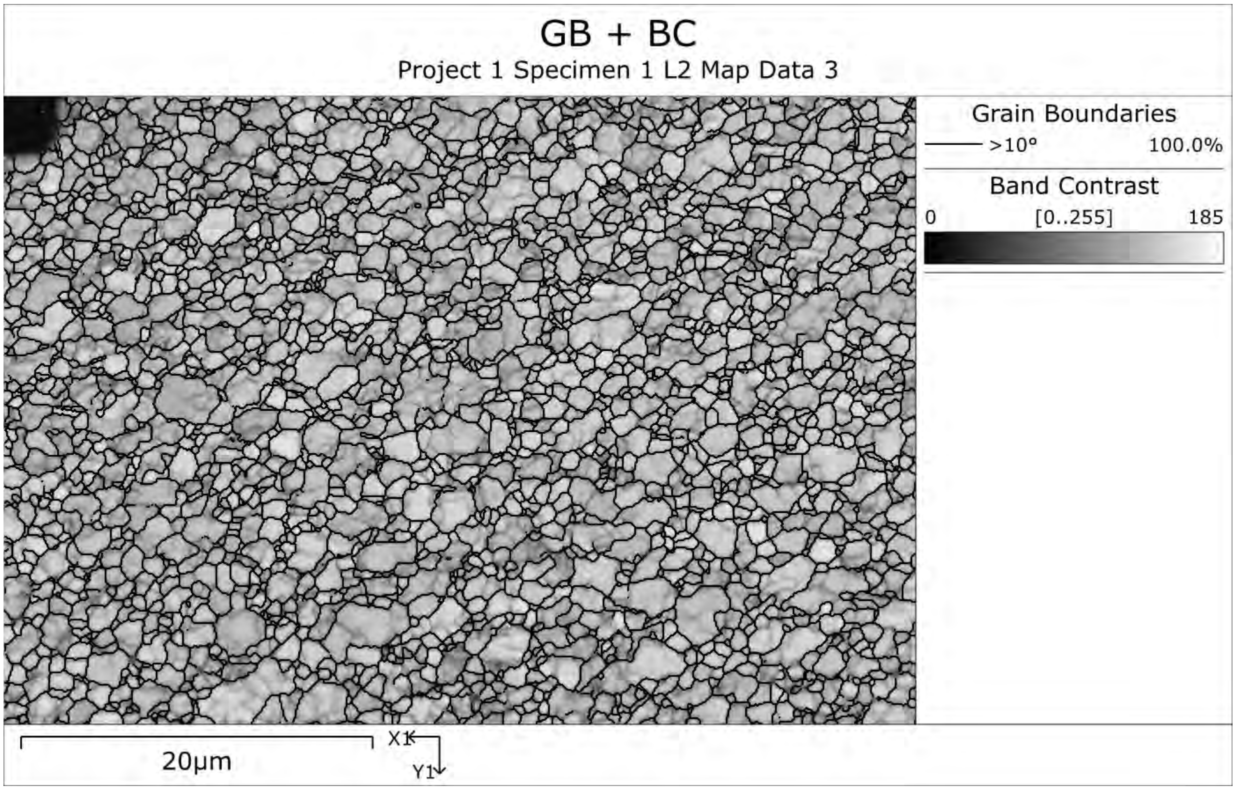

Figure 7.15. High Angle Grain Boundary + Band Contrast for L02: Condition C03, Sample SS23

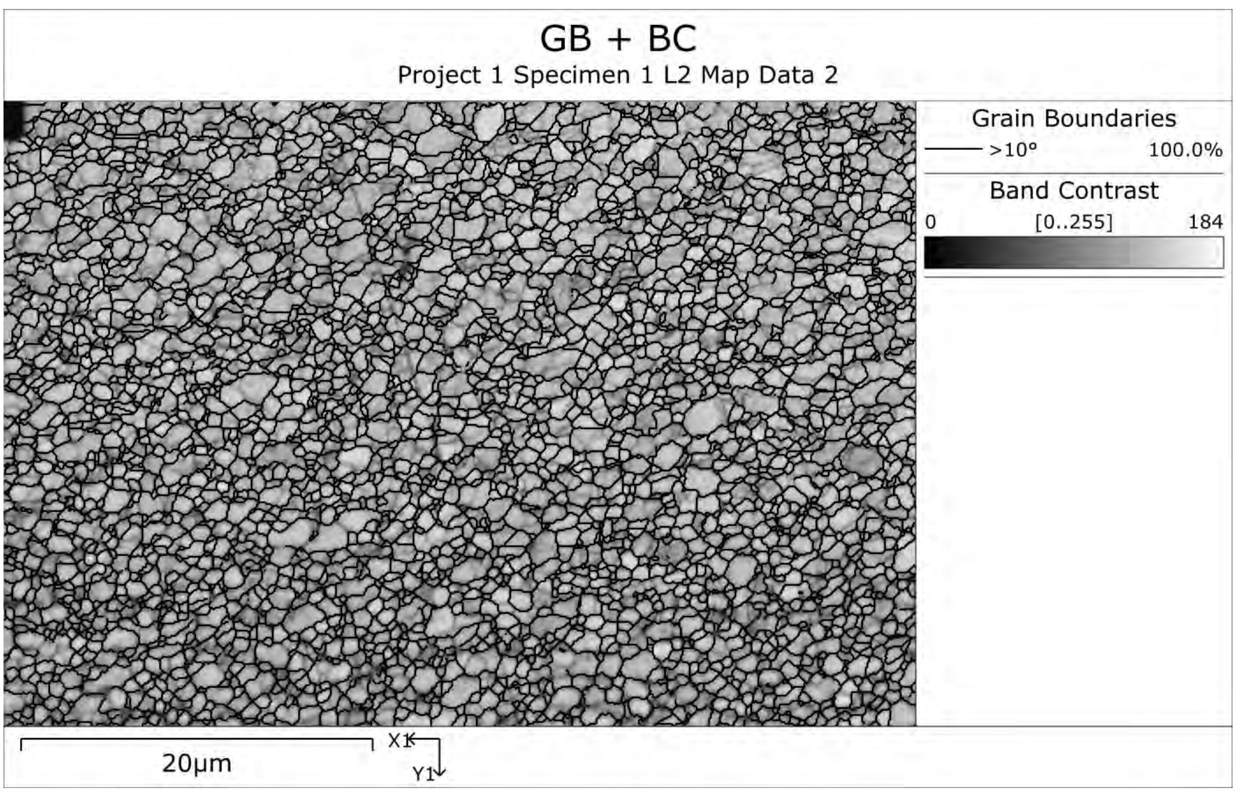

Figure 7.16. High Angle Grain Boundary + Band Contrast for L02: Condition C01, Sample SS34

7.3 Band Contrast + Low & High Angle Grain Boundaries

The following figures show the grain boundaries identified through segmentation with a setting of 10° the misorientation angle to identify high angle grain boundaries and 2° to identify low angle grain boundaries (shown in red on the images), overlaid on the band contrast image of the microstructure.

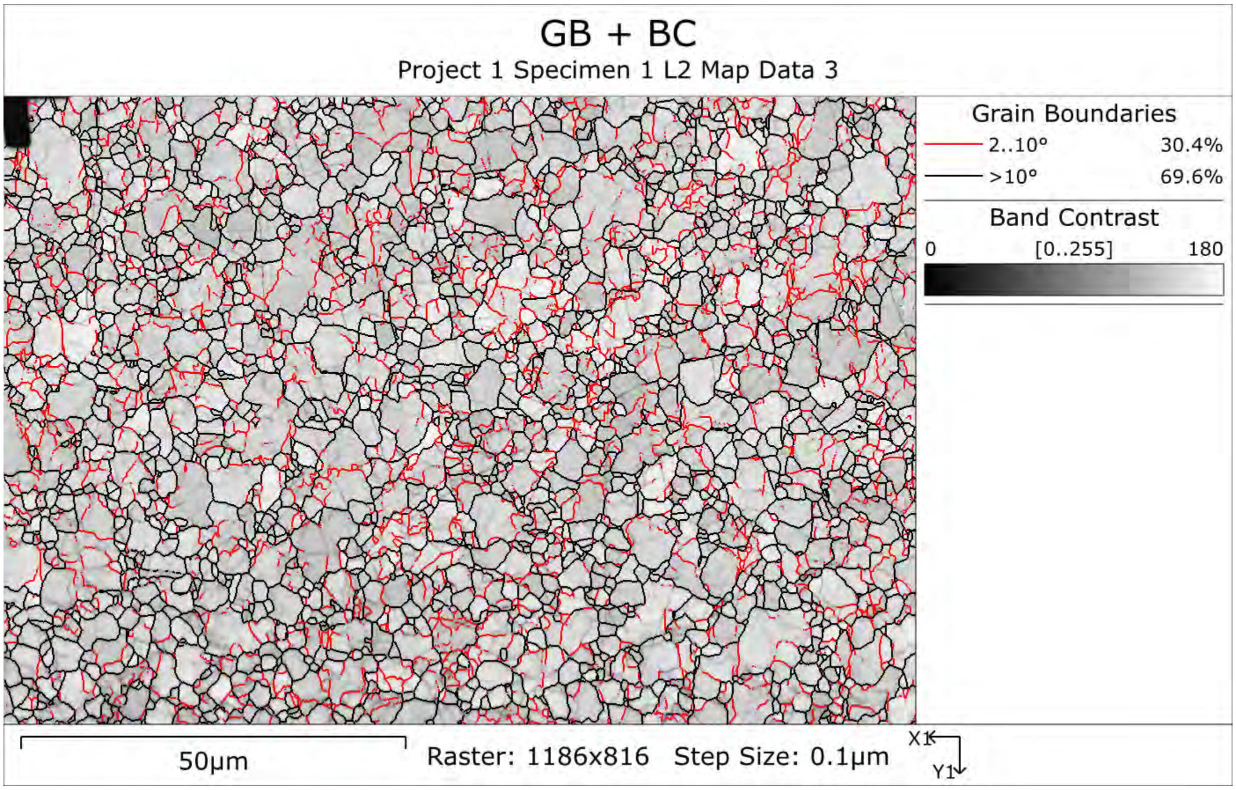

Figure 7.17. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C08, Sample SS17

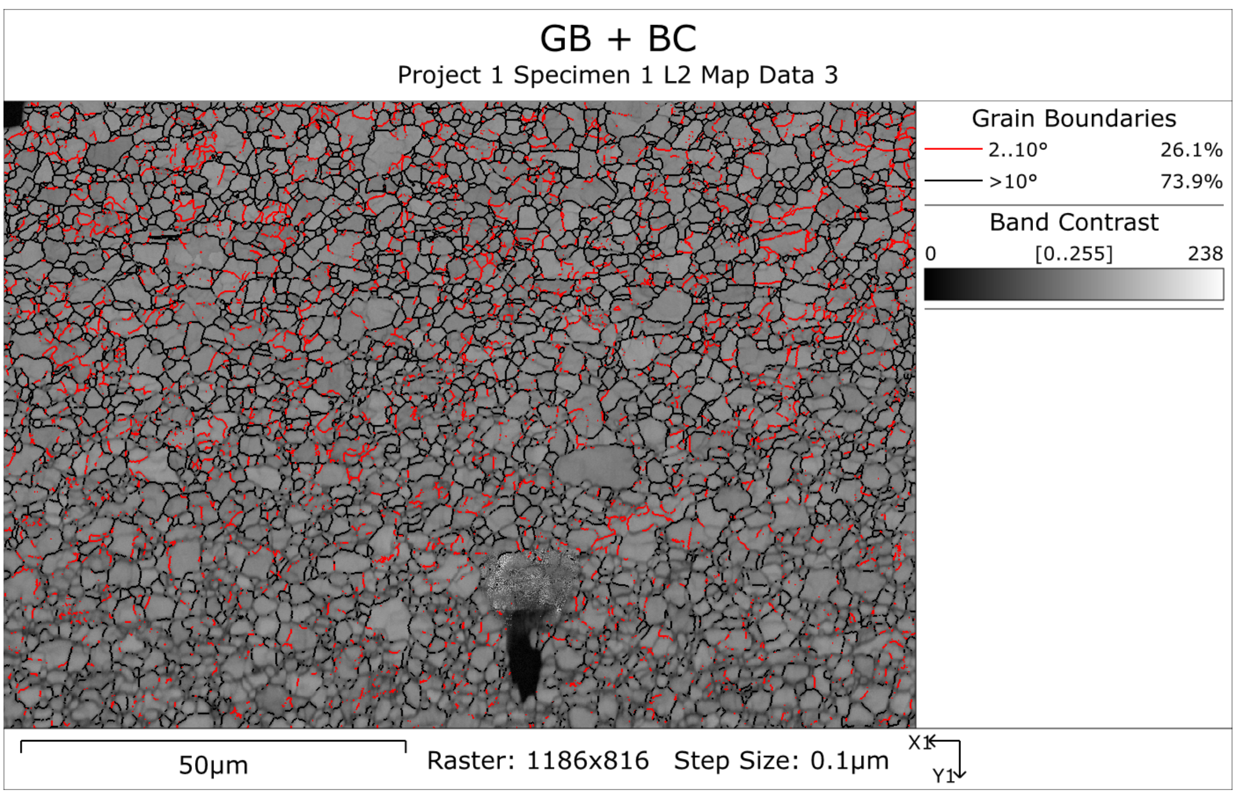

Figure 7.18. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C09, Sample SS18

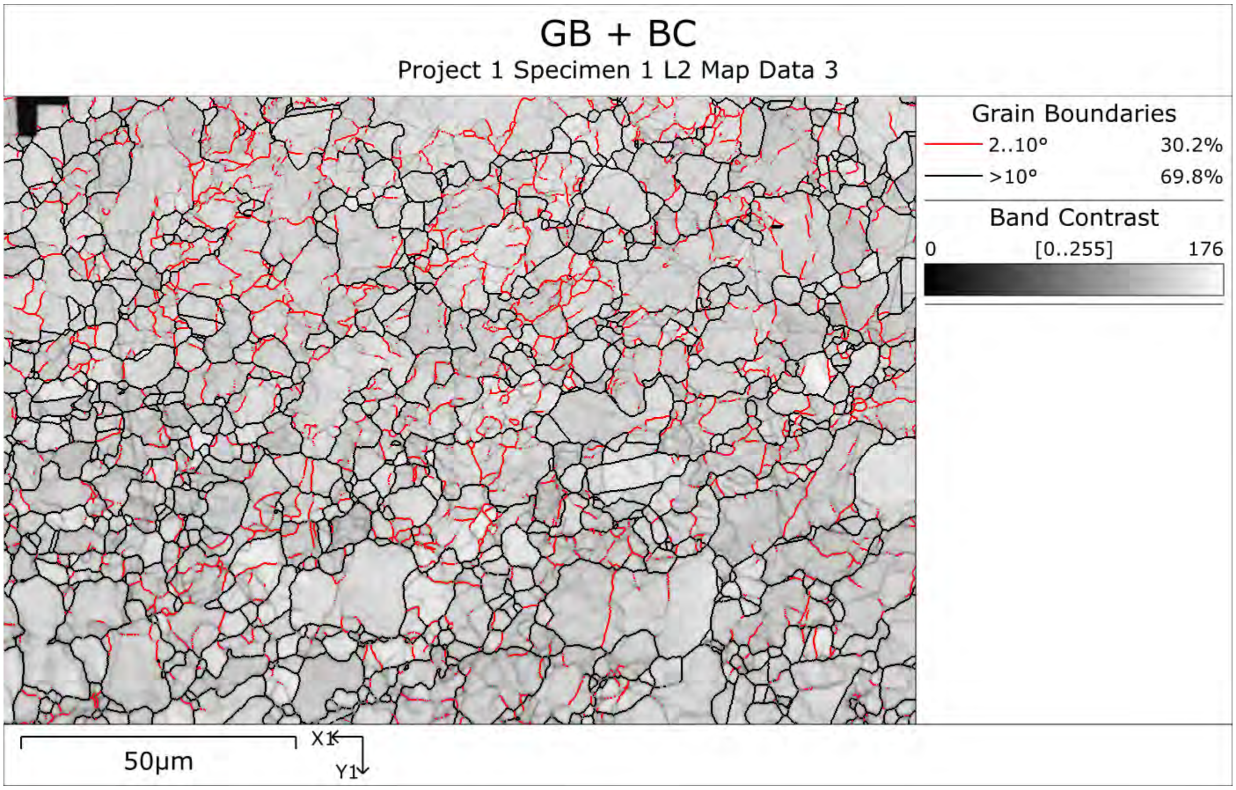

Figure 7.19. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C10, Sample SS19

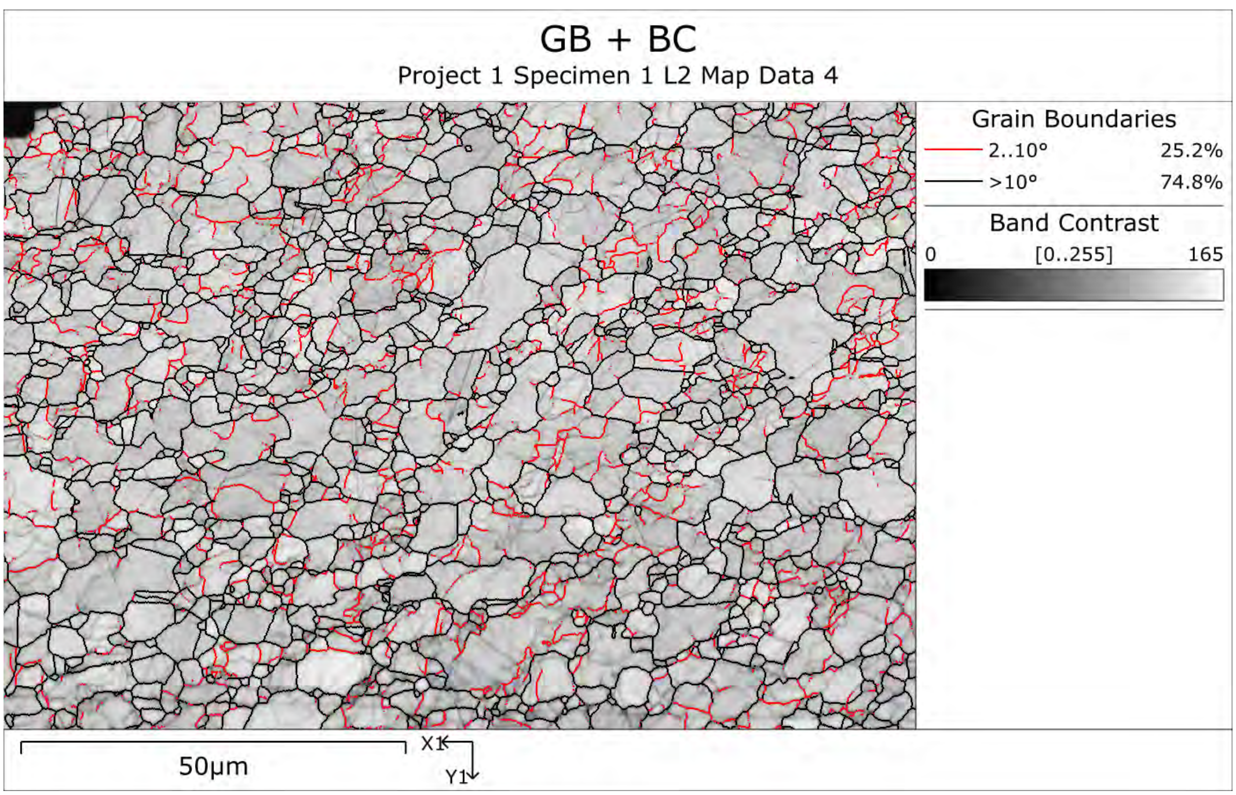

Figure 7.20. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C11, Sample SS20

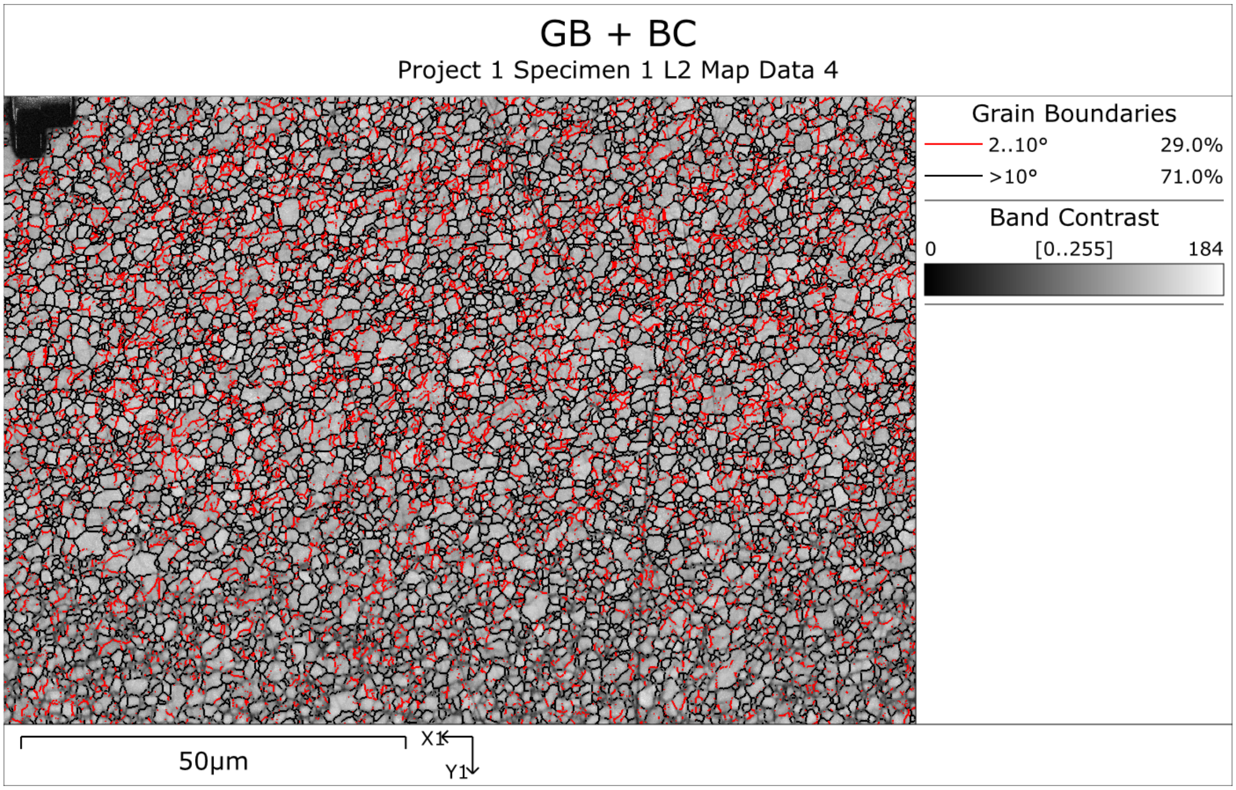

Figure 7.21. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C06, Sample SS21

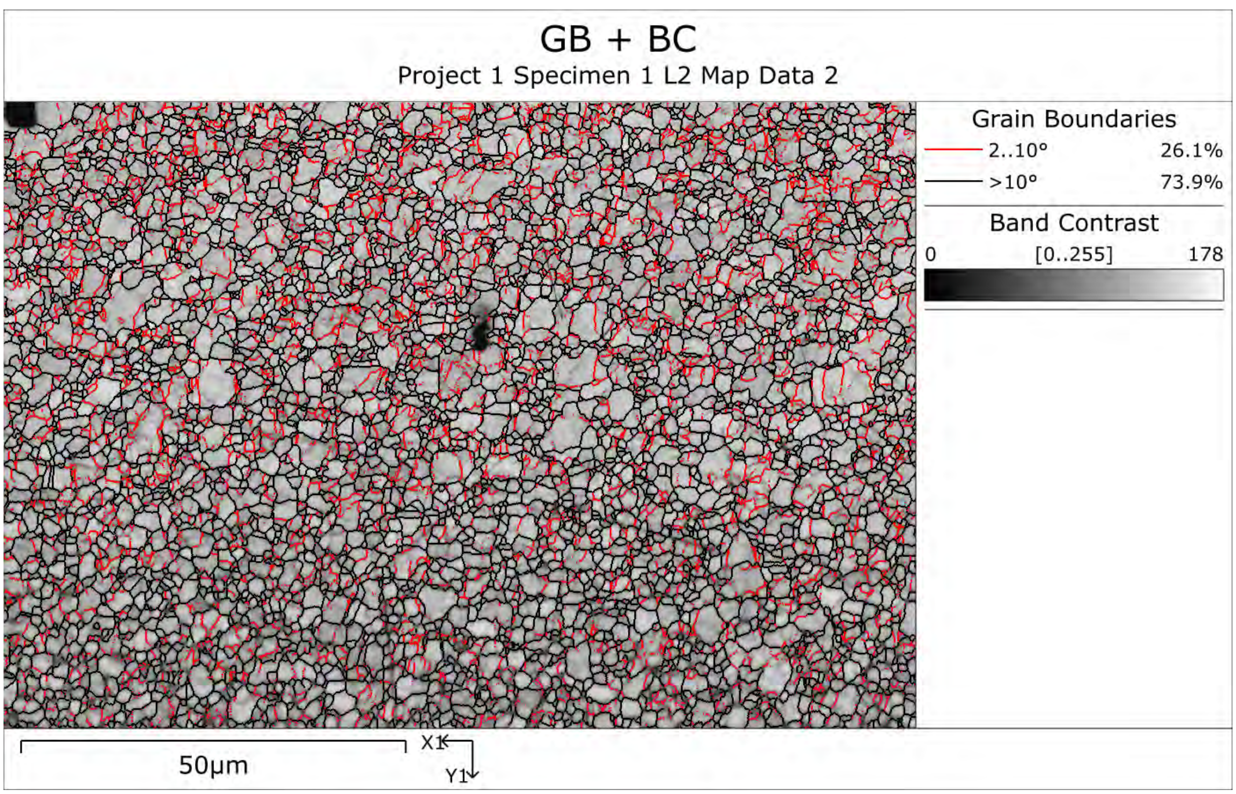

Figure 7.22. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C07, Sample SS22

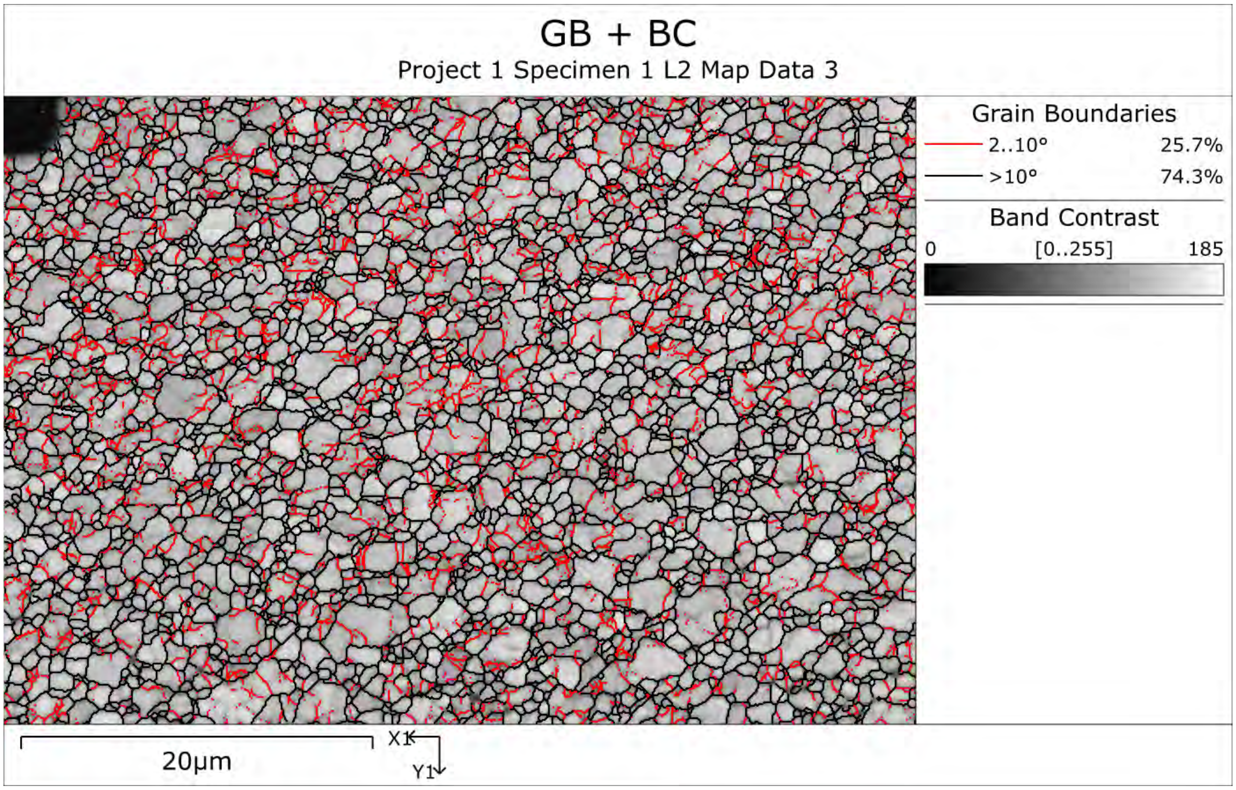

Figure 7.23. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C03, Sample SS23

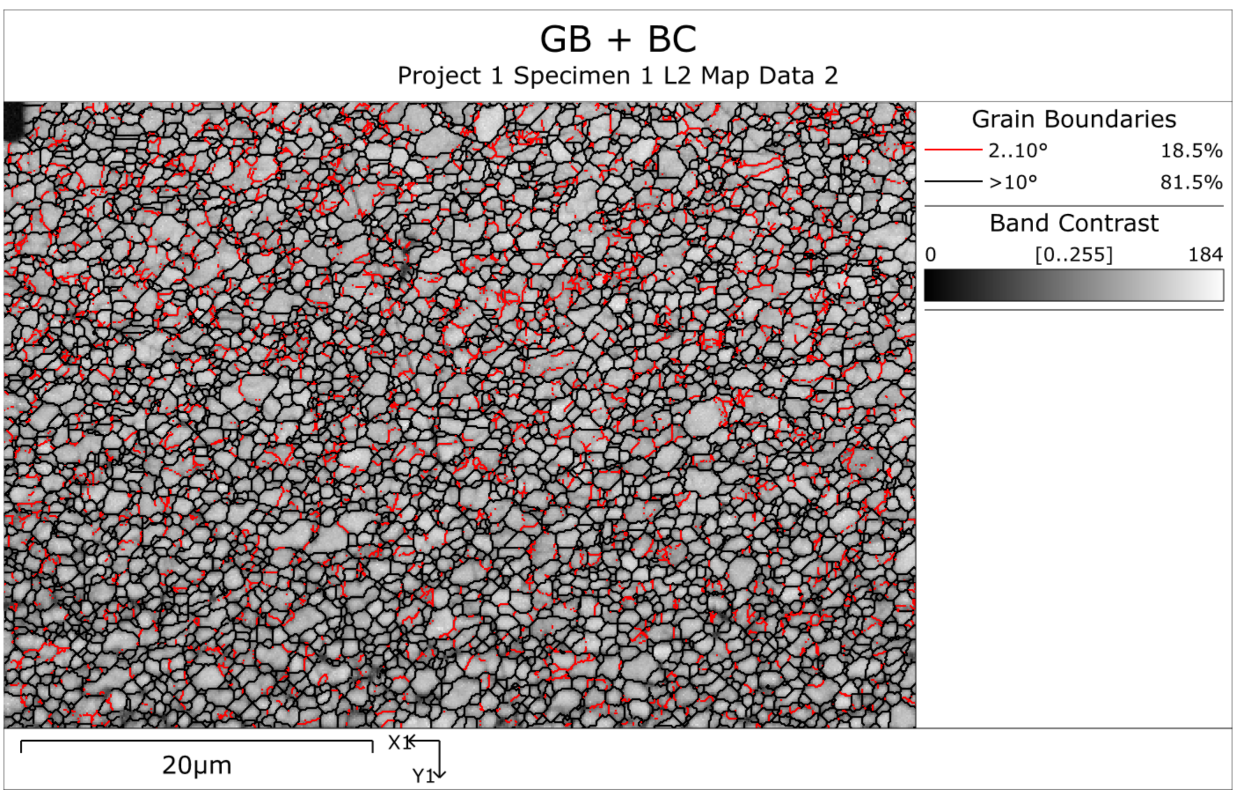

Figure 7.24. Low & High Angle Grain Boundaries + Band Contrast for L02: Condition C01, Sample SS34

7.4 Inverse Pole Function - X direction

The following figures show the IPF map in the x-direction with grain boundaries identified through segmentation.

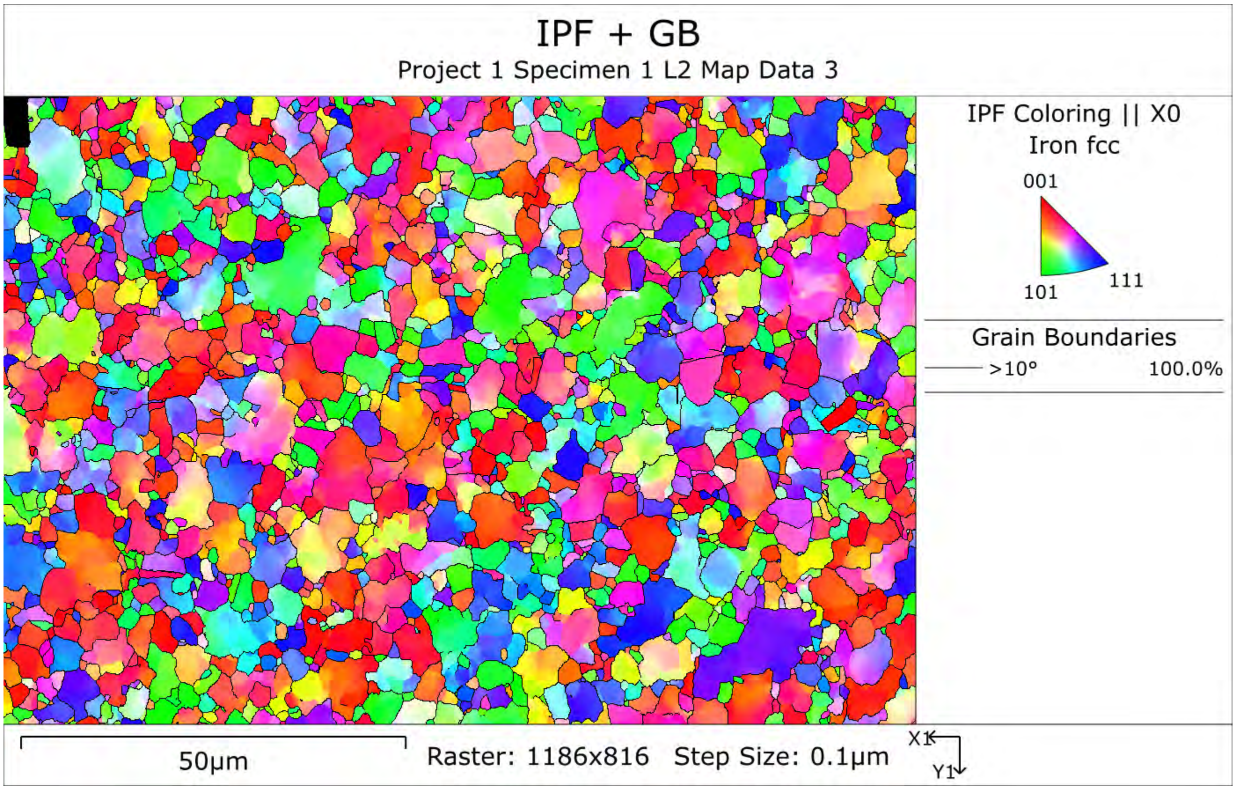

Figure 7.25. Inverse Pole Function - X Images for L02: Condition C08, Sample SS17

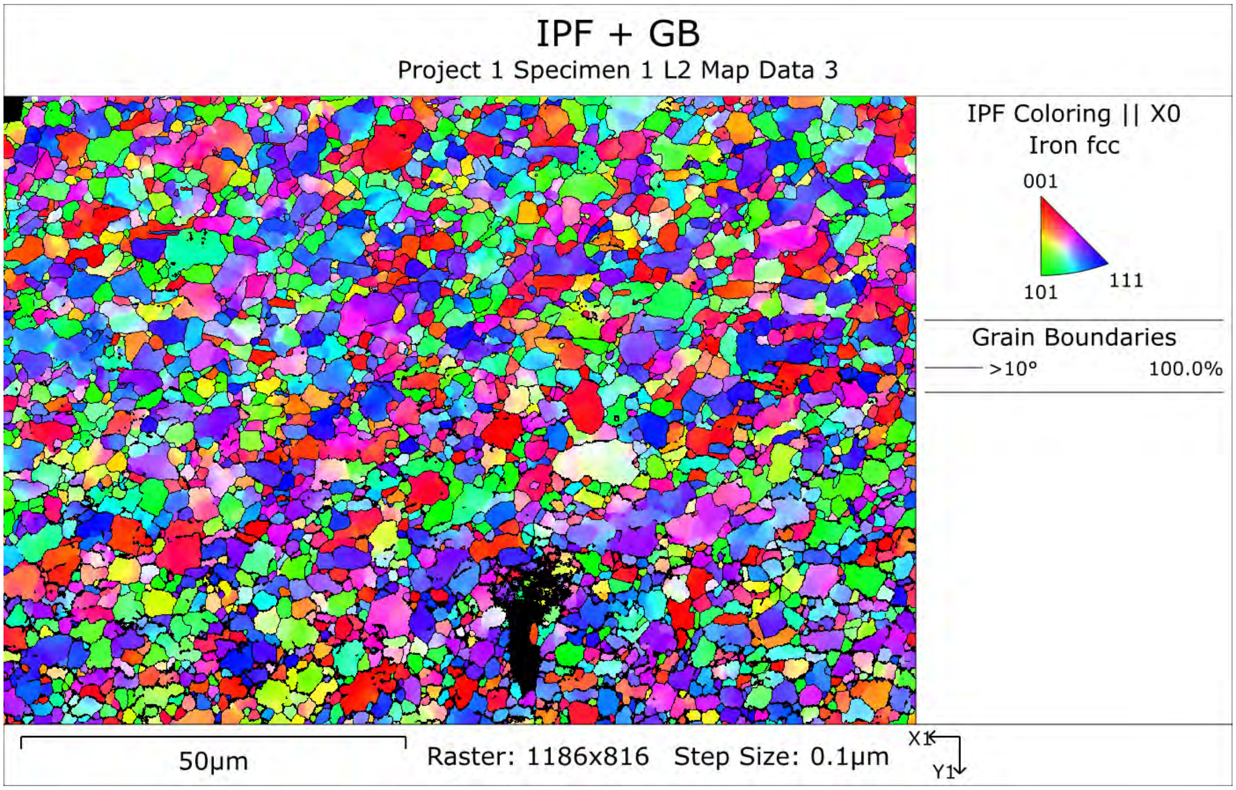

Figure 7.26. Inverse Pole Function - X Images for L02: Condition C09, Sample SS18

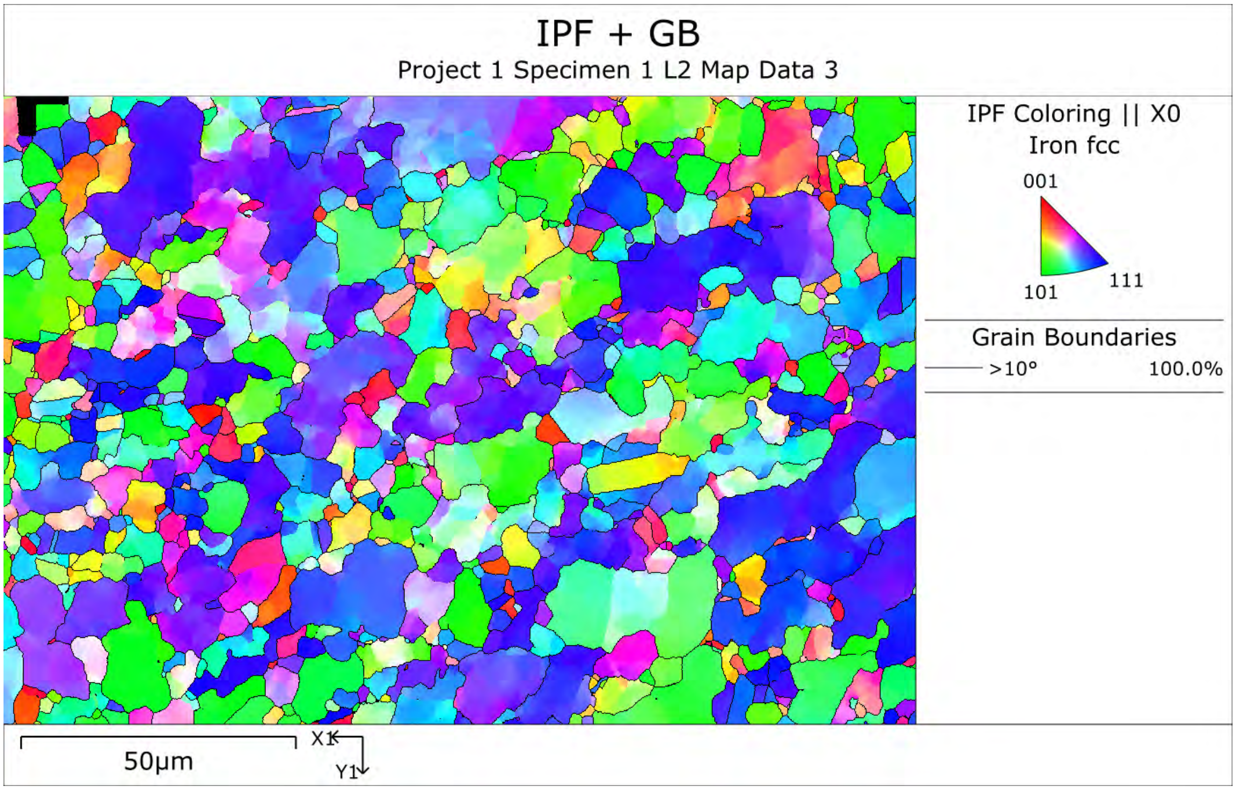

Figure 7.27. Inverse Pole Function - X Images for L02: Condition C10, Sample SS19

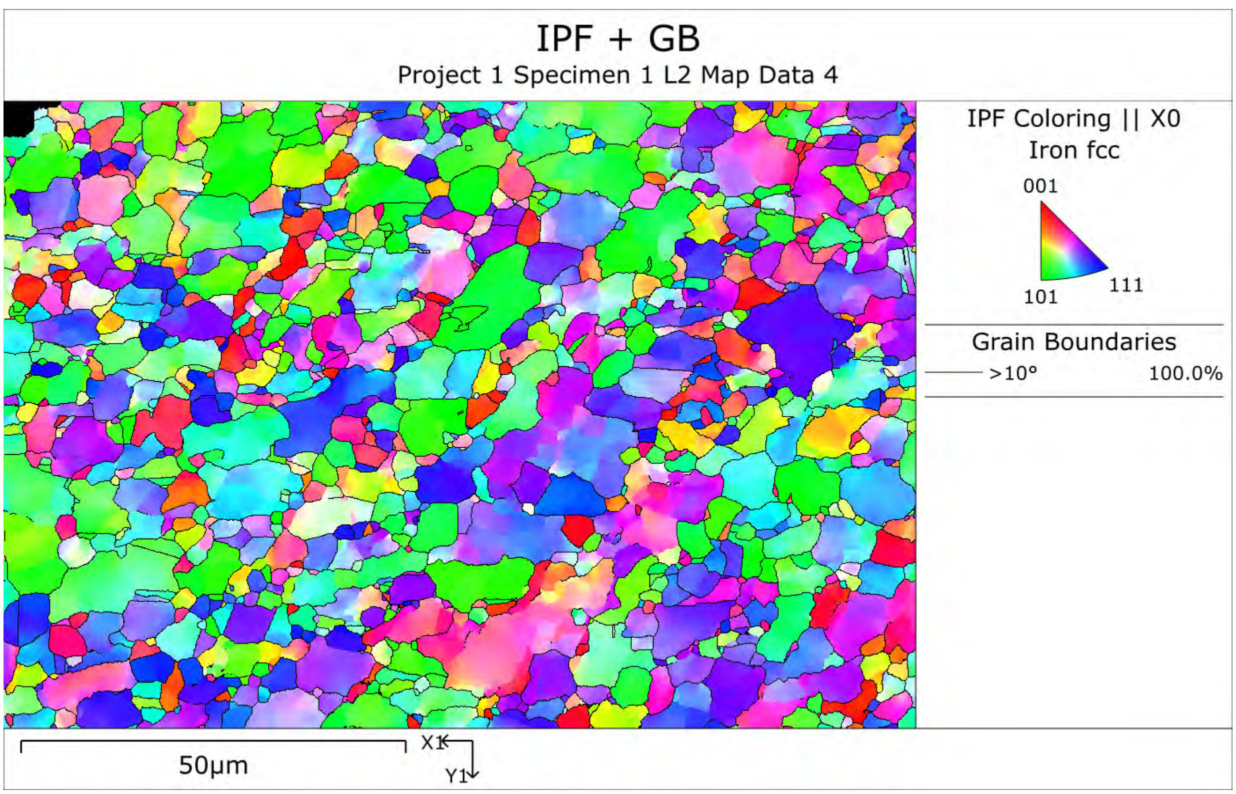

Figure 7.28. Inverse Pole Function - X Images for L02: Condition C11, Sample SS20

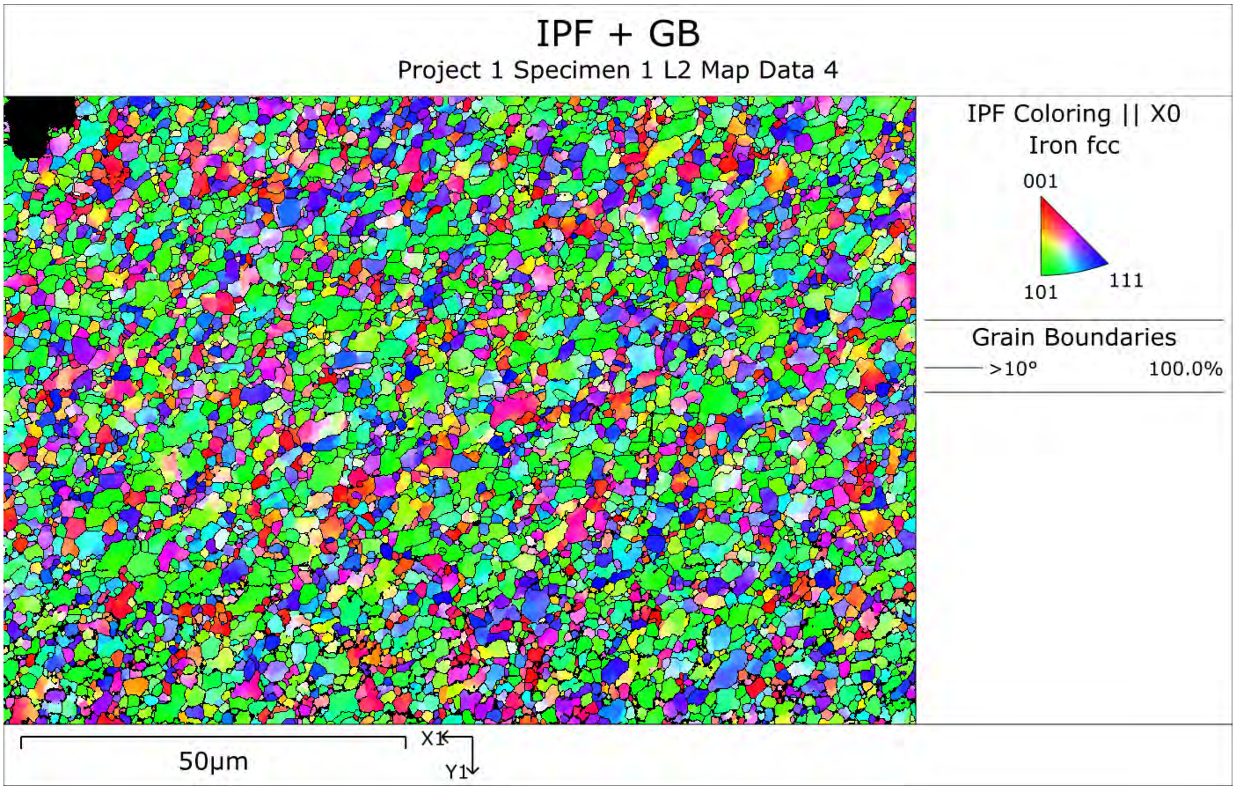

Figure 7.29. Inverse Pole Function - X Images for L02: Condition C06, Sample SS21

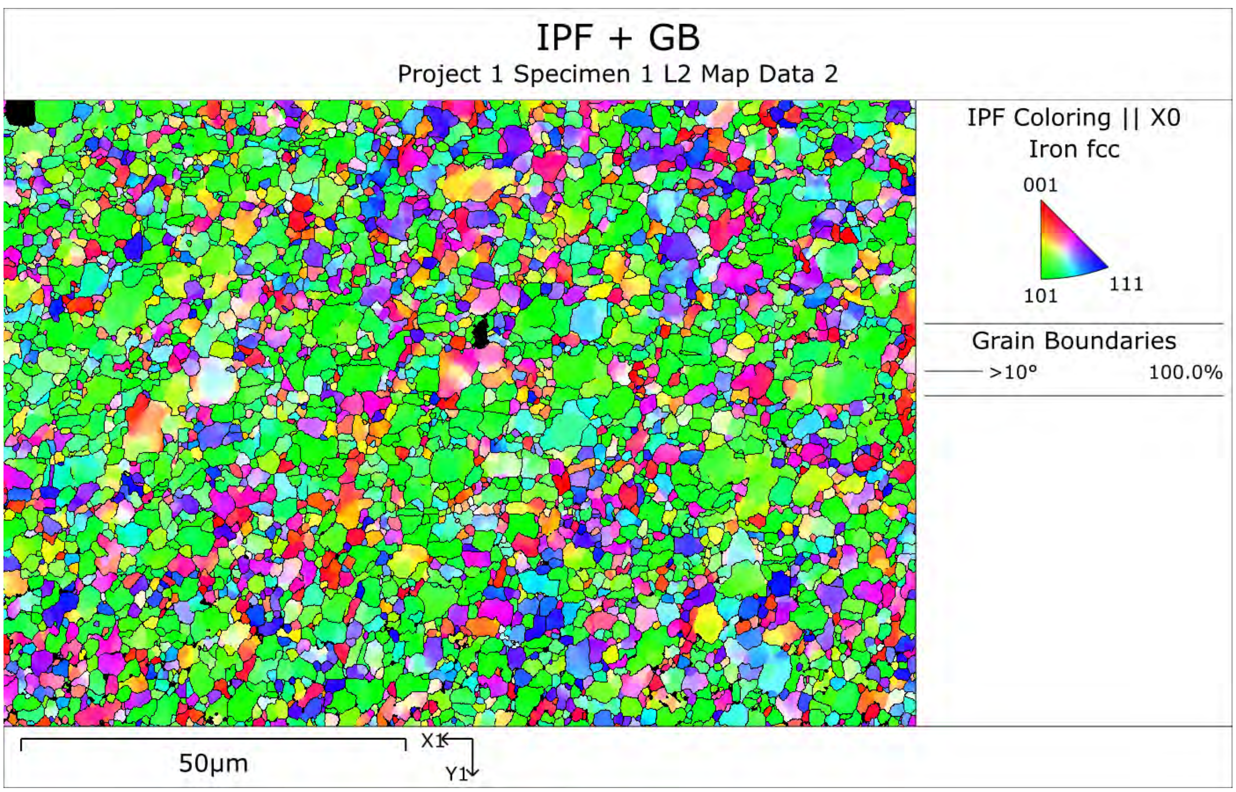

Figure 7.30. Inverse Pole Function - X Images for L02: Condition C07, Sample SS22

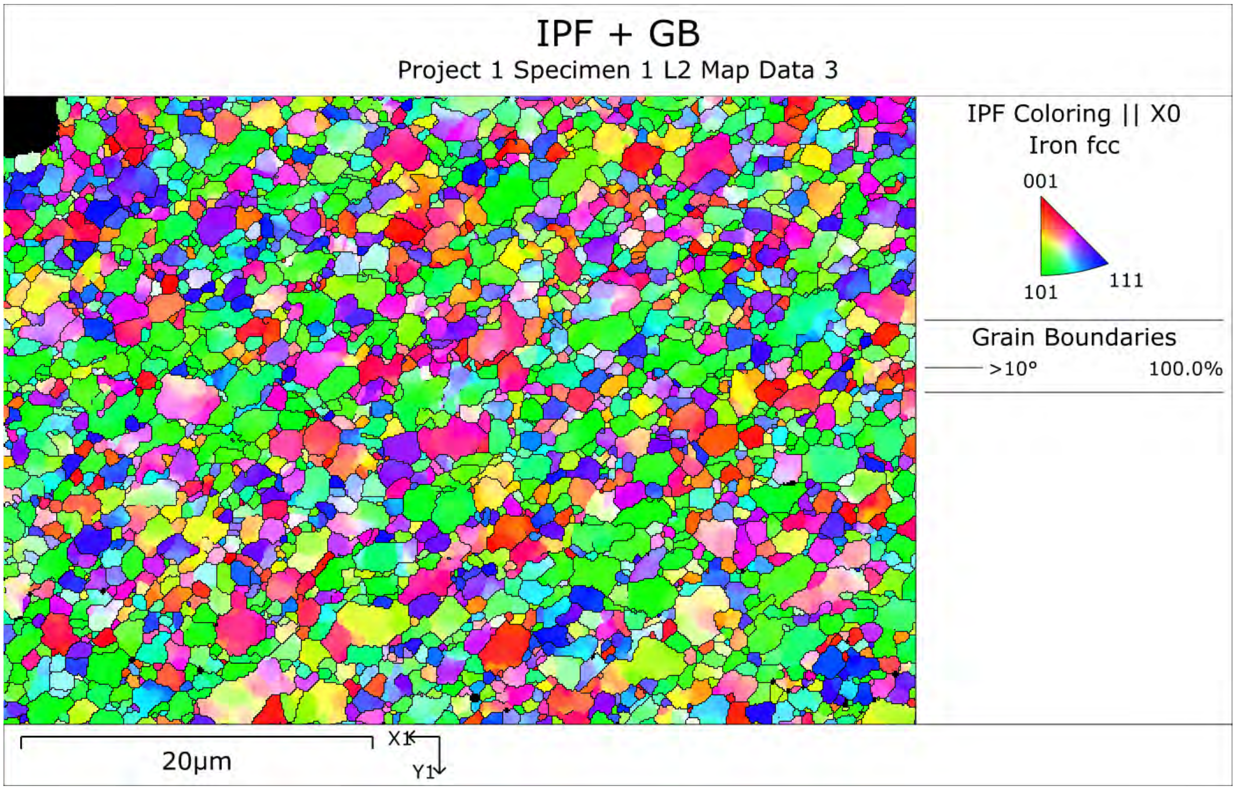

Figure 7.31. Inverse Pole Function - X Images for L02: Condition C03, Sample SS23

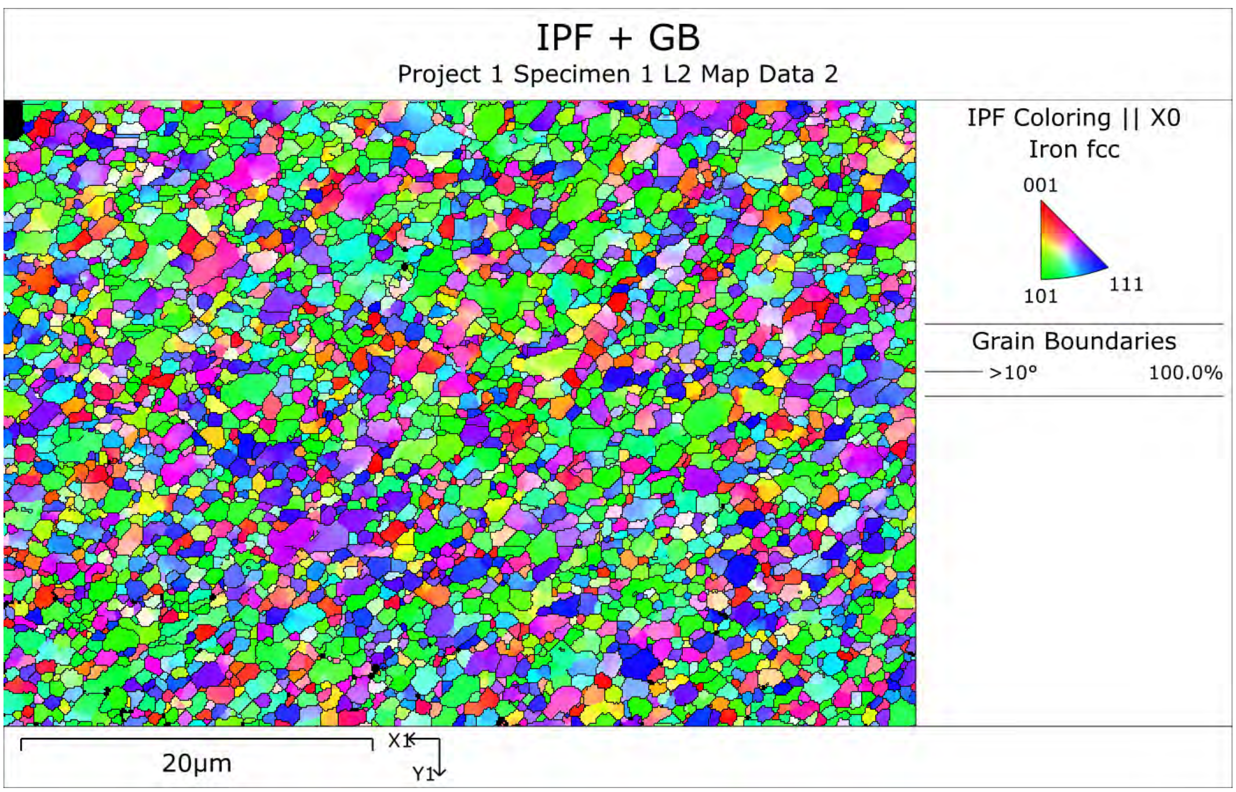

Figure 7.32. Inverse Pole Function - X Images for L02: Condition C01, Sample SS34

7.5 Inverse Pole Function - Y direction

The following figures show the IPF map in the y-direction with grain boundaries identified through segmentation.

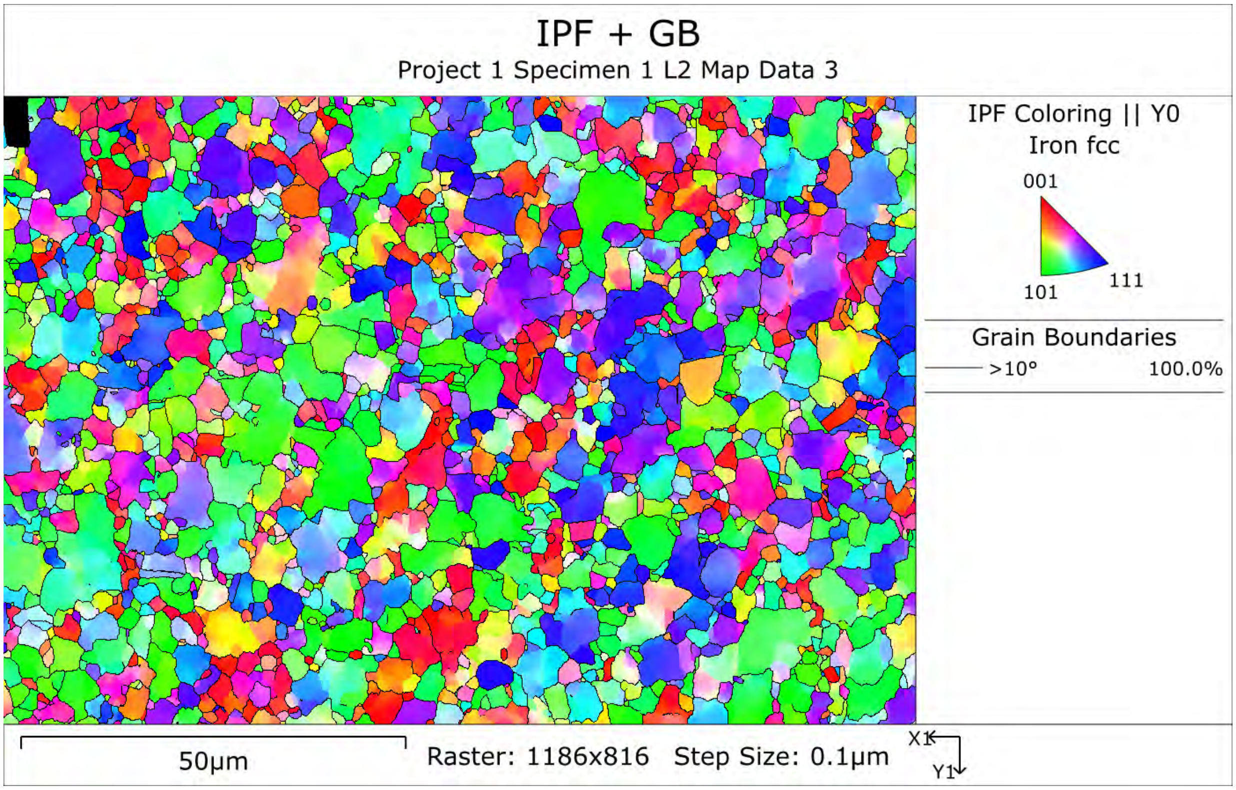

Figure 7.33. Inverse Pole Function - Y Images for L02: Condition C08, Sample SS17

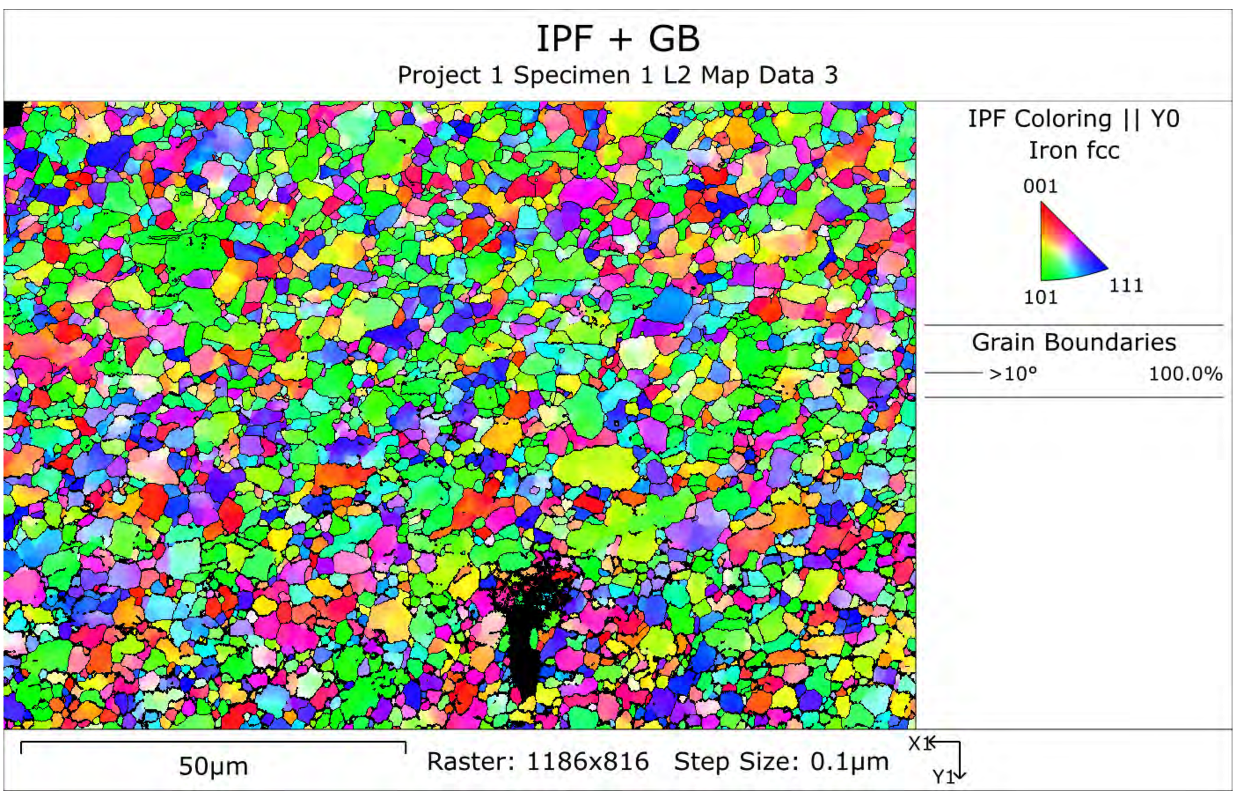

Figure 7.34. Inverse Pole Function - Y Images for L02: Condition C09, Sample SS18

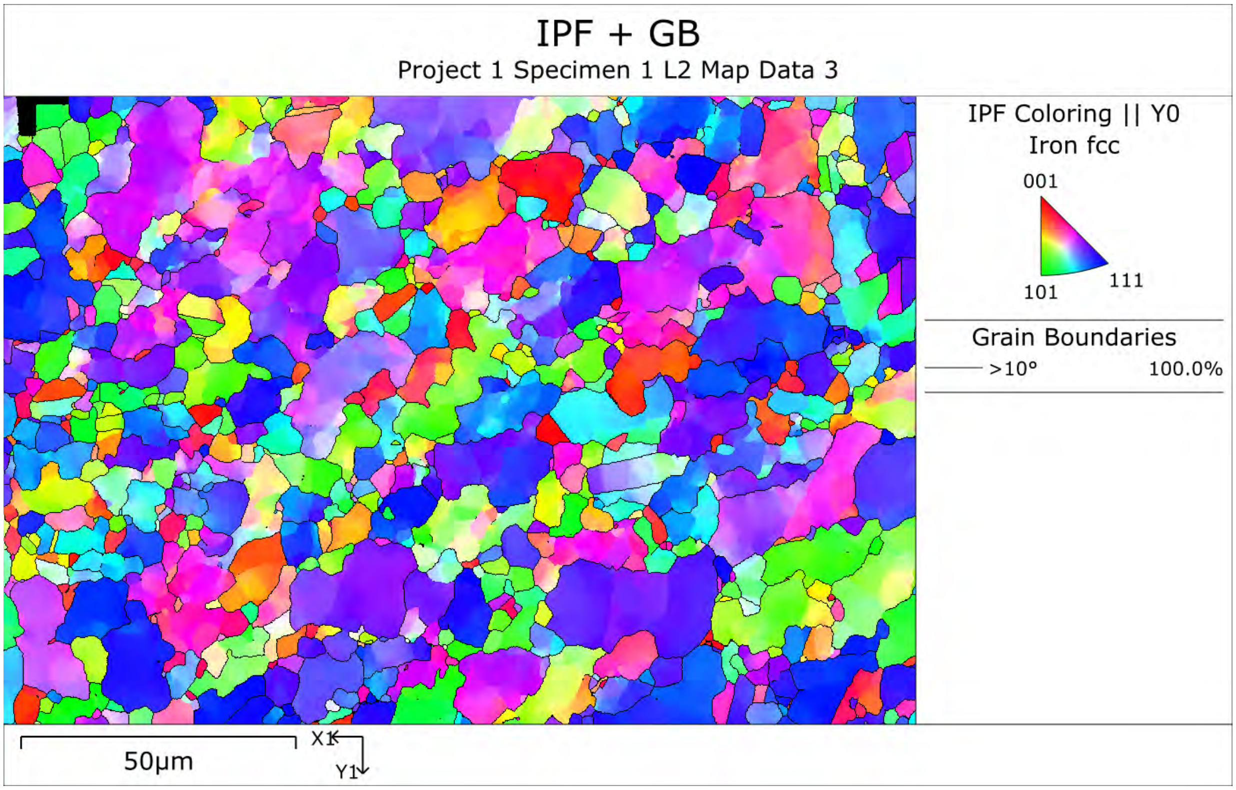

Figure 7.35. Inverse Pole Function - Y Images for L02: Condition C10, Sample SS19

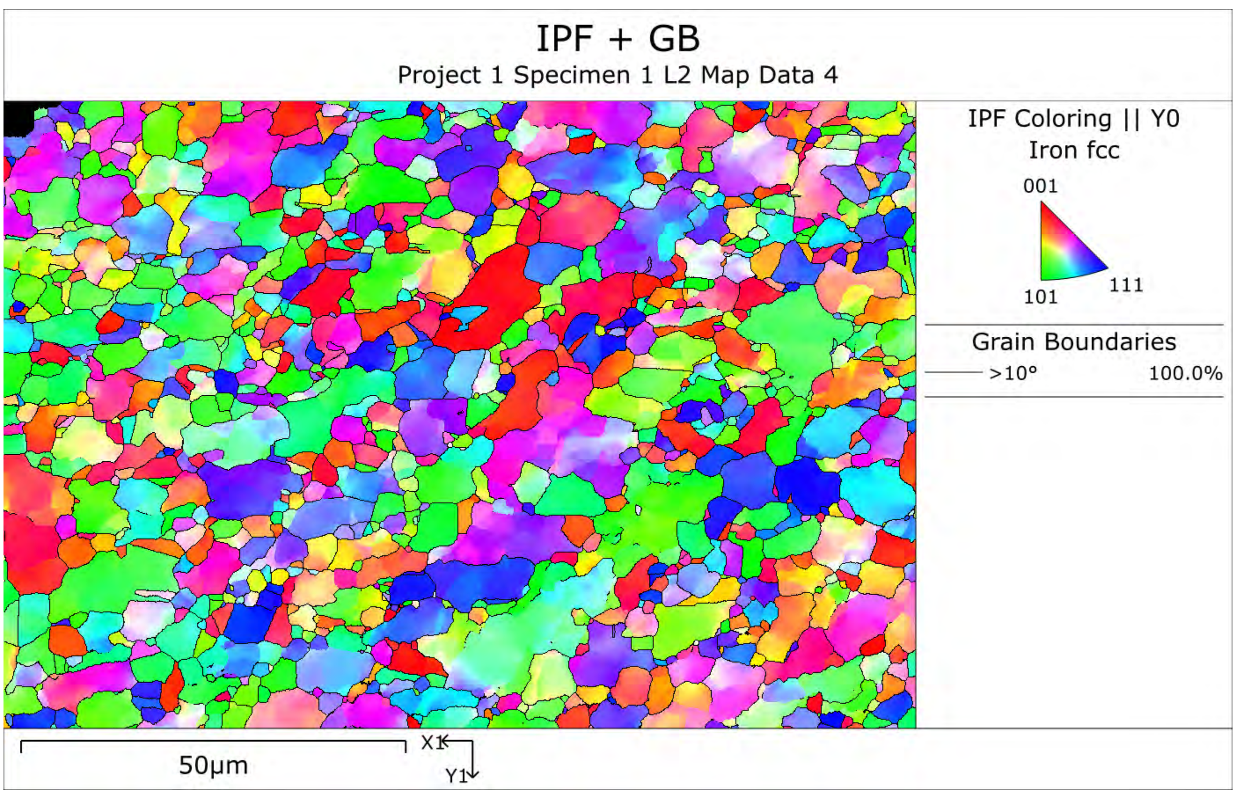

Figure 7.36. Inverse Pole Function - Y Images for L02: Condition C11, Sample SS20

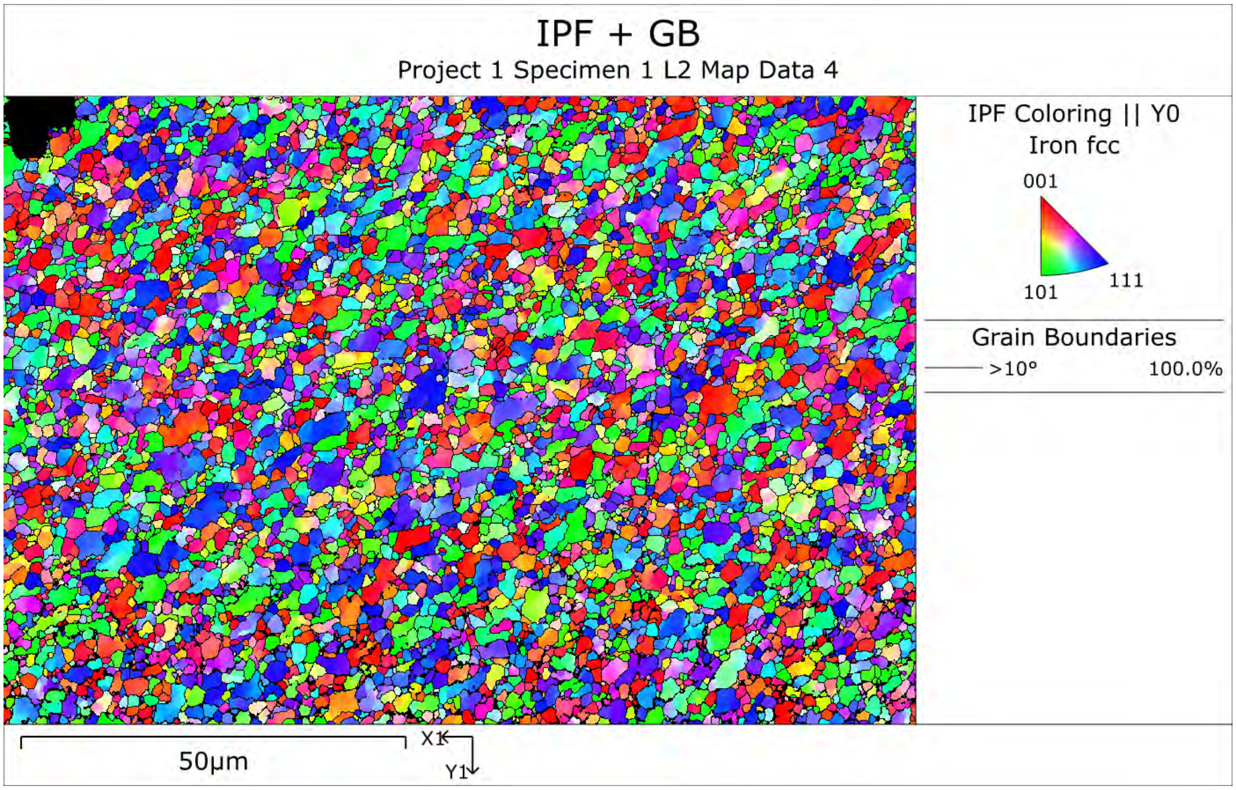

Figure 7.37. Inverse Pole Function - Y Images for L02: Condition C06, Sample SS21

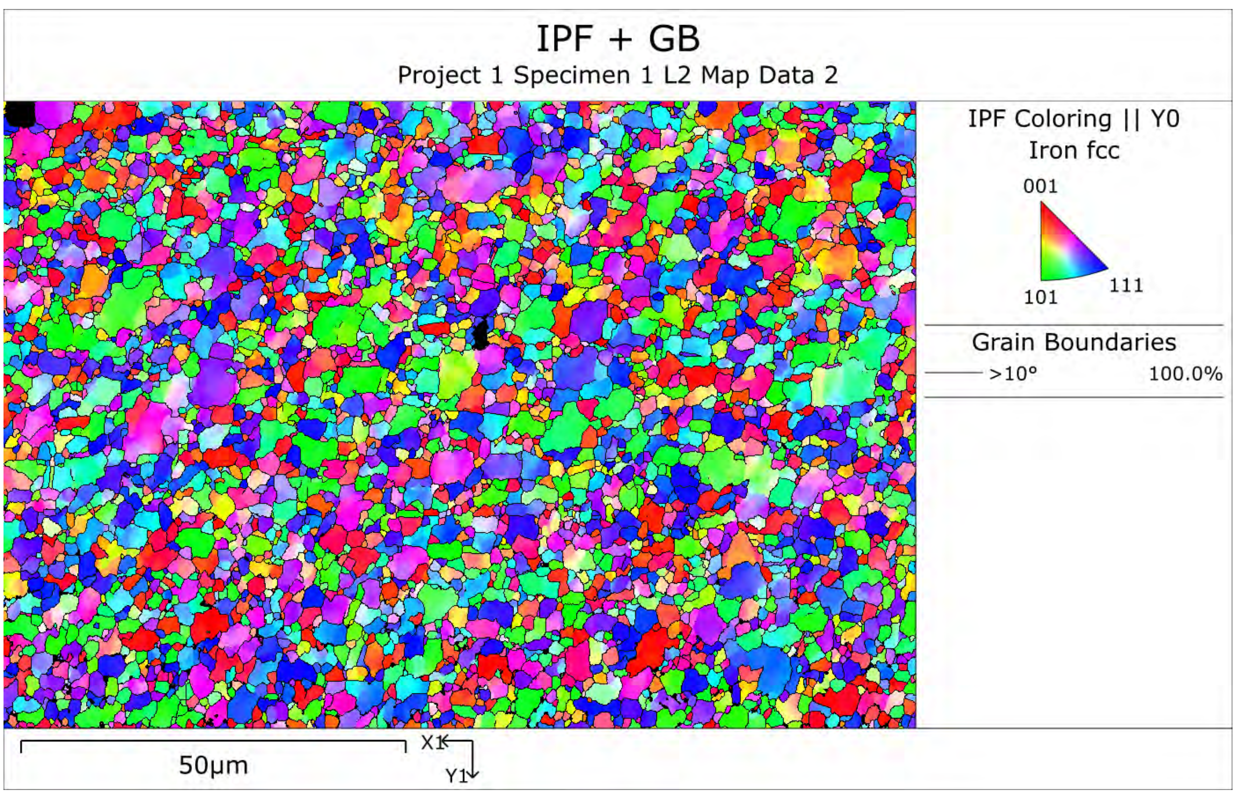

Figure 7.38. Inverse Pole Function - Y Images for L02: Condition C07, Sample SS22

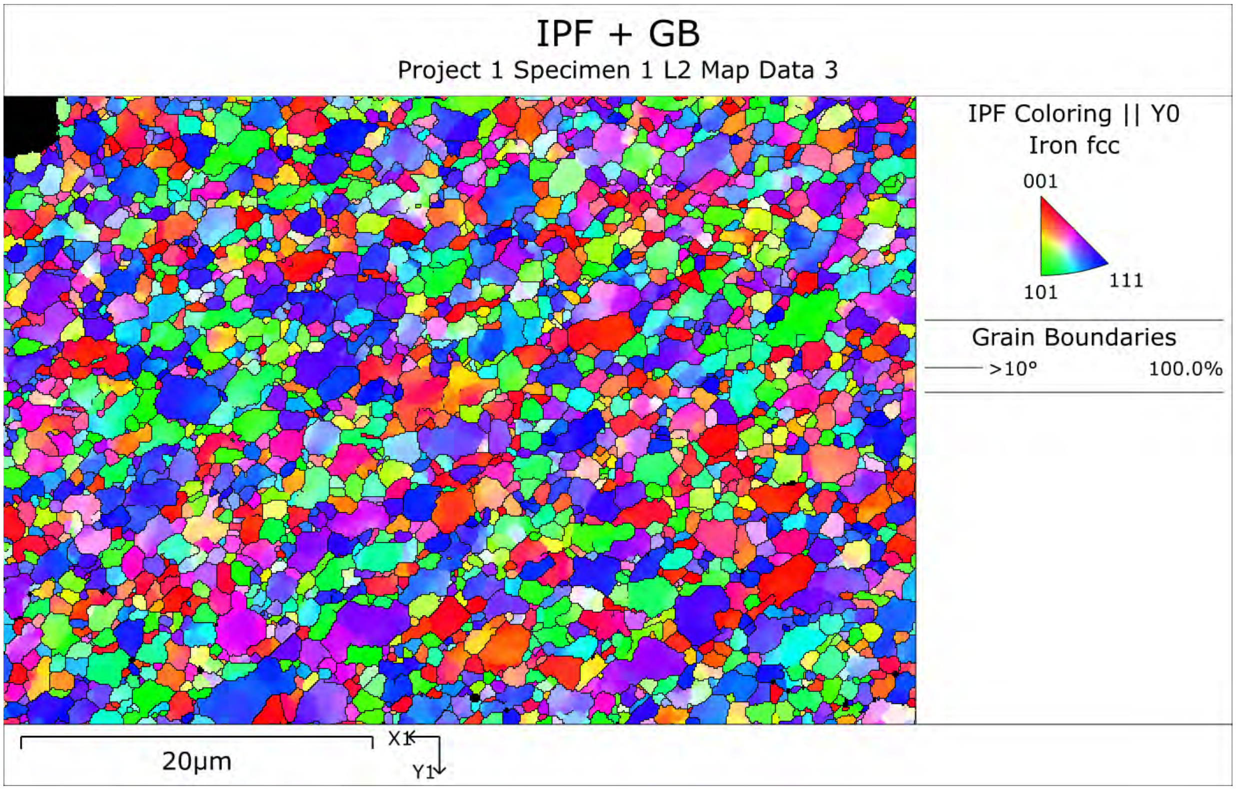

Figure 7.39. Inverse Pole Function - Y Images for L02: Condition C03, Sample SS23

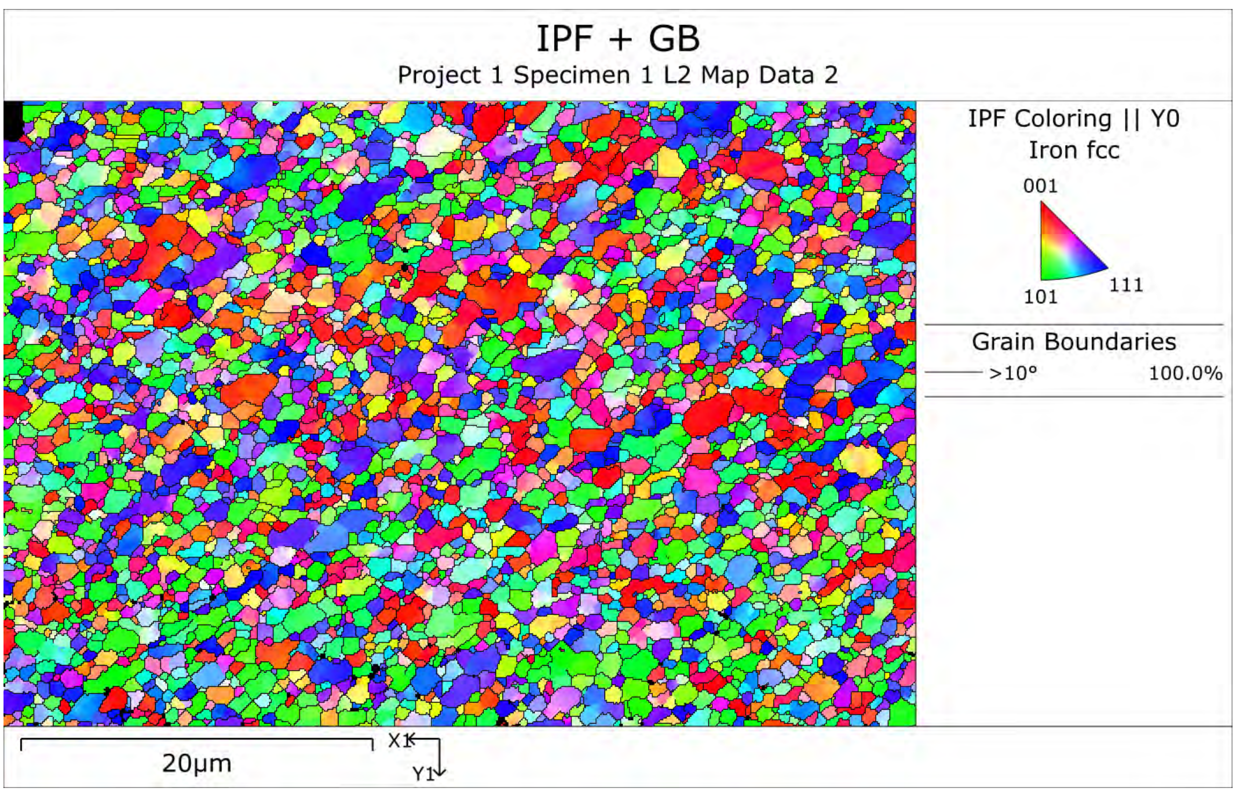

Figure 7.40. Inverse Pole Function - Y Images for L02: Condition C01, Sample SS34

7.6 Inverse Pole Function - Z direction

The following figures show the IPF map in the z-direction with grain boundaries identified through segmentation.

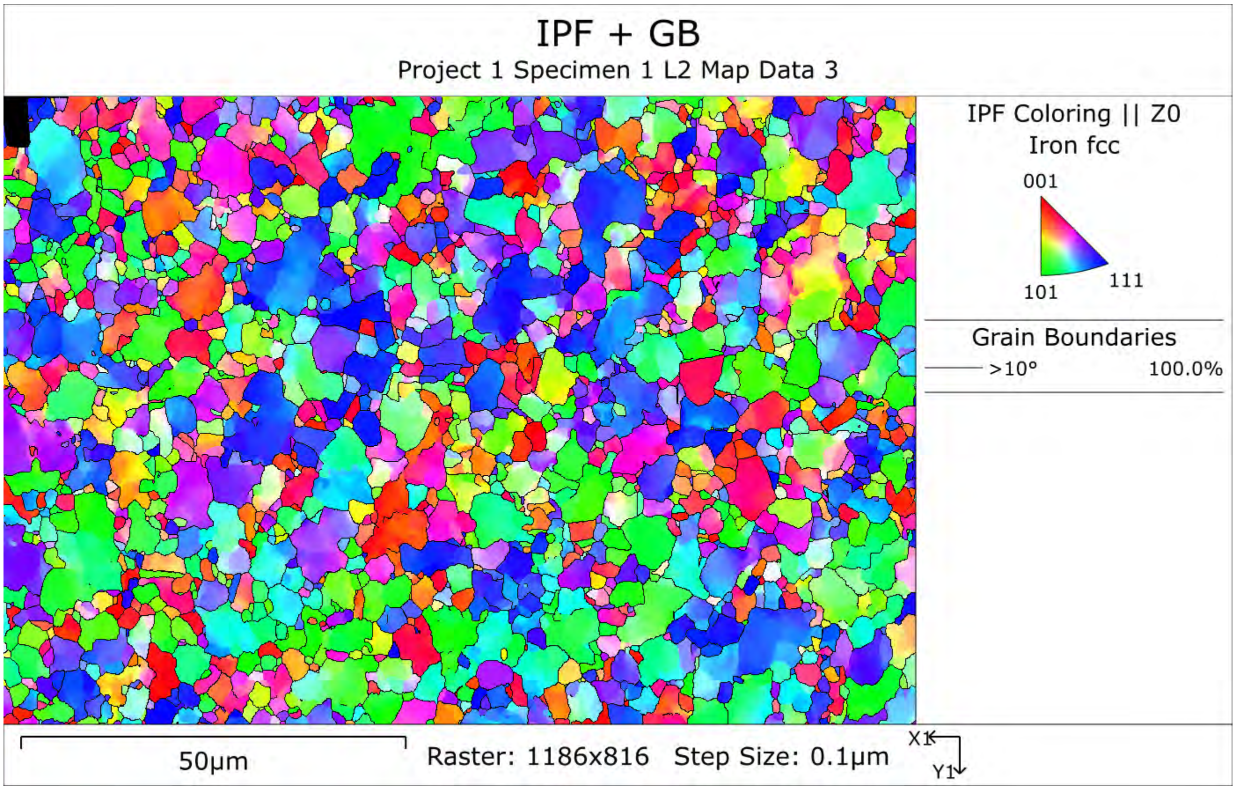

Figure 7.41. Inverse Pole Function - Z Images for L02: Condition C08, Sample SS17

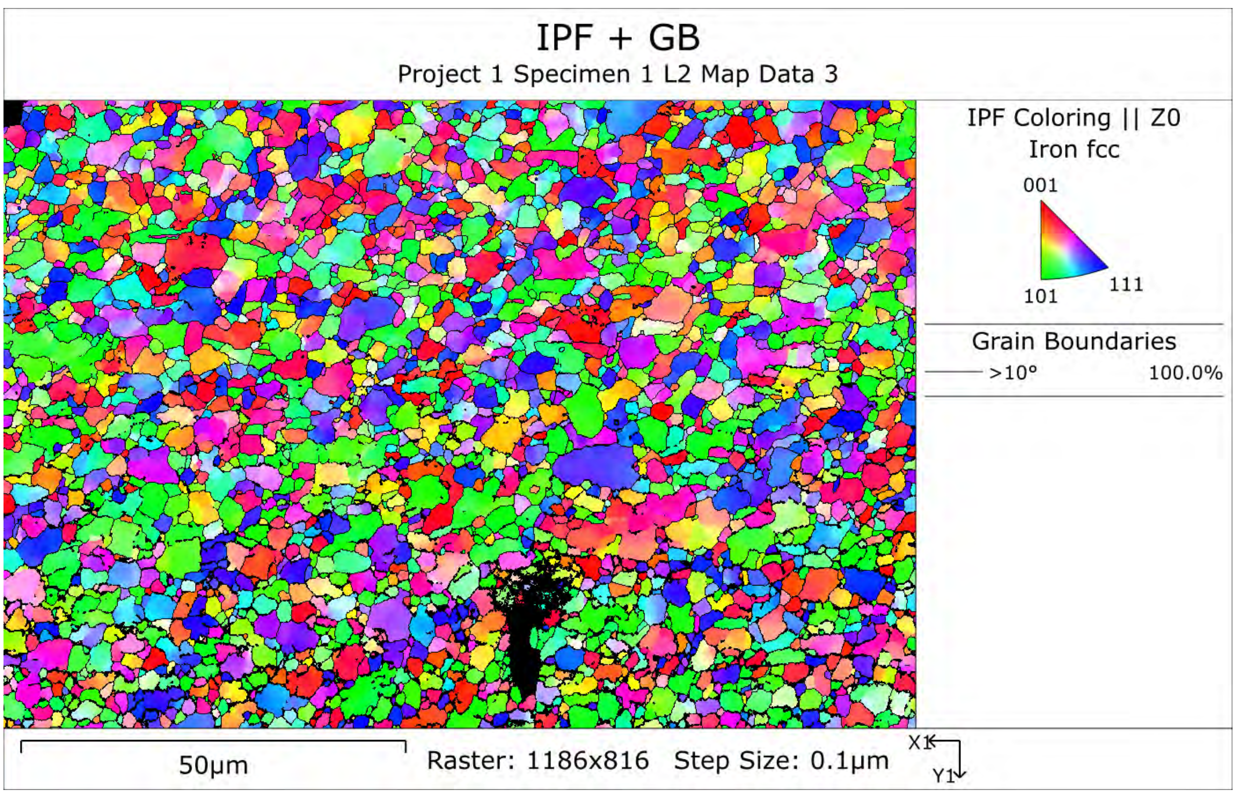

Figure 7.42. Inverse Pole Function - Z Images for L02: Condition C09, Sample SS18

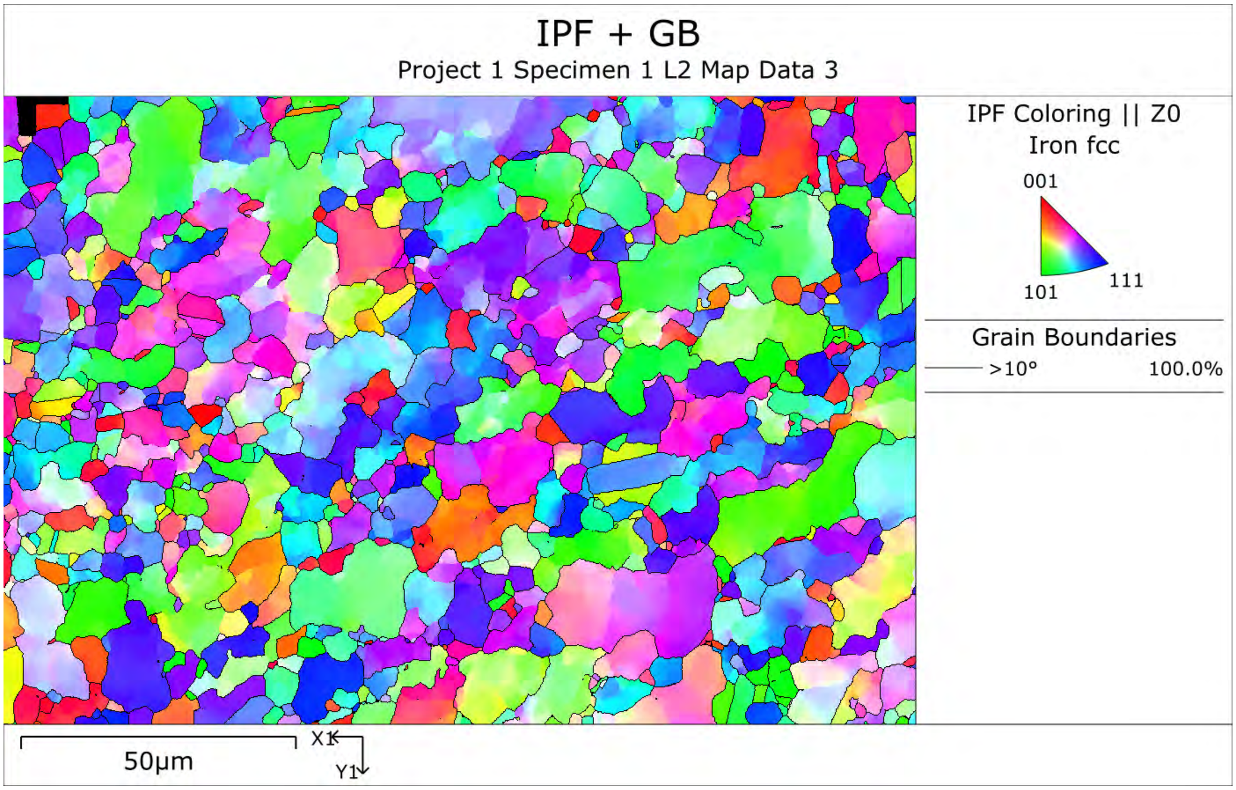

Figure 7.43. Inverse Pole Function - Z Images for L02: Condition C10, Sample SS19

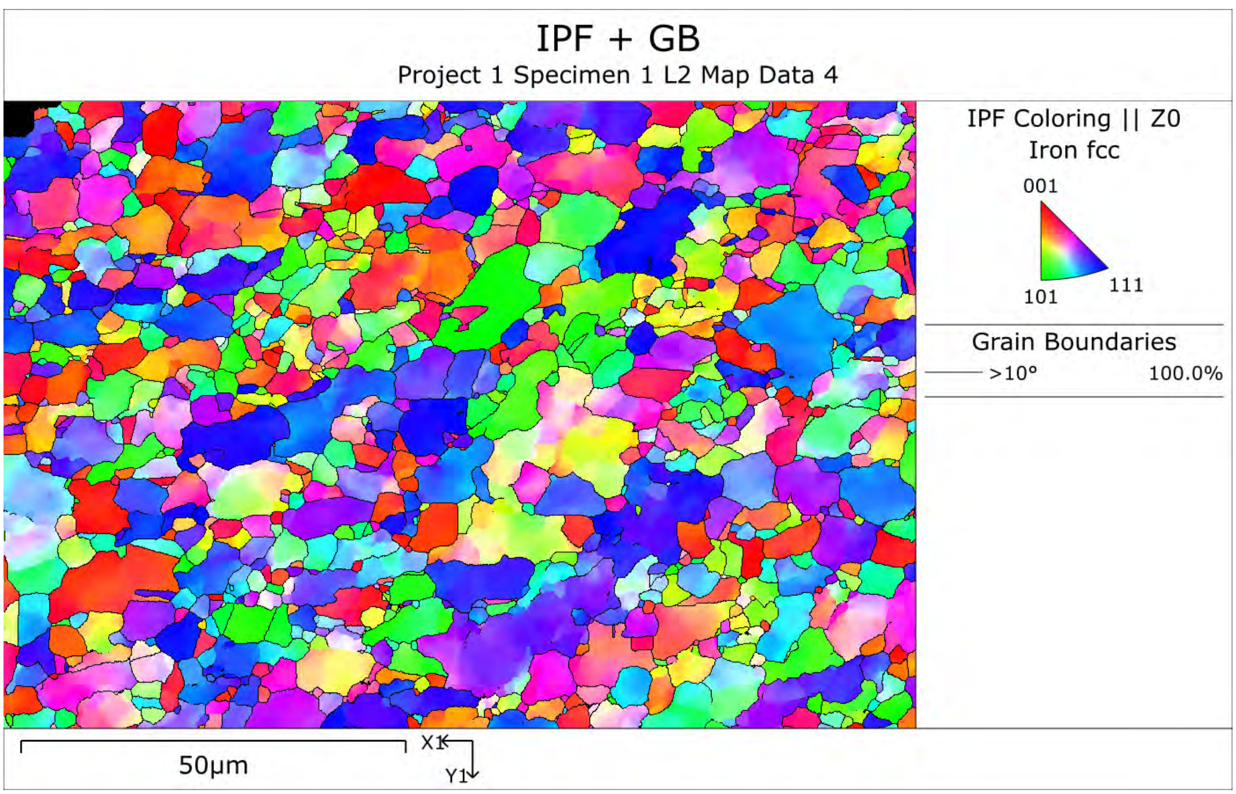

Figure 7.44. Inverse Pole Function - Z Images for L02: Condition C11, Sample SS20

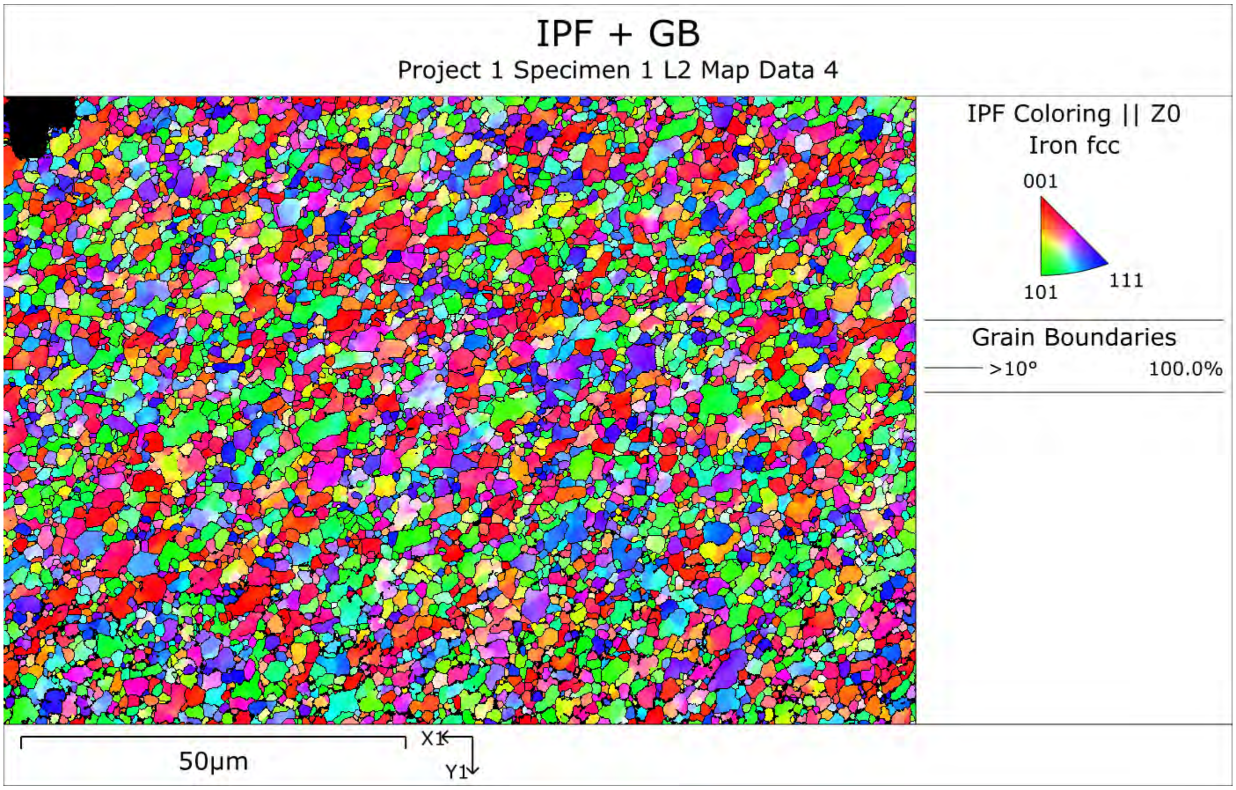

Figure 7.45. Inverse Pole Function - Z Images for L02: Condition C06, Sample SS21

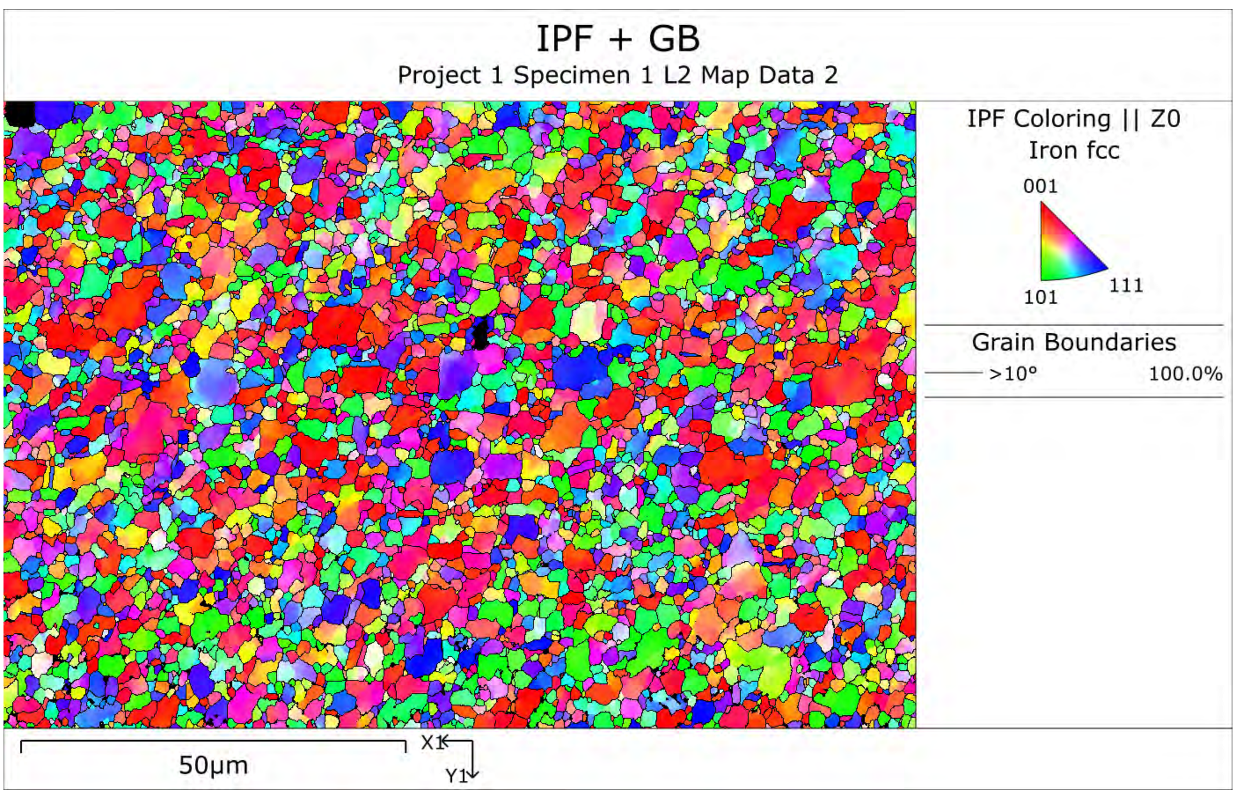

Figure 7.46. Inverse Pole Function - Z Images for L02: Condition C07, Sample SS22

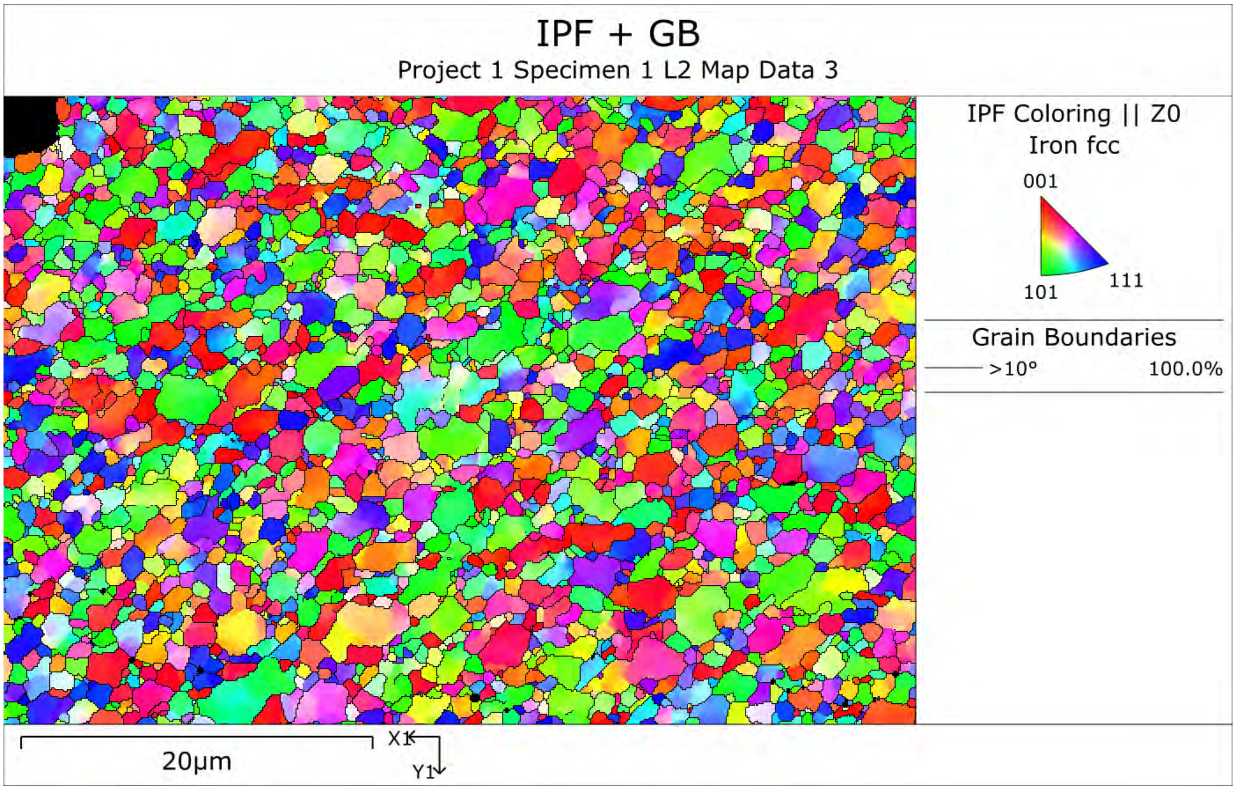

Figure 7.47. Inverse Pole Function - Z Images for L02: Condition C03, Sample SS23

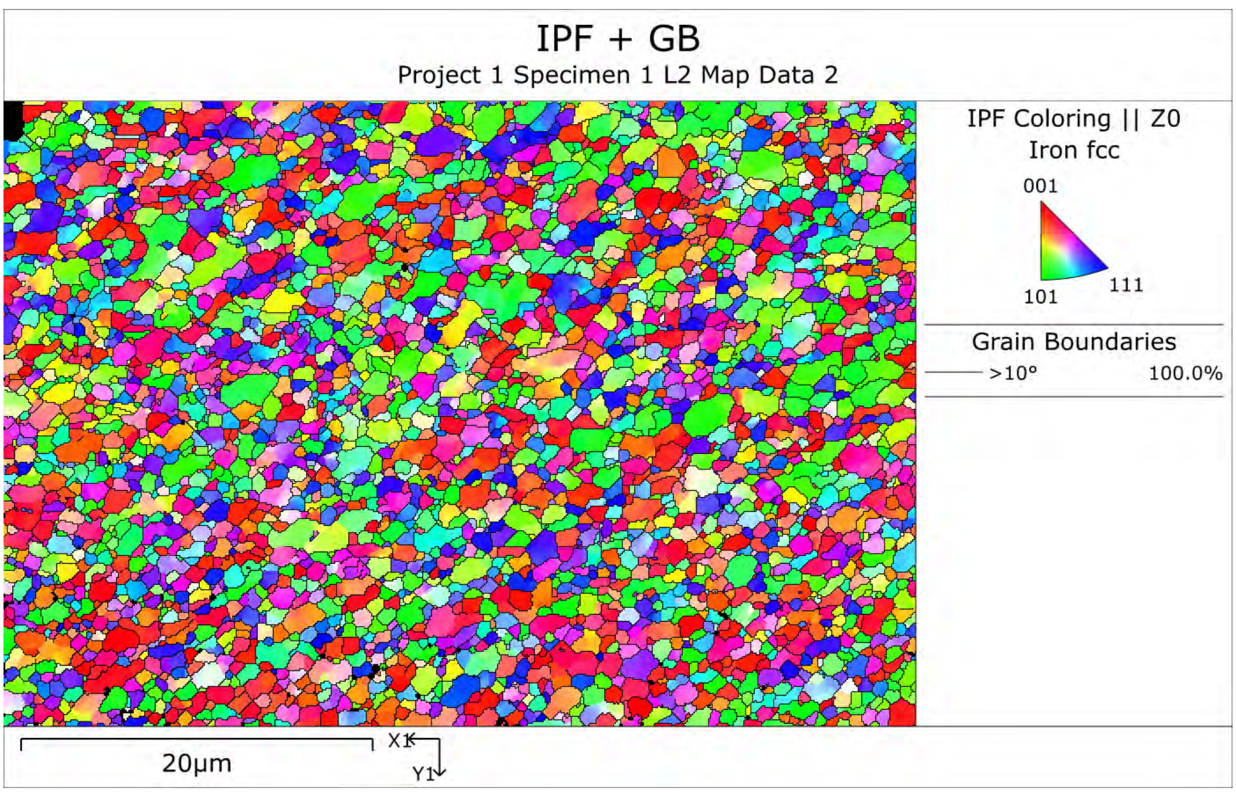

Figure 7.48. Inverse Pole Function - Z Images for L02: Condition C01, Sample SS34

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902 Battelle Boulevard
P.O. Box 999
Richland, WA 99354

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Attachment B: MCPC Round 3 Material Characterization Results for Modality HARDNESS

A Ortiz A Guzman
DR Todd O Linsuain
K Nwe

Attachment B contains 18 pages

Acknowledgments

This research was supported by the Materials Characterization, Prediction, and Control (MCPC) investment, under the Laboratory Directed Research and Development (LDRD) Program at Pacific Northwest National Laboratory (PNNL). PNNL is a multi-program national laboratory operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute under Contract No. DE-AC05-76RL01830.

Contents

Figures

Tables

1.0 Introduction

In hardness testing (also called ”micro” hardness testing for the dimensional-scale of the method), an indentation is made on the specimen by a diamond indenter through the application of a load. The size of the resultant indentation is measured with the help of a calibrated optical microscope, and the hardness is evaluated as the mean stress applied underneath the indenter. Hardness testing introduces local plastic stresses and corresponding strains into the material and is generally considered a destructive test. See Section 5.2 in Glass et al. (2024) for more general information about hardness testing (the reference is listed in Section 7.0 of the main report).

The ”Vickers” testing method is applied here on mounted and polished specimens. Data is obtained on a square grid covering the entire stir region and the surrounding unprocessed base material. Results are provided numerically and via several visualizations for each specimen. Visualizations are graphed with a consistent scale across all specimens in this dateset to facilitate visual comparisons across the specimens.

The following sections provide a summary of results for this modality to enhance dissemination of the large volume of similar data that are made available via the PNNL DataHub (`https://data.pnnl.gov`) platform.

2.0 Key Information and Results

The main portion of this report provides a summary of key information related to the modality, including processing conditions, identification information, and the numerical results of most focus in the project. Where to find the information and results in the main report is described below (Section, Table, and Figure numbers listed below are found in the main report):

- Nominal process conditions for the FSP experiments are defined in Table 1 and Table 3 (data is in two different sorting orders).
- The Condition, Sample, and Specimen ID matrix is defined in Table 2 that is necessary to decode the nominal conditions applied to a particular specimen.
- Details about grid spacing for hardness sample collection is defined in Section 4.0.
- Tabulation of the hardness results considered most representative per Sample ID is listed in Table 3 with discussion of what the particular result means discussed earlier in the section.
- Hardness value versus nominal processing temperature listed in Table 3 and Table 4 is displayed graphically in Figure 4.
- A classical means for evaluating the structure-property relationship is to compare hardness versus the inverse square root of mean grain diameter. This is shown in Figure 6 to highlight consistency of results across a range of conditions.

3.0 Instrumentation

The following instrument was utilized in testing of specimens described in this attachment. Specifications are provided below.

Hardness Tester Model CM-700AT

- Company Name: Sun-Tec Corporation
- Clark
- MOD: CM ARS9000
- SER#: CM908123

4.0 Results Files

Section 6.0 of the main report describes the structure of data for the modality. This includes general approaches for naming files, organizing data by each unique dataset, and collecting information from across multiple datasets of a modality in the ENSEMBLE_DATA directory. Below is a summary of the files produced for each unique dataset. Items after the first entry describe derived and interpreted data files.

- {Dataset name}.csv (DATA Directory): The key ”raw” text file produced by the tester for the specimen. hardness value is listed in column ”HV”. The (x, y) spacial location is listed in columns ”X” and ”Y” with the origin at (0, 0) arbitrarily chosen. Most negative X and Y values correspond to the upper left position. Missing values for ”HV” generally represent locations where voids occurred.

- {Dataset name}*.jpg (VISUALIZATION Directory): The raw data is plotted in numerous different ways, including with/without interpolation between points, with/without plot scales and axes, with/without a Gaussian fitting of the stir region used to spatially registering the center of the stir region. Note that the color scales are identical across all datasets.

- {Dataset name}*_weld_data.txt (ANALYSIS Directory): The text file contains pertinent information regarding hardness value determined in the entire stir zone, hardness value closest to the stir zone center, and the average value on a small 0.5x0.5 mm region near the center. Standard deviation of average values are listed. The location of the stir zone is listed in mm relative to the coordinates of the raw data.

The most useful data across multiple datasets is captured in directory ENSEMBLE-INFO for this modality. The information is provided for convenience and is directly replicated from the related datasets. Refer to the individual datasets for details. In the case of conflicting information between items found in this directory and with individual datasets, the individual dataset information takes precedence

- HARDNESS_Values.{csv, xlsx}: Contains a summary of all hardness data from across all the datasets as a convenience. This data is assembled from the individual dataset files.

5.0 Notes and Comments

The following observations are made about data and files for this modality:

1. NONE

6.0 Tabular Results

All hardness data is listed below in Table 6.1 for the entire stir region (SR), the ”window” (Wind) described earlier near the center, and the single point value closest to the center of the stir region. This data is obtained from the HARDNESS_Values.xls file described earlier and includes values for average and standard deviation.

Table 6.1. Listing of all hardness data.

Sample ID	Condition ID	SR Ave	SR Std	Wnd Avg	Wnd Std	Center Point
SS17	C08	235.43	13.34	229.06	5.97	225.14
SS18	C09	257.49	23.41	233.41	8.61	225.56
SS19	C10	228.31	20.12	228.04	7.70	227.69
SS20	C11	240.32	19.31	217.99	8.75	207.16
SS21	C06	266.12	16.66	260.67	7.75	259.42
SS22	C07	262.22	15.57	249.47	6.68	251.93
SS23	C03	284.88	16.63	282.53	8.03	287.02
SS34	C01	291.22	23.21	288.96	8.92	282.82

7.0 Graphics

The following sub sections display key graphics for convenience. Other graphics are available with the electronic data and the color scale used in all graphics is the same.

7.1 No Interpolation, Stir Region Fit Shown

The following figures show graphical results for all datasets collected in the round. These graphs indicate the discrete data with no interpolation between points and include a black curve showing a Gaussian shape fit to the data that is used to identify if a data point is within or outside the stir region. This fit was used to select data points to include in the ”entire stir region” average and standard deviation values previously listed in Table 6.1. Versions without scale bars and axis labels are available.

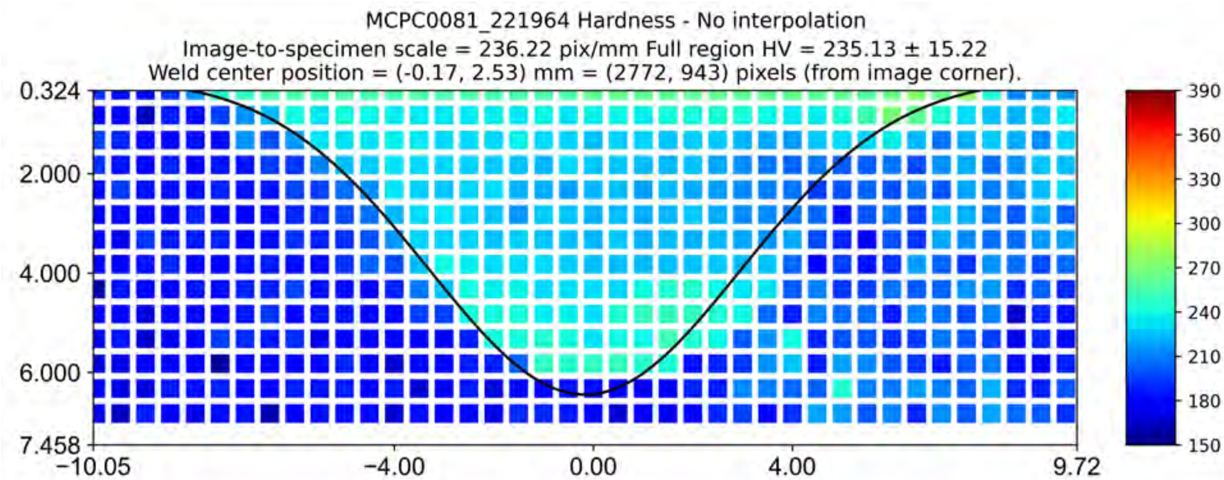

Figure 7.1. Full Stir Region Region with no Interpolation: Condition C08, Sample SS17

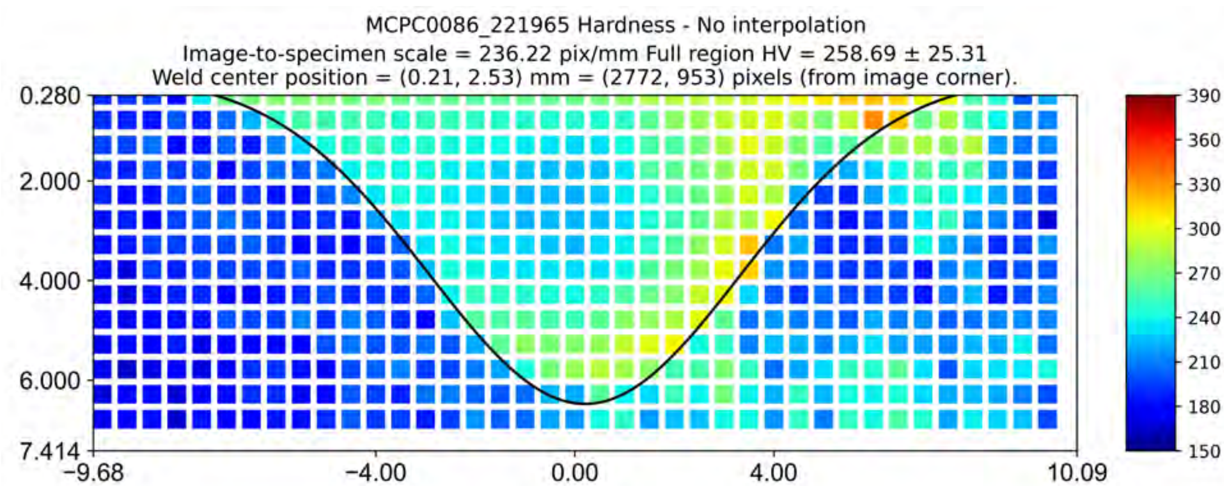

Figure 7.2. Full Stir Region Region with no Interpolation: Condition C09, Sample SS18

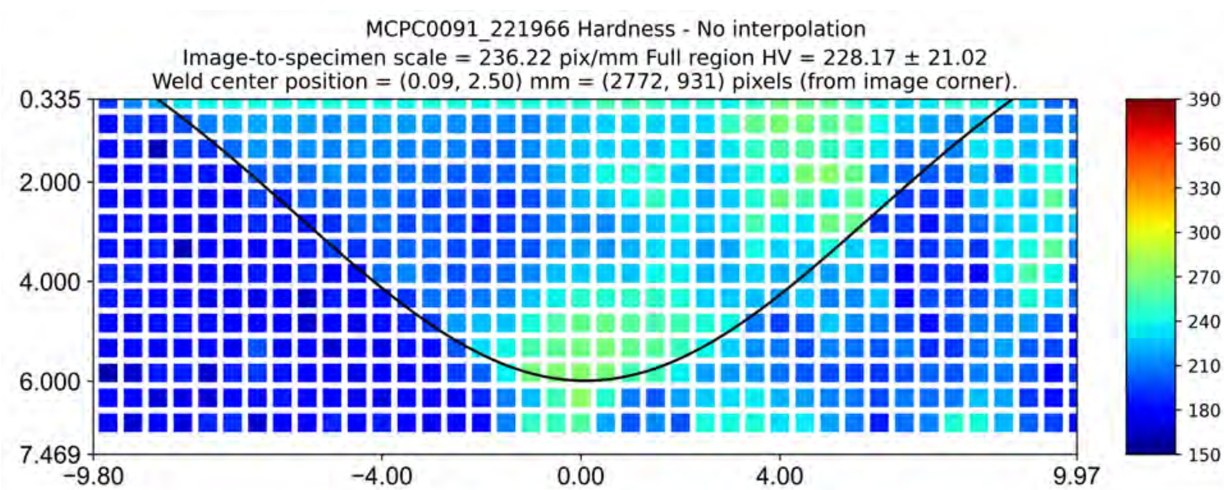

Figure 7.3. Full Stir Region Region with no Interpolation: Condition C10, Sample SS19

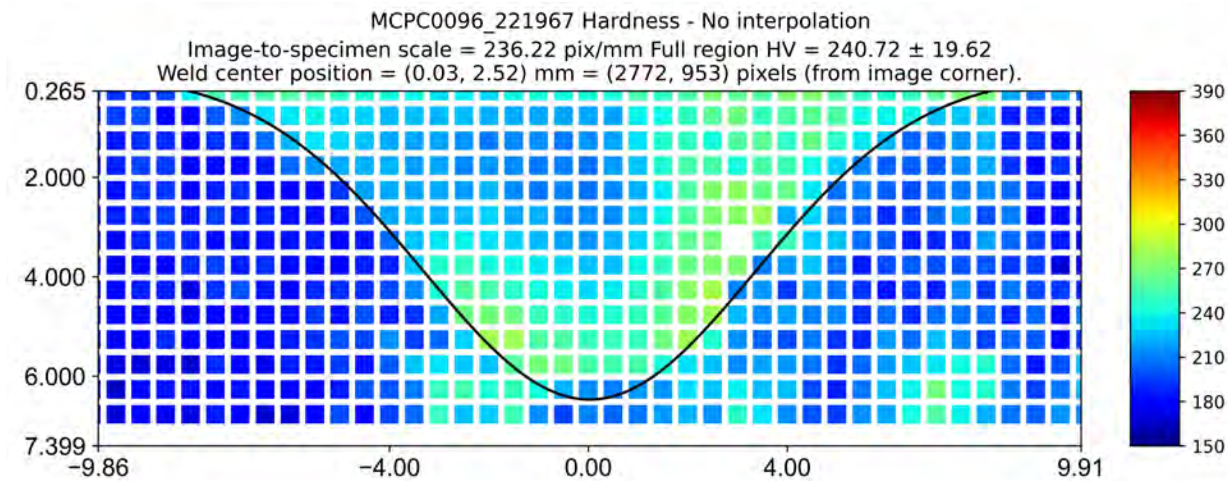

Figure 7.4. Full Stir Region Region with no Interpolation: Condition C11, Sample SS20

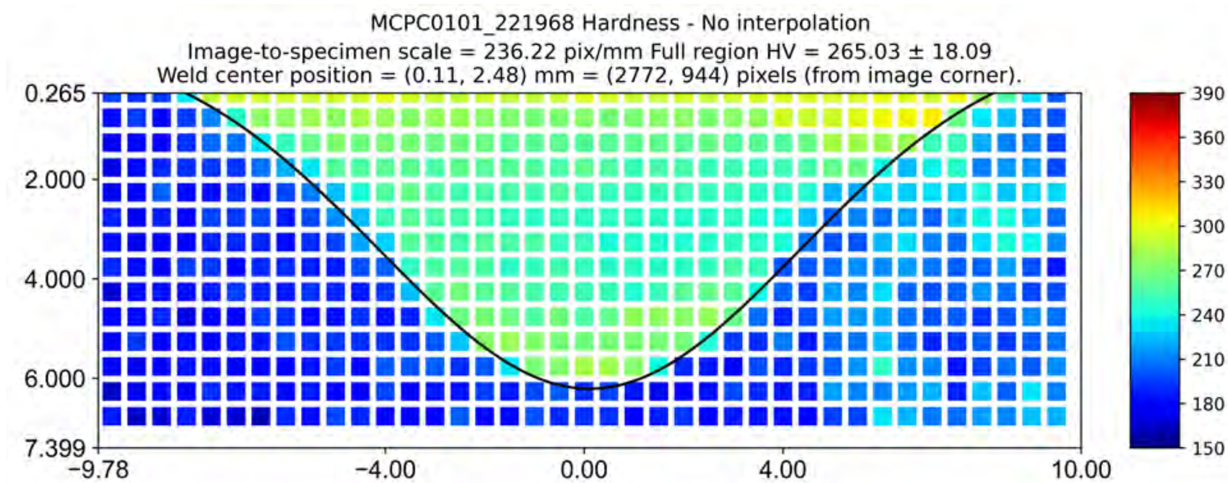

Figure 7.5. Full Stir Region Region with no Interpolation: Condition C06, Sample SS21

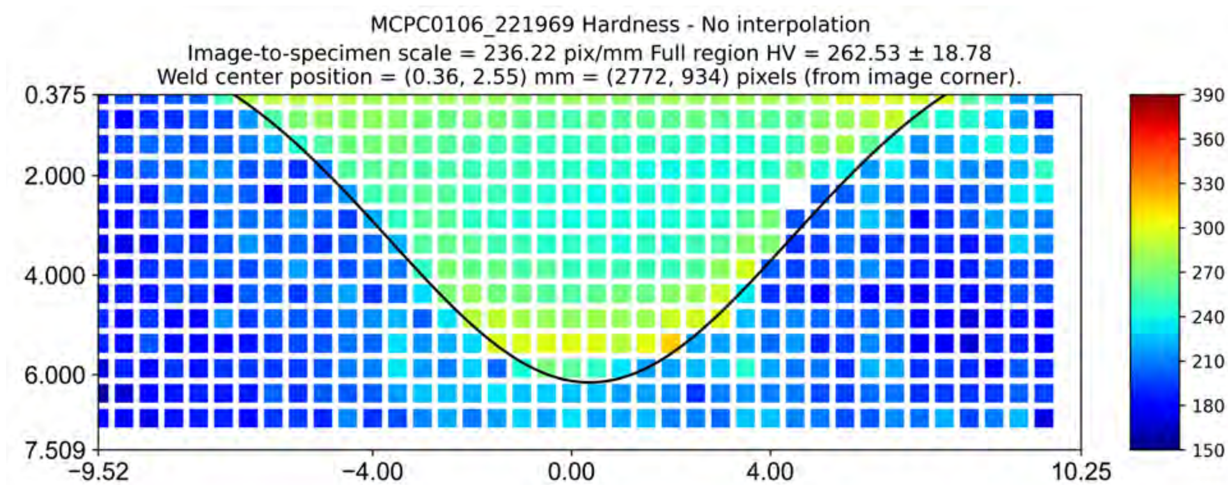

Figure 7.6. Full Stir Region Region with no Interpolation: Condition C07, Sample SS22

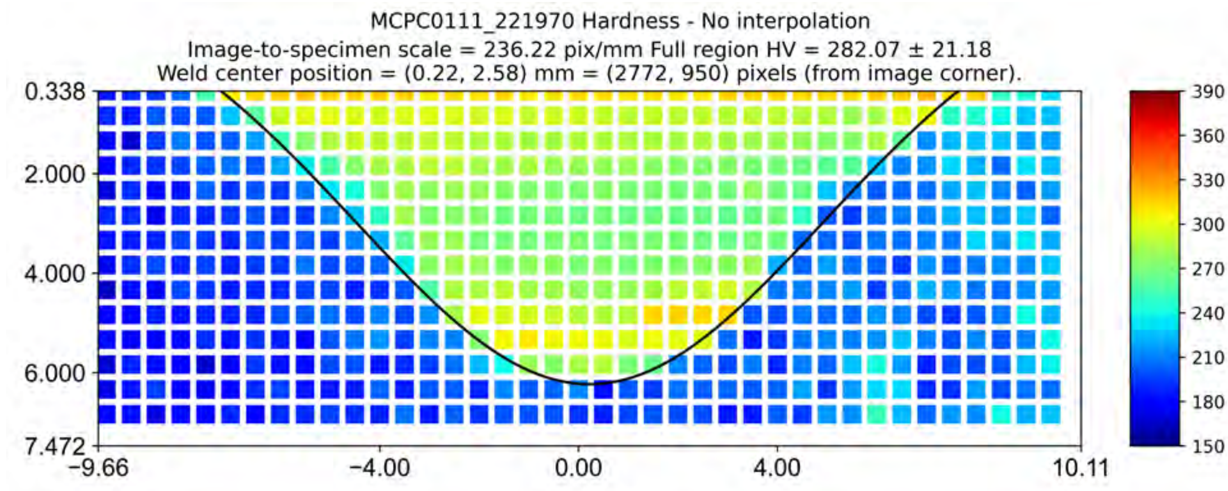

Figure 7.7. Full Stir Region Region with no Interpolation: Condition C03, Sample SS23

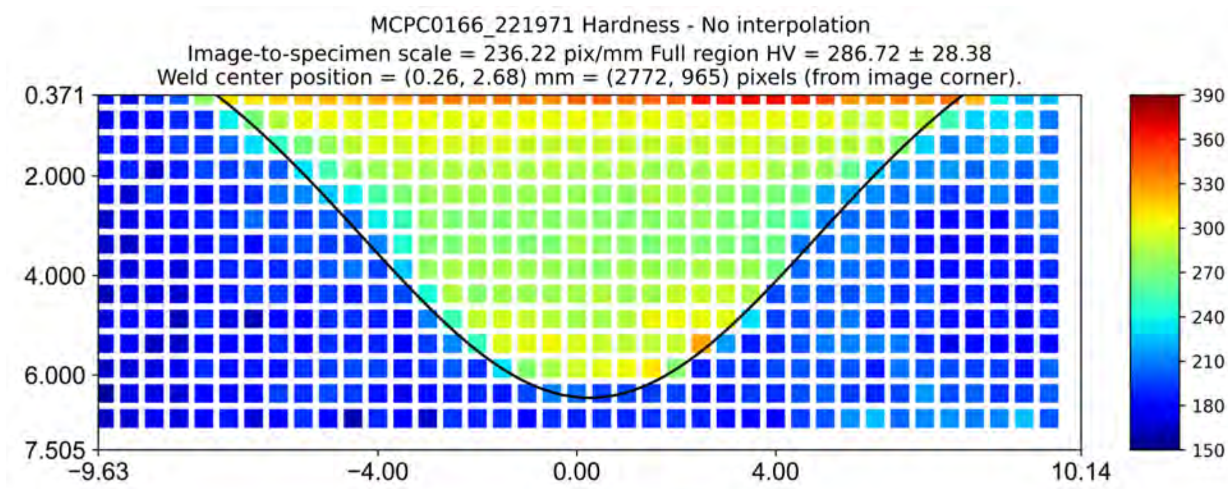

Figure 7.8. Full Stir Region Region with no Interpolation: Condition C01, Sample SS34

7.2 With Interpolation, Stir Region Fit Shown

The following figures show graphical results for all datasets in the round. These graphs are the same as the previous section, except that interpolation between points is performed.

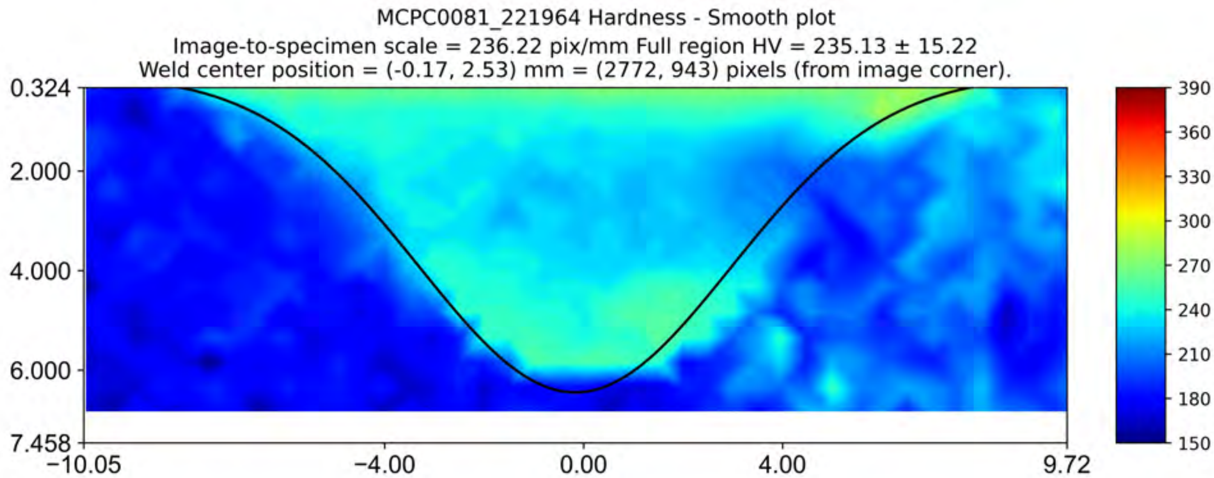

Figure 7.9. Full Stir Region Region with Interpolation: Condition C08, Sample SS17

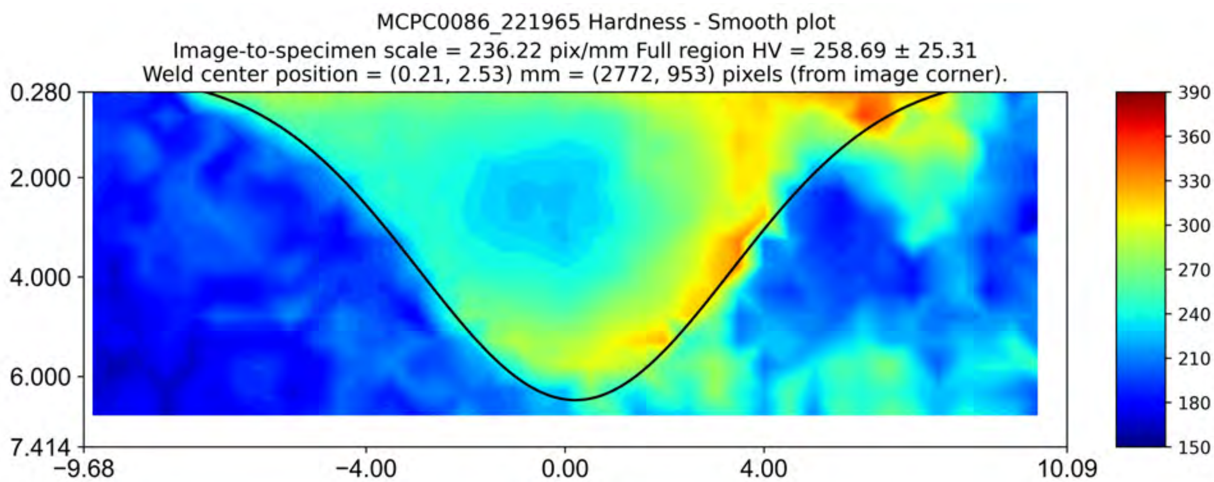

Figure 7.10. Full Stir Region Region with Interpolation: Condition C09, Sample SS18

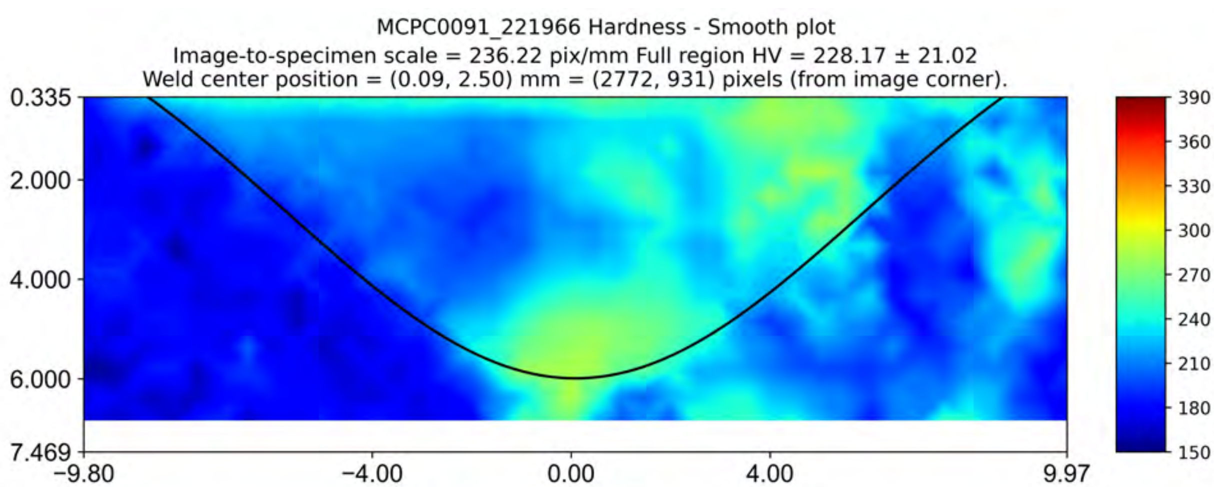

Figure 7.11. Full Stir Region Region with Interpolation: Condition C10, Sample SS19

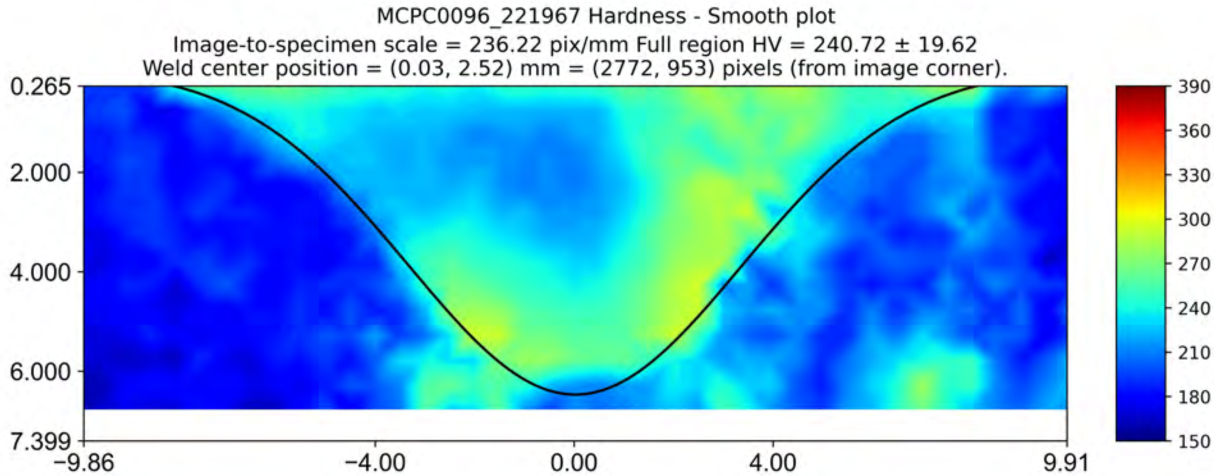

Figure 7.12. Full Stir Region Region with Interpolation: Condition C11, Sample SS20

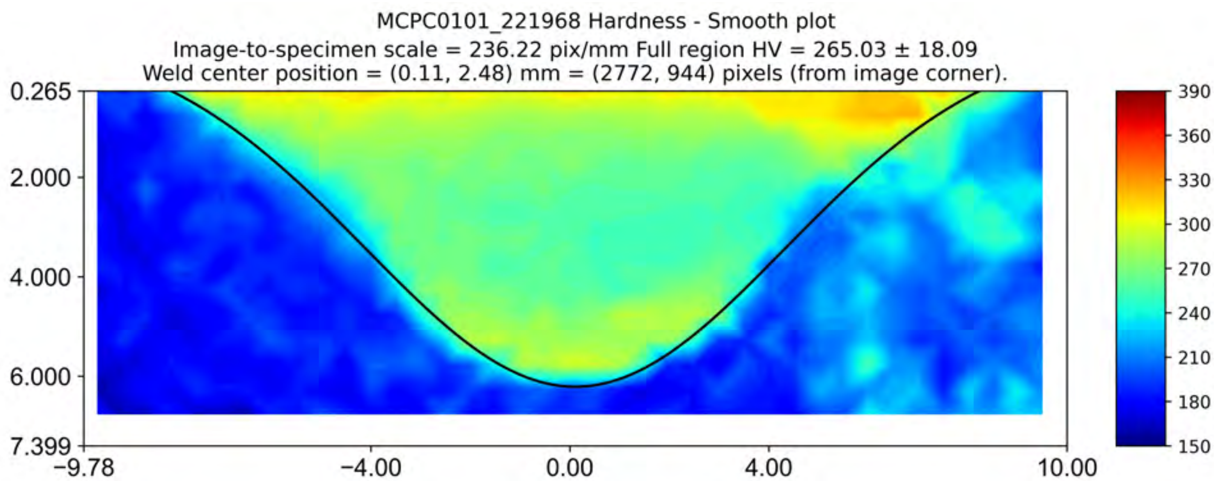

Figure 7.13. Full Stir Region Region with Interpolation: Condition C06, Sample SS21

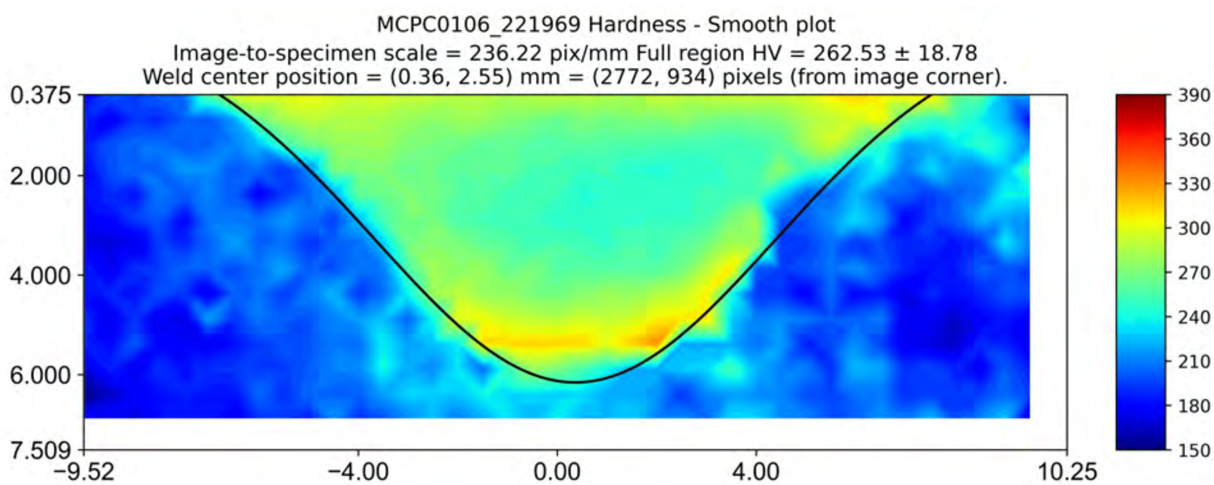

Figure 7.14. Full Stir Region Region with Interpolation: Condition C07, Sample SS22

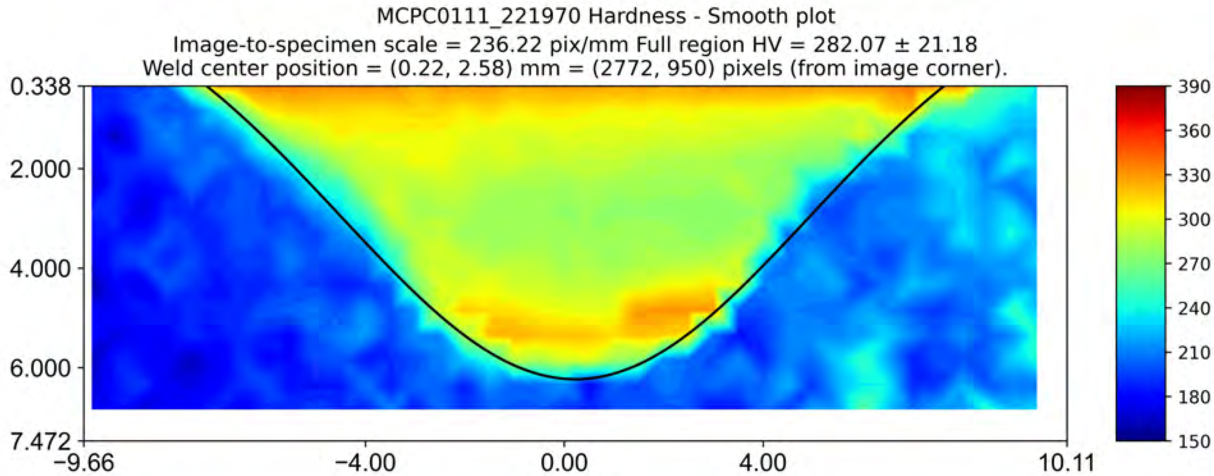

Figure 7.15. Full Stir Region Region with Interpolation: Condition C03, Sample SS23

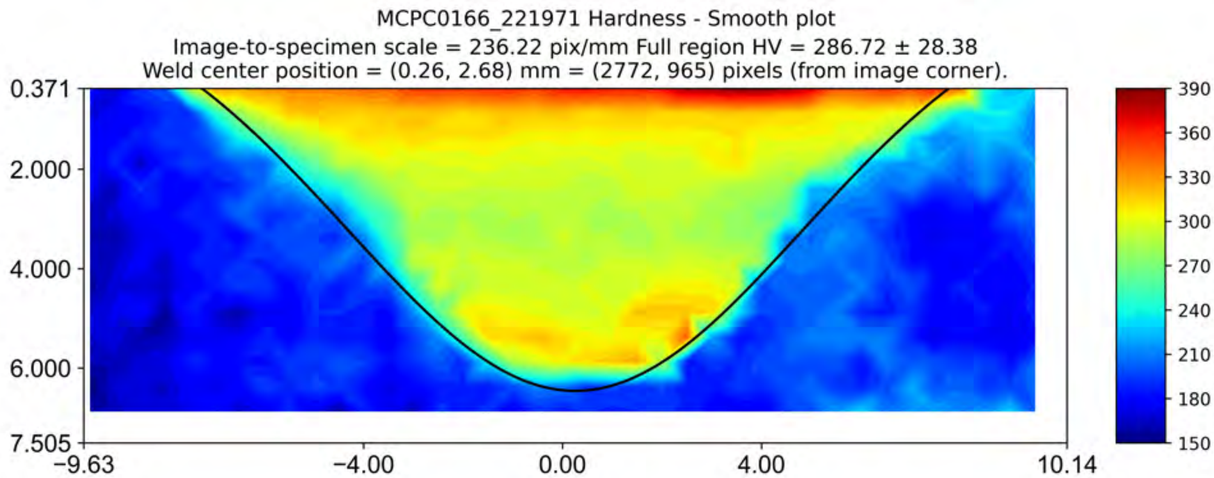

Figure 7.16. Full Stir Region Region with Interpolation: Condition C01, Sample SS34

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P.O. Box 999
Richland, WA 99354

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Attachment C: MCPC Round 3 Material Characterization Results for Modality OPTICAL

A Ortiz K Nwe
DR Todd A Guzman

Attachment C contains 21 pages

Acknowledgments

This research was supported by the Materials Characterization, Prediction, and Control (MCPC) investment, under the Laboratory Directed Research and Development (LDRD) Program at Pacific Northwest National Laboratory (PNNL). PNNL is a multi-program national laboratory operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute under Contract No. DE-AC05-76RL01830.

Contents

Figures

Tables

No Tables are associated with this Attachment

1.0 Introduction

Optical microscopy uses visible light and a system of lenses to generate magnified images of a specimen that has been mounted and polished. Contrasting agents and etchants can be used to reveal granular structures and other properties. Optical microscopy is traditional technique that is often performed early in a workflow to assist in identifying defects and areas of interest for more advanced microscopy techniques.

The following sections provide a summary of results for this modality to enhance dissemination of the large volume of similar data that are made available via the PNNL DataHub (https://data.pnnl.gov) platform.

2.0 Key Information and Results

The main portion of this report provides a summary of key information related to the modality, including processing conditions, identification information, and the numerical results of most focus in the project. Where to find the information and results in the main report is described below (Section, Table, and Figure numbers listed below are found in the main report). Note that application of optical microscopy did not results in quantitative data, so no tabular or quantitative results are provided in the main report or here for the modality.

- Nominal process conditions for the FSP experiments ar defined in Table 1 and Table 3 (data is in two different sorting orders).

- The Condition, Sample, and Specimen ID matrix is defined in Table 2 that is necessary to decode the nominal conditions applied to a particular specimen.

- Details about the modality data collection approach is defined in Section 4.0. This includes information about magnification levels for optical microscopy.

3.0 Instrumentation

An Olympus Model DP74 was used to collect all results

4.0 Results Files

A 100x magnification image (10x ocular and 10x magnification) is available for all stir regions where the specimens were etched to reveal more contrast of the grain structure. Two versions, one without and one with a scale bar were provided. The files have the following in their names:

- 10X_Nugget-Region-Etched
- 10X_Nugget-Region-Etched_scale

5.0 Notes and Comments

The following observations are made about data and files for this modality:

1. Imagery with a scale bar was in gray scale, but saved with RGB color channels when adding the scale. This resulted in the imagery containing a scale bar being 3x larger than the original image.

2. The microscope used to collect this data automatically produces the montage image and individual tiles captured as the instrument raster scans the specimens cannot be saved by the proprietary software. When creating montages, the software applies subtle blending between tiles that is acceptable for visual evaluation. However, machine learning applications may be impacted by the blending and may need to avoid using these regions of the montage.

3. Spatial location and rotation data to align the center of the stir region is only approximate.

6.0 Tabular Results

No tabular results are available for this modality.

7.0 Graphics

The following sub sections display key graphics for convenience:

7.1 Optical Imagery Results

Optical imagery for available specimens follows. Imagery for the control is not provided. The displayed images have been slightly rotated to improve alignment and image size reduced to 2000 pixels in width for handling convenience. The raw (gigabit sized) files are provided with electronic data.

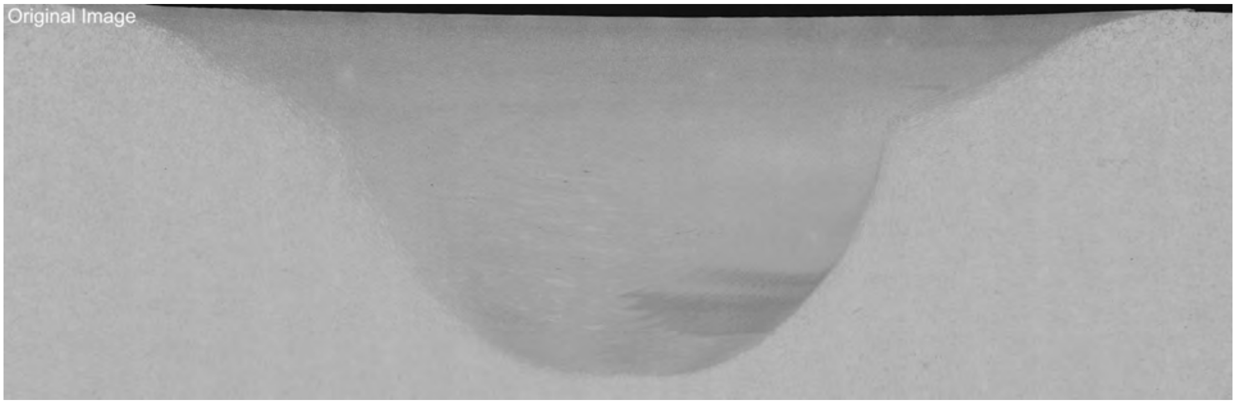

Figure 7.1. Optical Imagery at 100x mag: Condition C08, Sample SS17

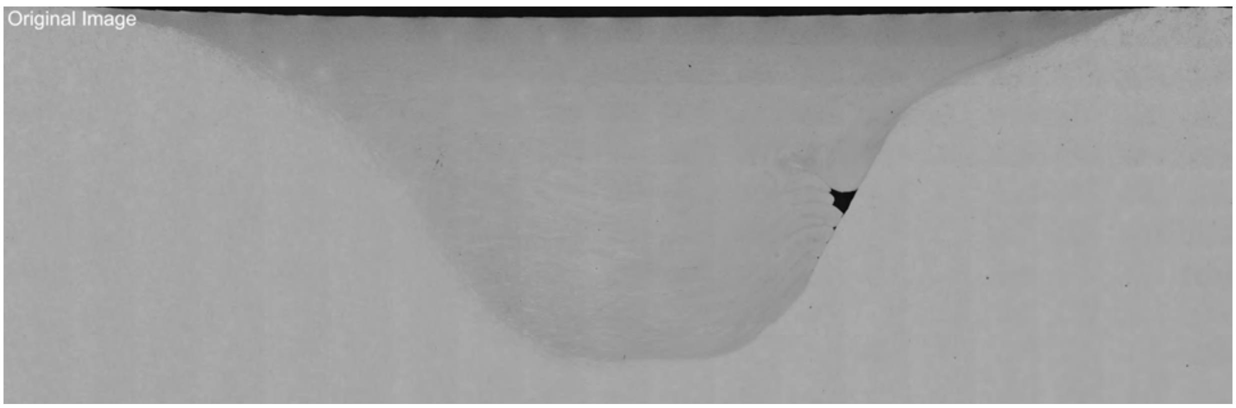

Figure 7.2. Optical Imagery at 100x mag: Condition C09, Sample SS18

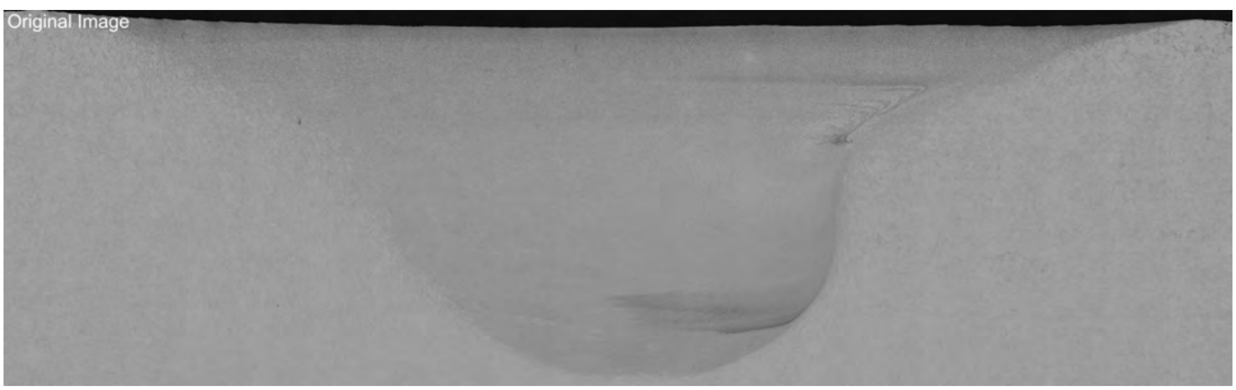

Figure 7.3. Optical Imagery at 100x mag: Condition C10, Sample SS19

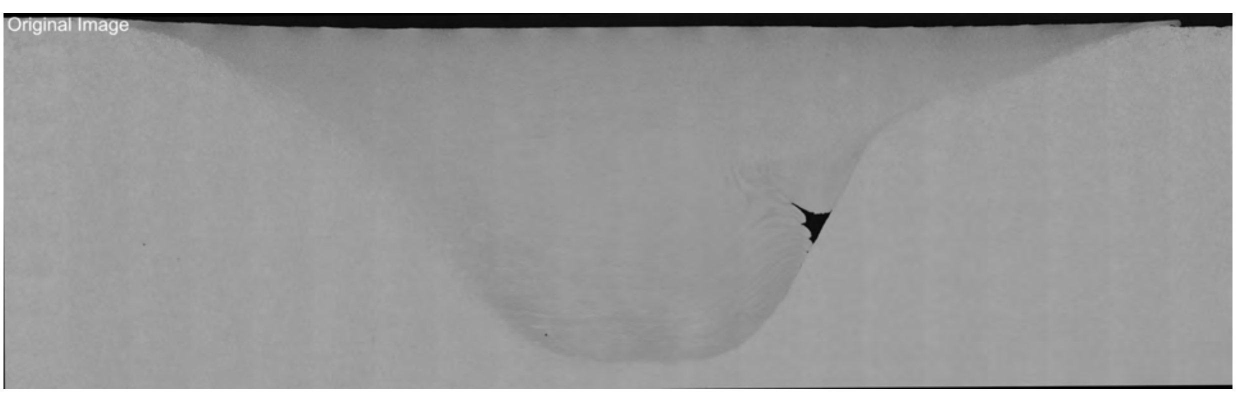

Figure 7.4. Optical Imagery at 100x mag: Condition C11, Sample SS20

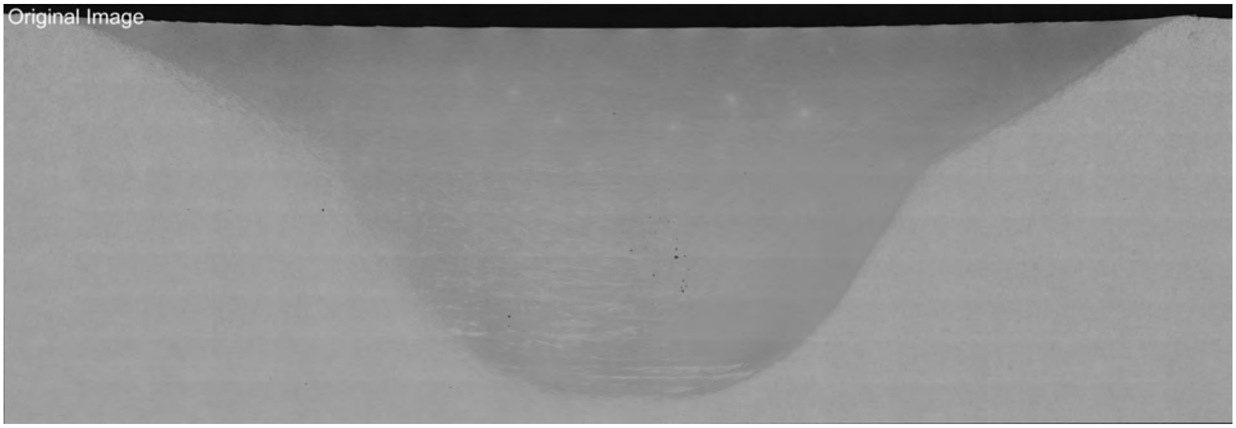

Figure 7.5. Optical Imagery at 100x mag: Condition C06, Sample SS21

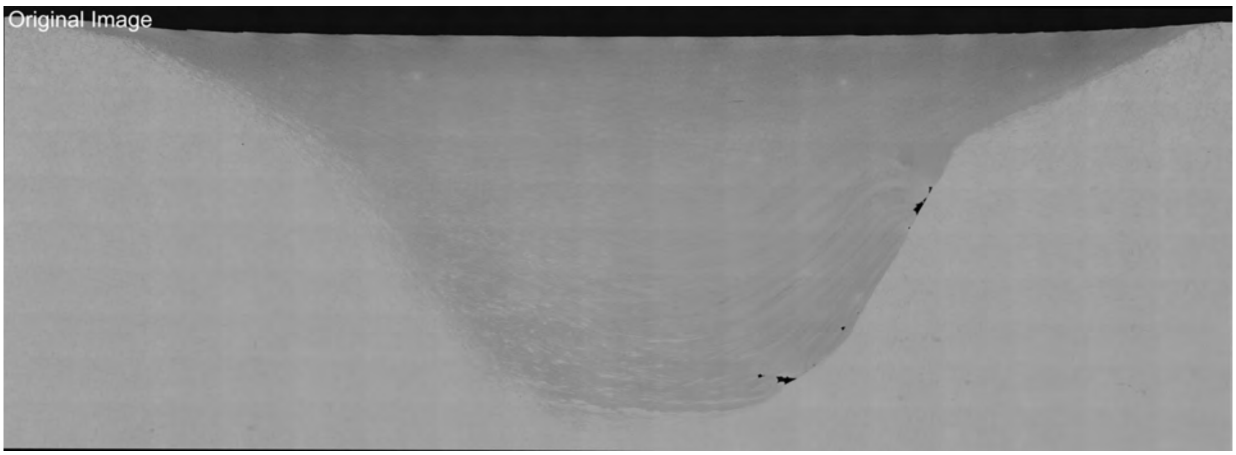

Figure 7.6. Optical Imagery at 100x mag: Condition C07, Sample SS22

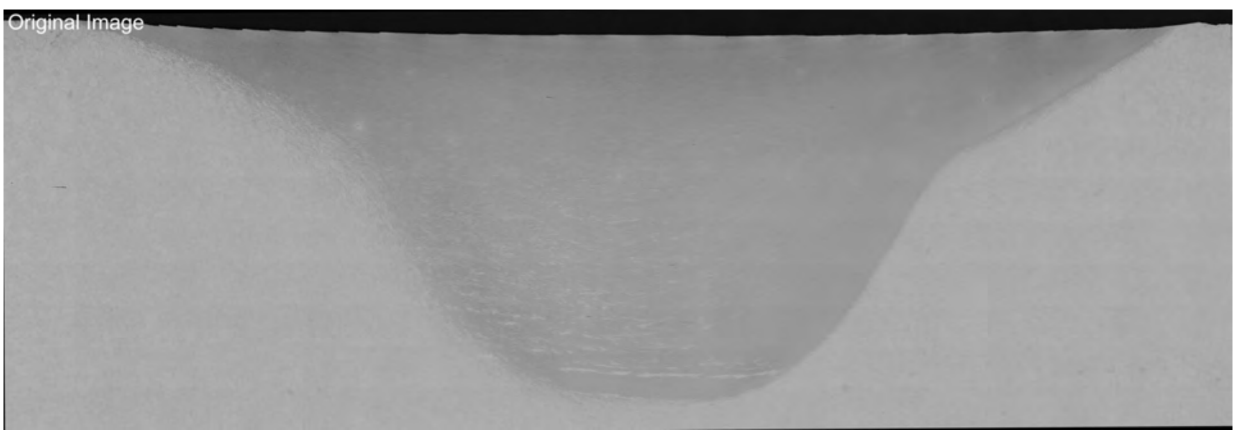

Figure 7.7. Optical Imagery at 100x mag: Condition C03, Sample SS23

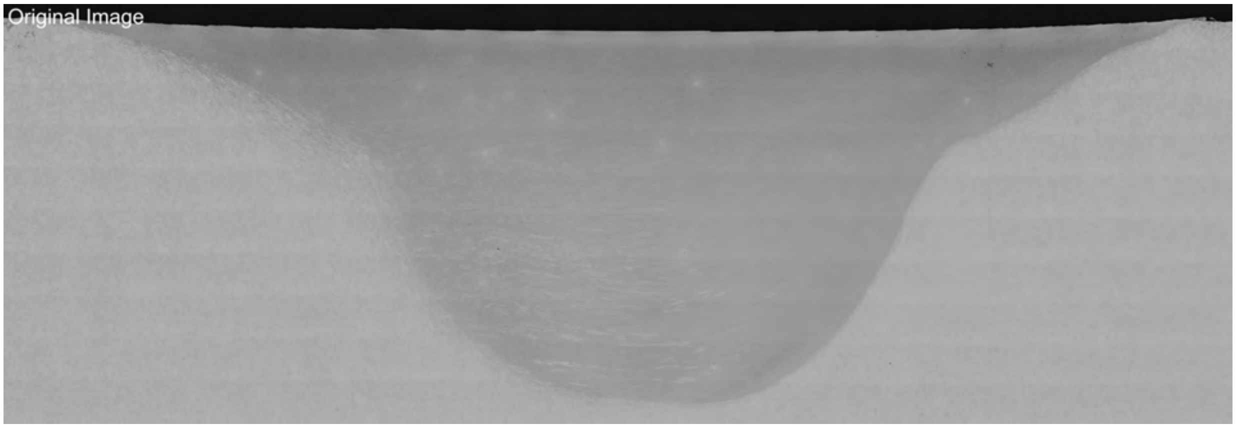

Figure 7.8. Optical Imagery at 100x mag: Condition C01, Sample SS34

7.2 Results Collages, 11 mm Width

The following figures show collages of all the results with the stir region in each specimen cropped to 11 mm in width. Note that the image for each specimen in the collage contains a label listing identification information, results for mean grain diameter and hardness, along with the nominal processing conditions. These values are the same as listed in the main report, except that the hardness values reflect stir region average value found in Attachment B, Table 6.1 (SR Ave). instead of the value for the small window near the center that is listed in Table 3 of the main report.

Three versions of each image collage are provided. One with no refinement of the gray scale pixel values (the raw image) but with some rotational adjustments, one with a global equalization of the gray scale pixel values meant to reveal larger trends in the data that can be hidden in low contrast regions, and a third in which local histogram equalization is applied to reveal very localized variations.

Other collage versions are available in electronic files with different cropping width and arrangements of the collage images.

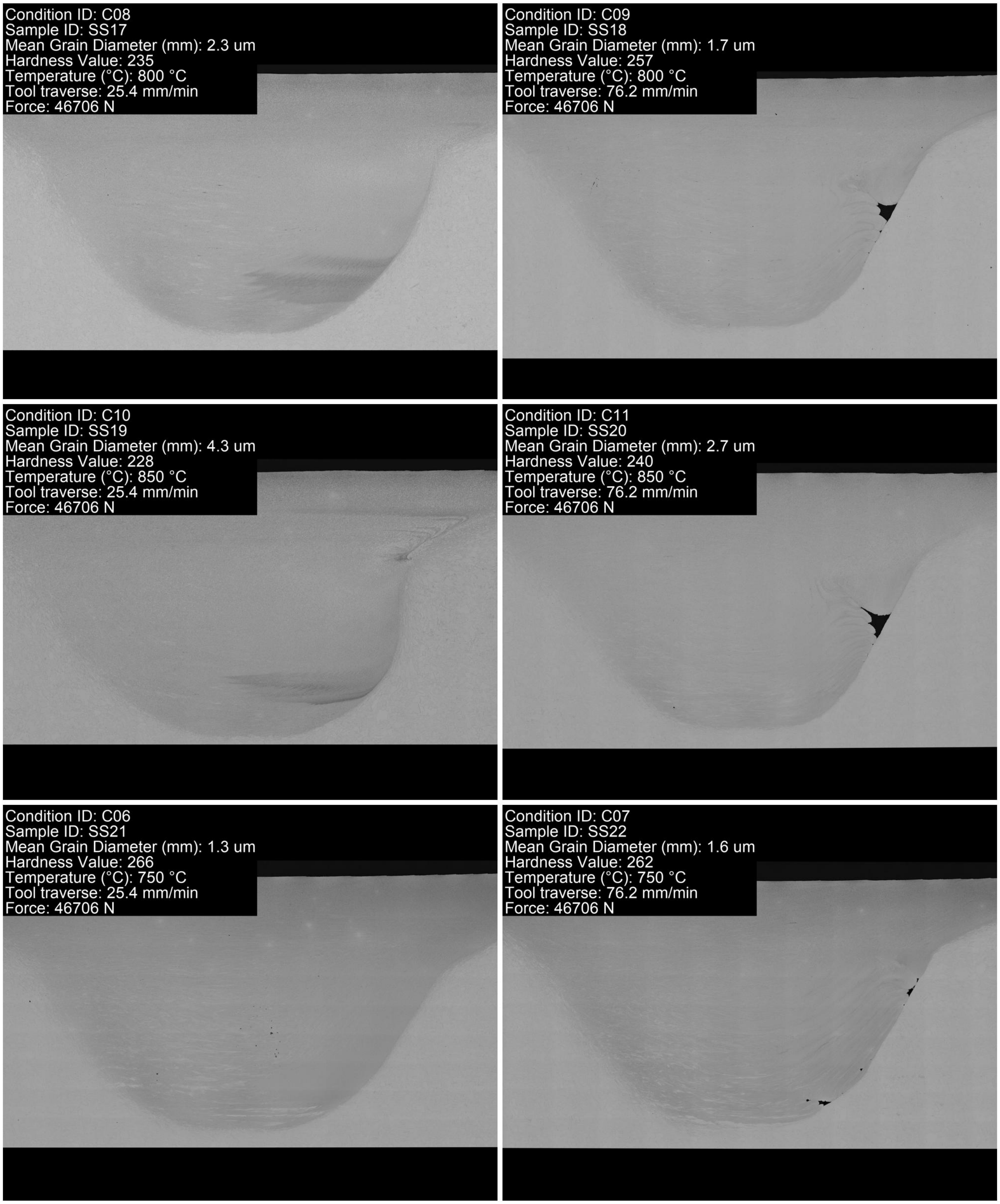

Figure 7.9. Stir Region Collage: Image number 1

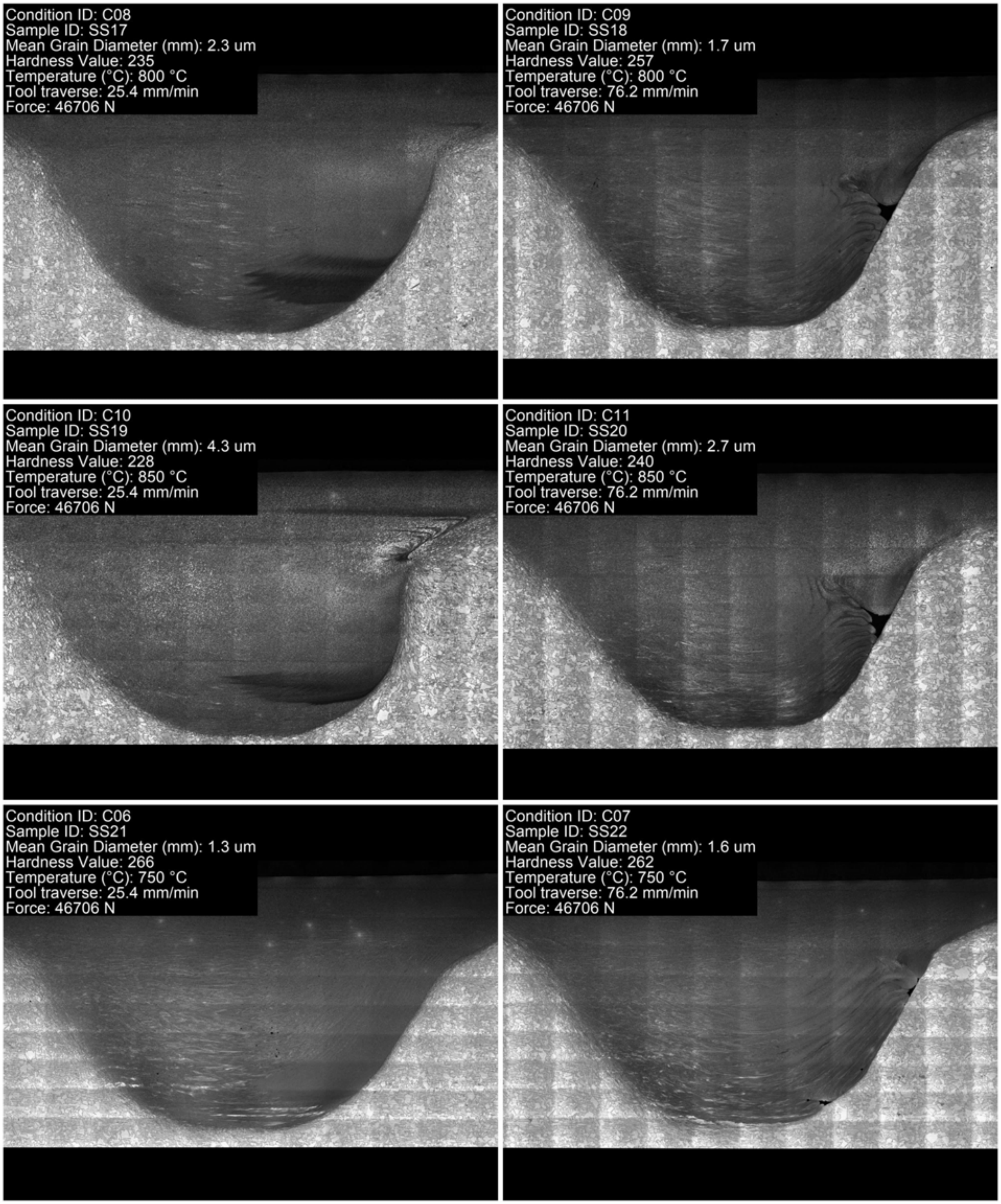

Figure 7.10. Stir Region Collage: Image number 2

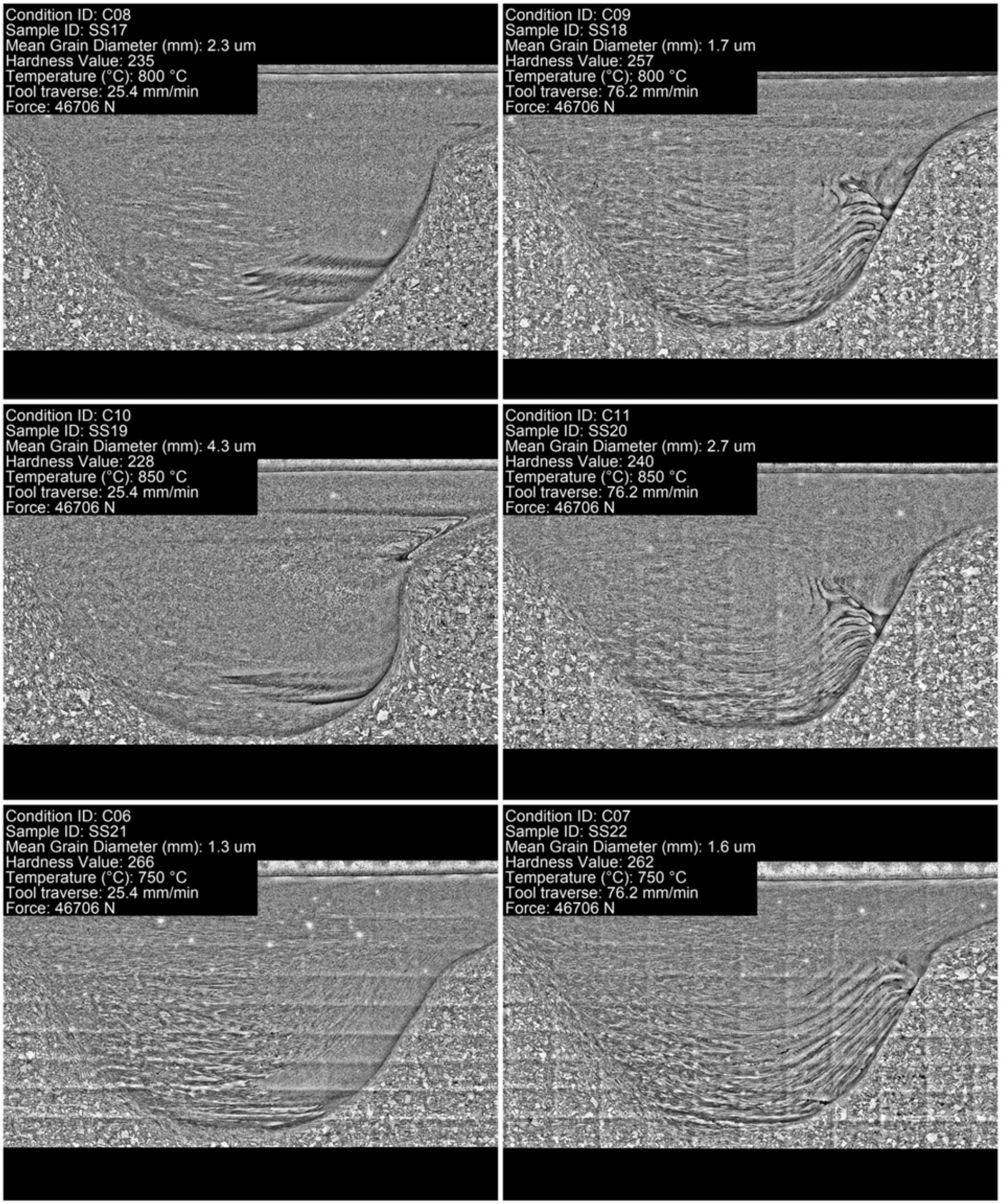

Figure 7.11. Stir Region Collage: Image number 3

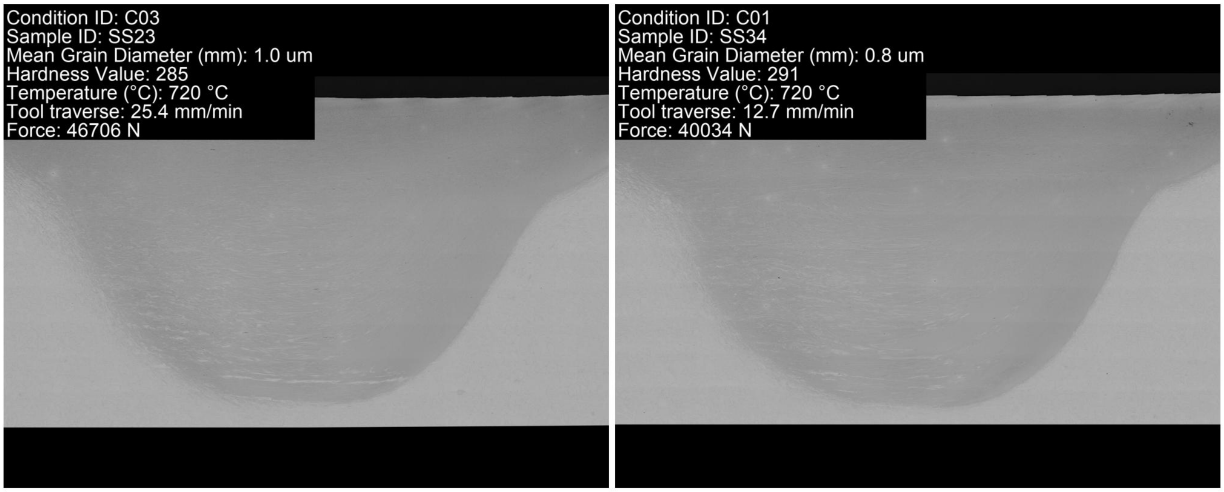

Figure 7.12. Stir Region Collage: Image number 4

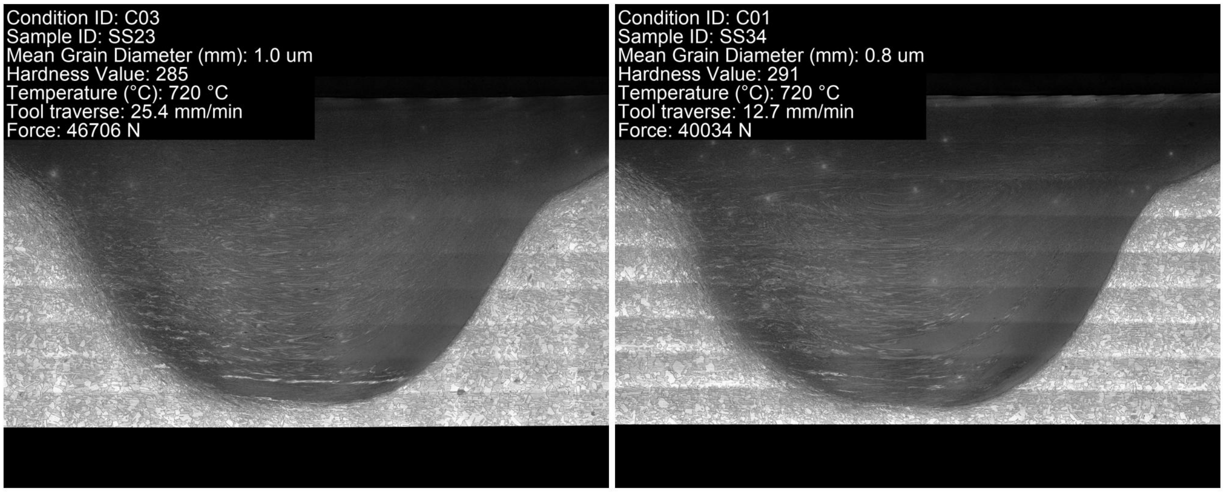

Figure 7.13. Stir Region Collage: Image number 5

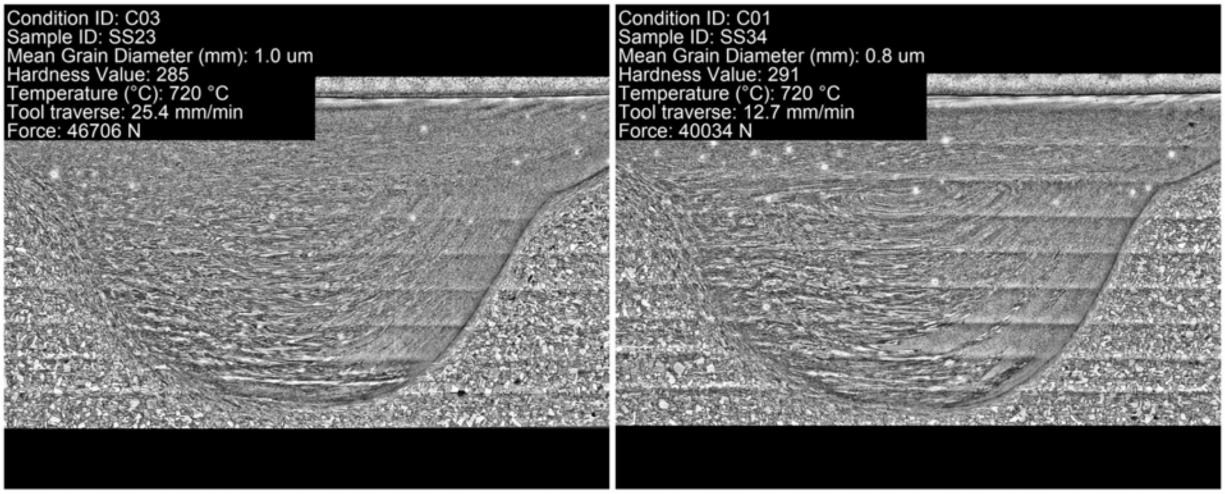

Figure 7.14. Stir Region Collage: Image number 6

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P.O. Box 999
Richland, WA 99354

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Attachment D: MCPC Round 3 Material Characterization Results for Modality SEM

M Pole K Nwe
DR Todd A Guzman

Attachment D contains 30 pages

Acknowledgments

This research was supported by the Materials Characterization, Prediction, and Control (MCPC) investment, under the Laboratory Directed Research and Development (LDRD) Program at Pacific Northwest National Laboratory (PNNL). PNNL is a multi-program national laboratory operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute under Contract No. DE-AC05-76RL01830.

Contents

Figures

Tables

No Tables are associated with this Attachment

1.0 Introduction

Backscatter Scanning Electron Microscopy (SEM) is a technique that uses accelerated electrons in the primary beam of a scanning electron microscope to diffract atomic layers in crystalline materials. These diffracted electrons can be detected when they impinge on a phosphor screen and generate visible lines

The following sections provide a summary of results for this modality to enhance dissemination of the large volume of similar data that are made available via the PNNL DataHub (https://data.pnnl.gov) platform.

2.0 Key Information and Results

The main portion of this report provides a summary of key information related to the modality, including processing conditions, identification information, and the numerical results of most focus in the project. Where to find the information and results in the main report is described below (Section, Table, and Figure numbers listed below are found in the main report). Note that application of SEM did not results in quantitative data, so no tabular or quantitative results are provided in the main report or here for the modality.

- Nominal process conditions for the FSP experiments are defined in Table 1 and Table 3 (data is in two different sorting orders).

- The Condition, Sample, and Specimen ID matrix is defined in Table 2 that is necessary to decode the nominal conditions applied to a particular specimen.

- Details about the modality data collection approach is defined in Section 4.0. This includes information about magnification levels for SEM.

3.0 Instrumentation

The following instrument was utilized in testing of specimens:

Thermoscientific Helios Hydra UX, Plamsa Focused Ion Beam microscope

4.0 Results Files

4.1 DATA Directory Contents

Data files obtained for each dataset from the instrumentation are saved in the associated DATA directory. A single SEM tile is obtained at each location at various magnifications and accelerator voltages.

- **(name).tif** (TIF Image type): The SEM image.

4.2 VISUALIZATION Directory Contents

The following visualizations are provided:

- **CROPPED_IMAGES** (Directory): Contains each of the images in DATA, but the fiducial mark and gutter information have been cropped off each image to assist machine learning applications.

5.0 Notes and Comments

The following observations are made about data and files for this modality:

1. None

6.0 Tabular Results

No tabular results are available for this modality.

7.0 Graphics

SEM results obtained at Location L02 are provided in this section. Measurements were performed at multiple magnifications and acceleration voltages with one panel per magnitude, voltage. The most typical magnifications were 3500 and 8000, but others were also used depending on the size of grains. The following subsections show results at 20 kV acceleration voltage for the range of magnifications. Note that the fiducial markers are visible in the upper left corner of all figures. And with careful inspection, the same grains can be located in EBSD results for the same samples in Attachment A.

7.1 SEM with 2500x Magnification

SEM images at 2500x magnification are provided in this section. Only one specimen was measured at this magnification.

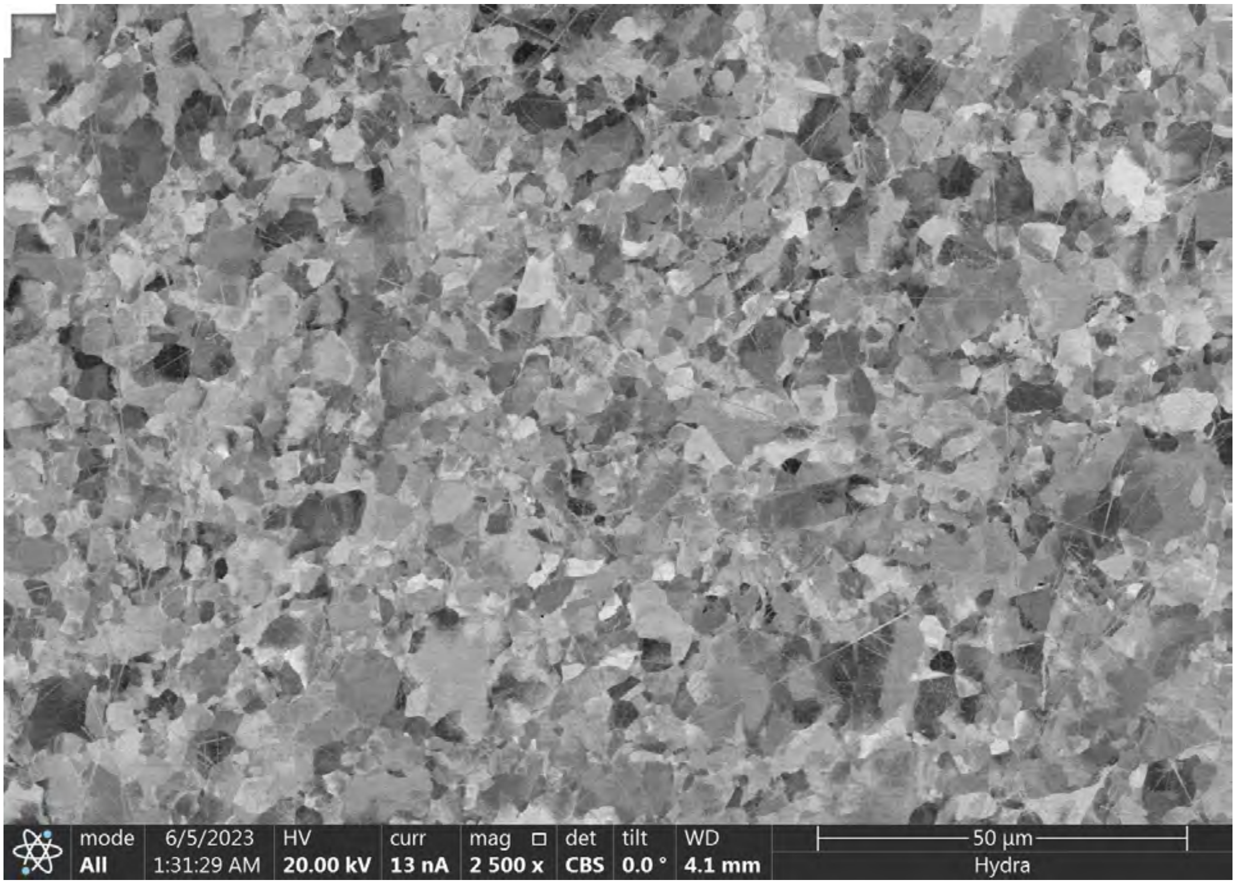

Figure 7.1. SEM Results at 2500 magnification for L02: Condition C10, Sample SS19

7.2 SEM with 3500x Magnification

SEM images at 3500x magnification are provided in this section. Seven specimens were measured at this magnification.

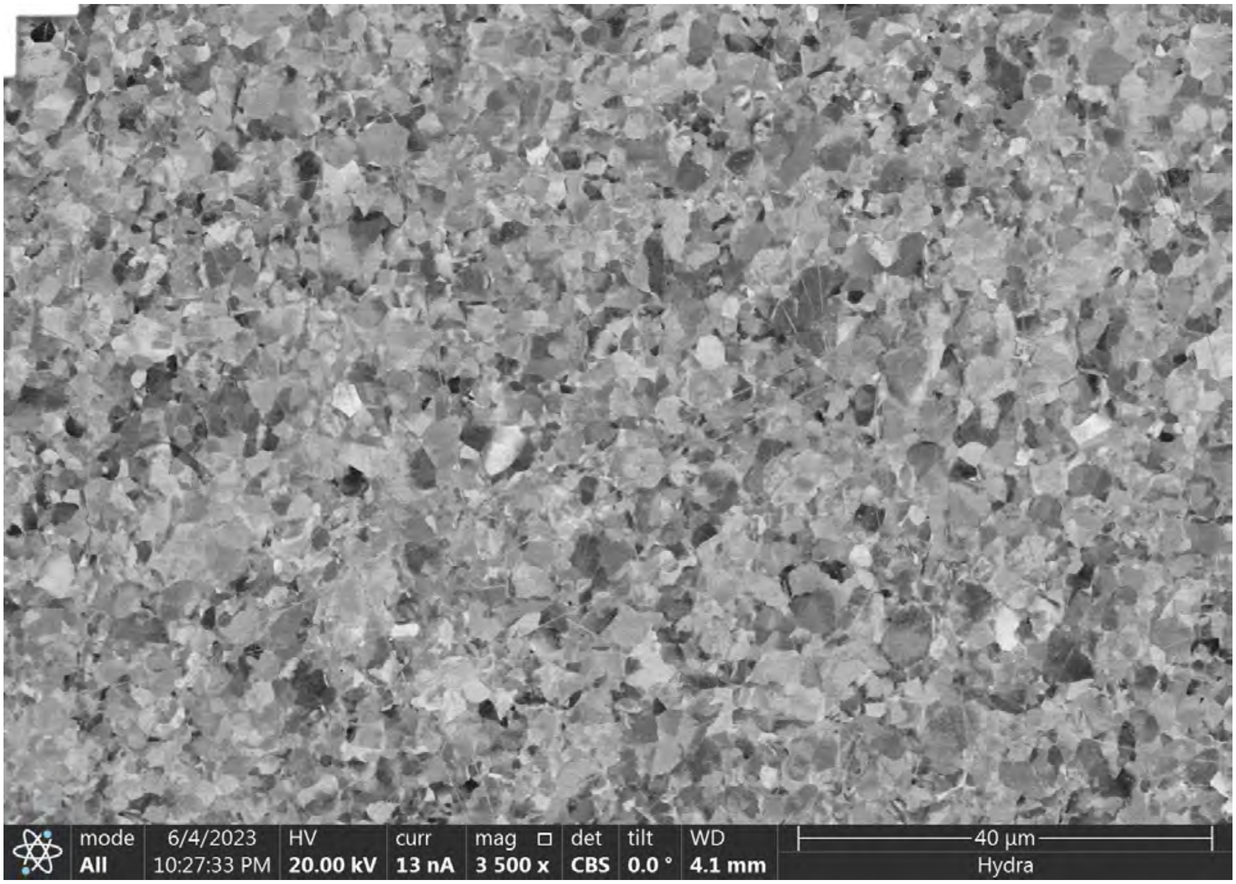

Figure 7.2. SEM Results at 3500 magnification for L02: Condition C08, Sample SS17

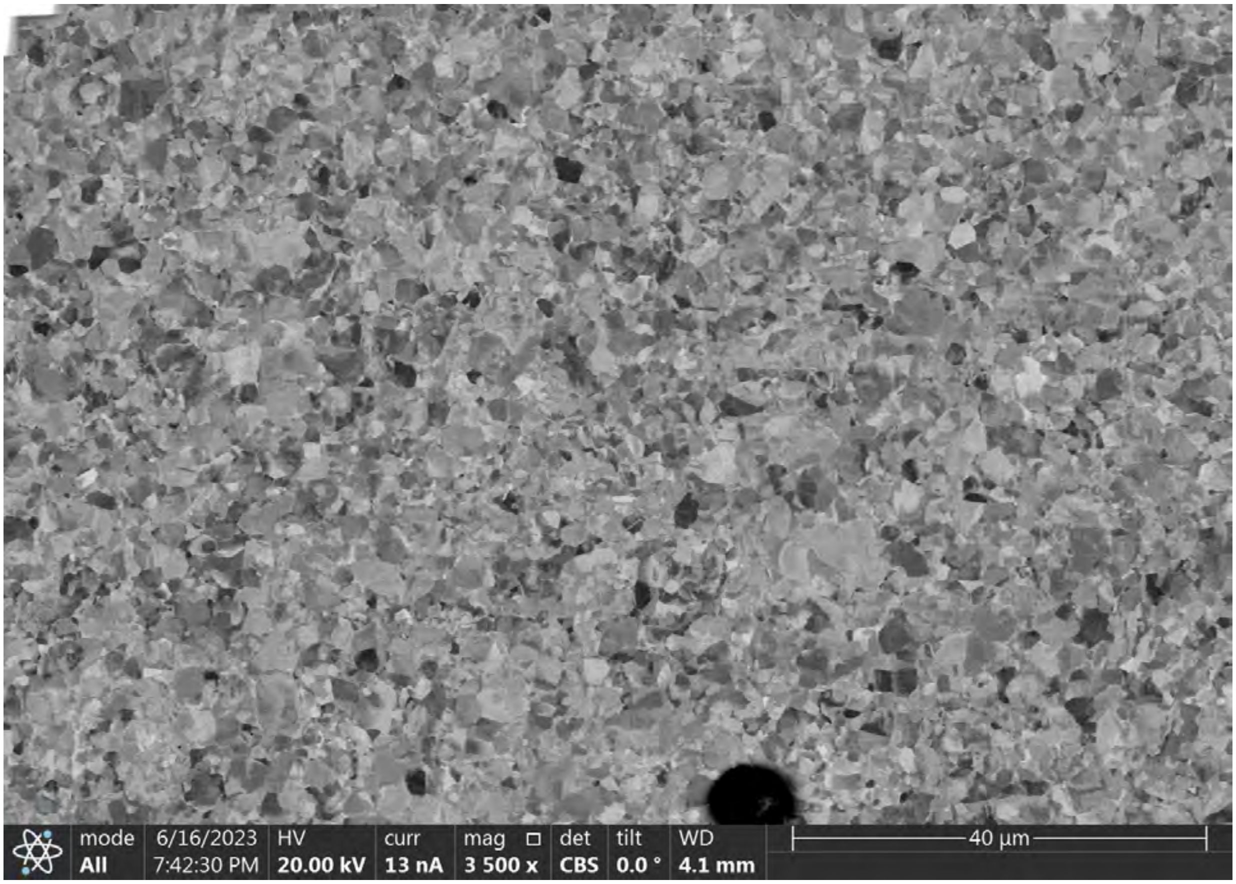

Figure 7.3. SEM Results at 3500 magnification for L02: Condition C09, Sample SS18

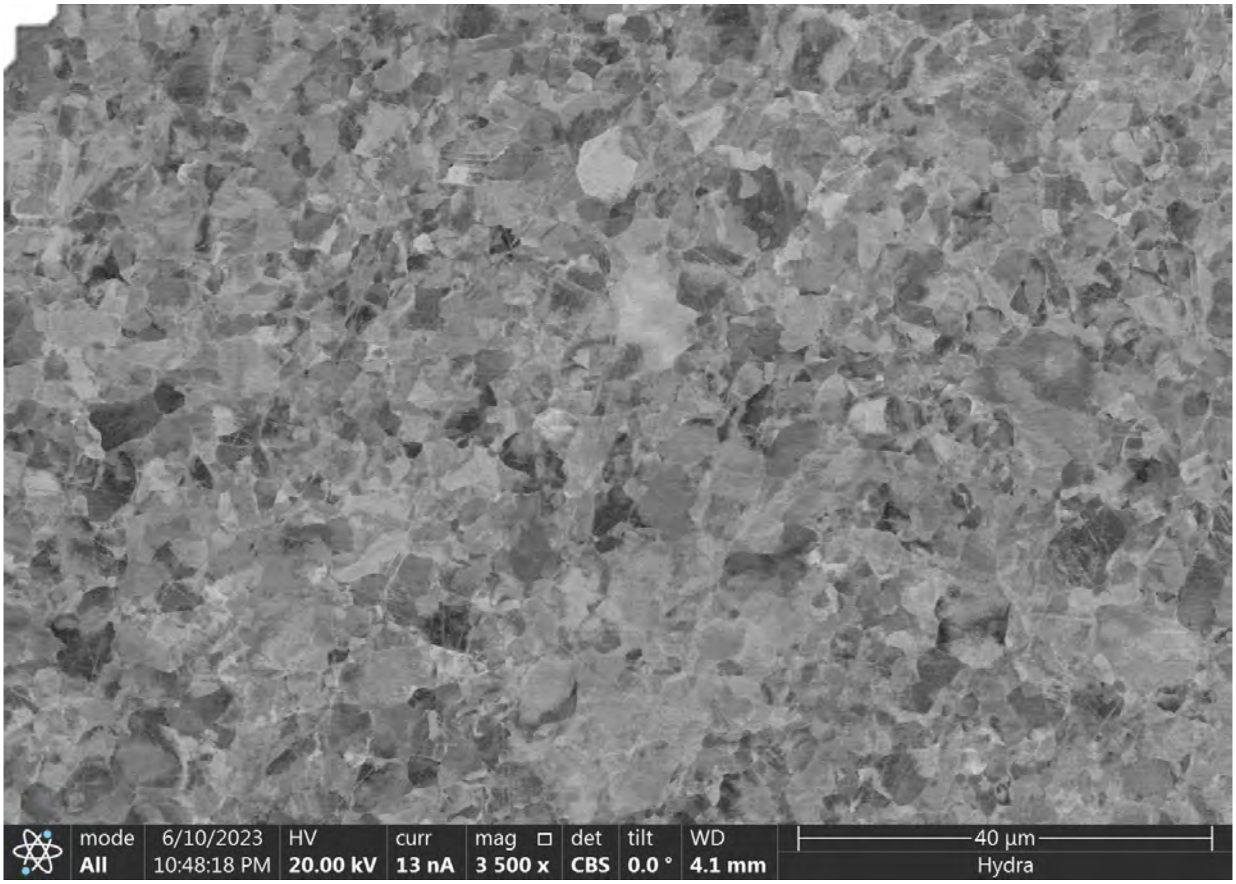

Figure 7.4. SEM Results at 3500 magnification for L02: Condition C11, Sample SS20

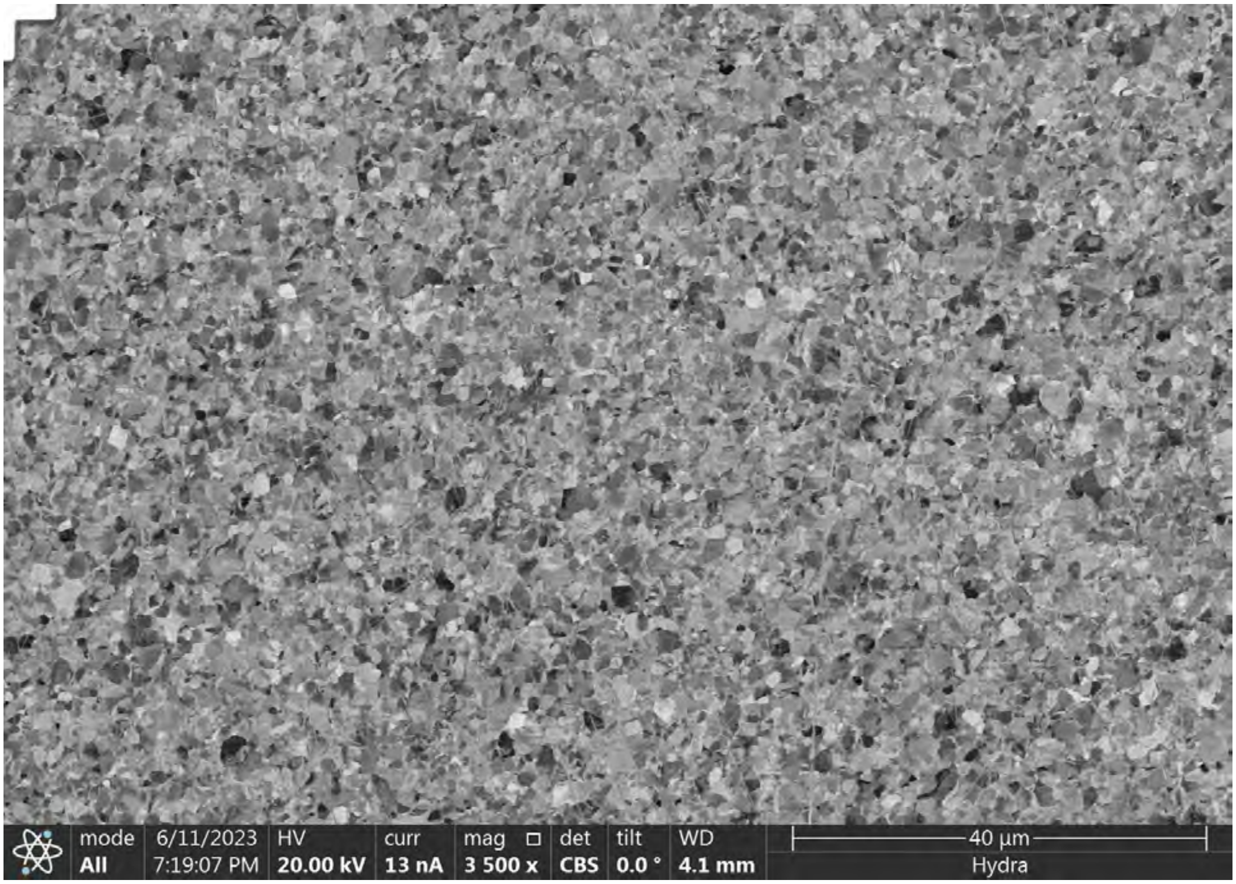

Figure 7.5. SEM Results at 3500 magnification for L02: Condition C06, Sample SS21

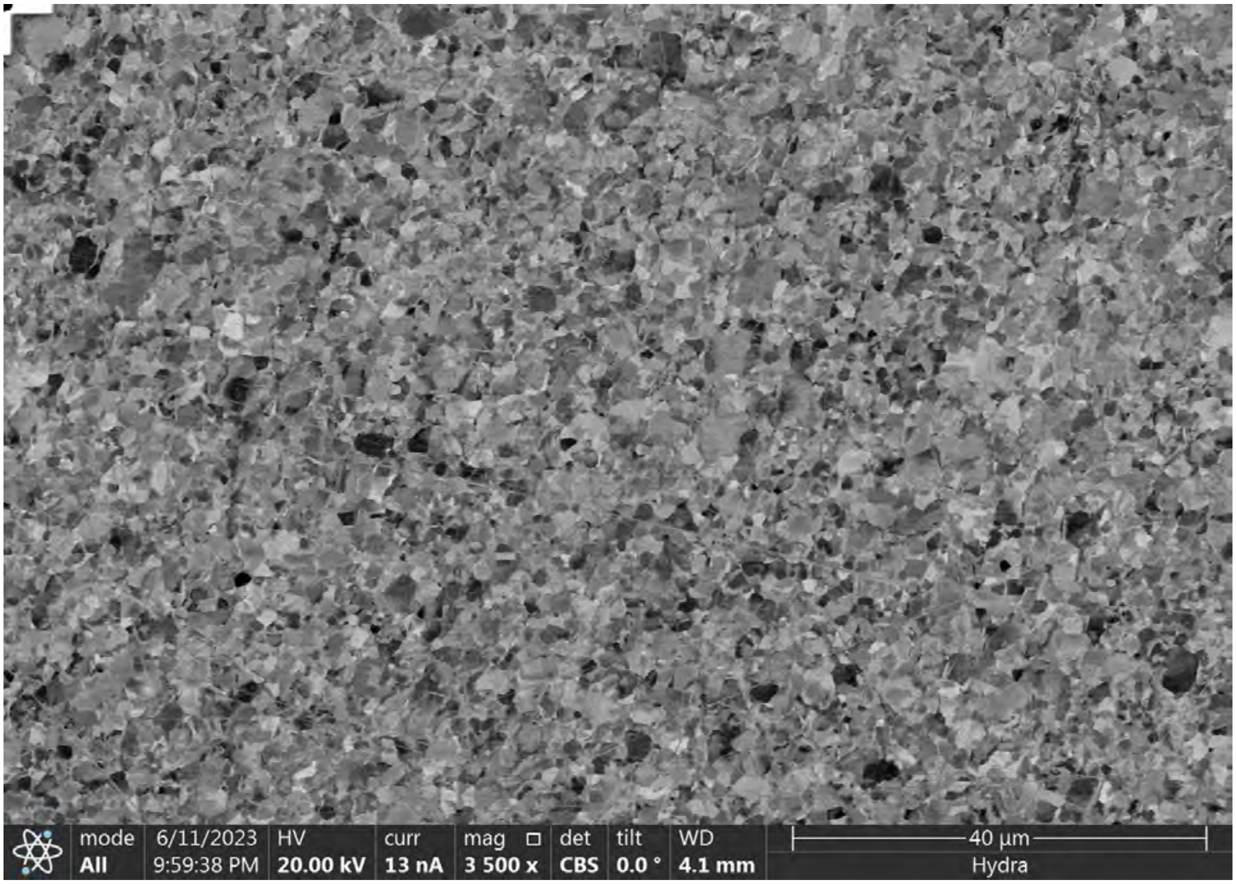

Figure 7.6. SEM Results at 3500 magnification for L02: Condition C07, Sample SS22

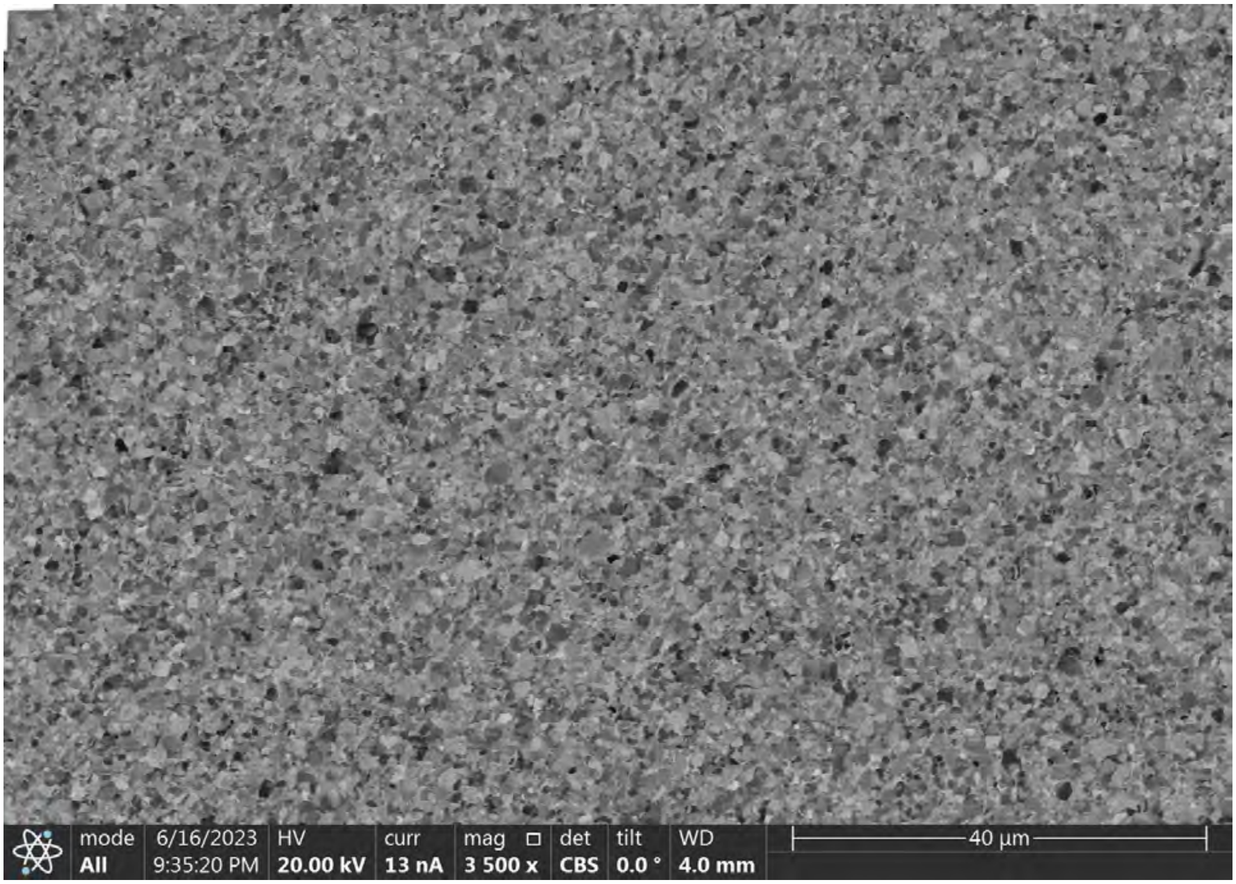

Figure 7.7. SEM Results at 3500 magnification for L02: Condition C03, Sample SS23

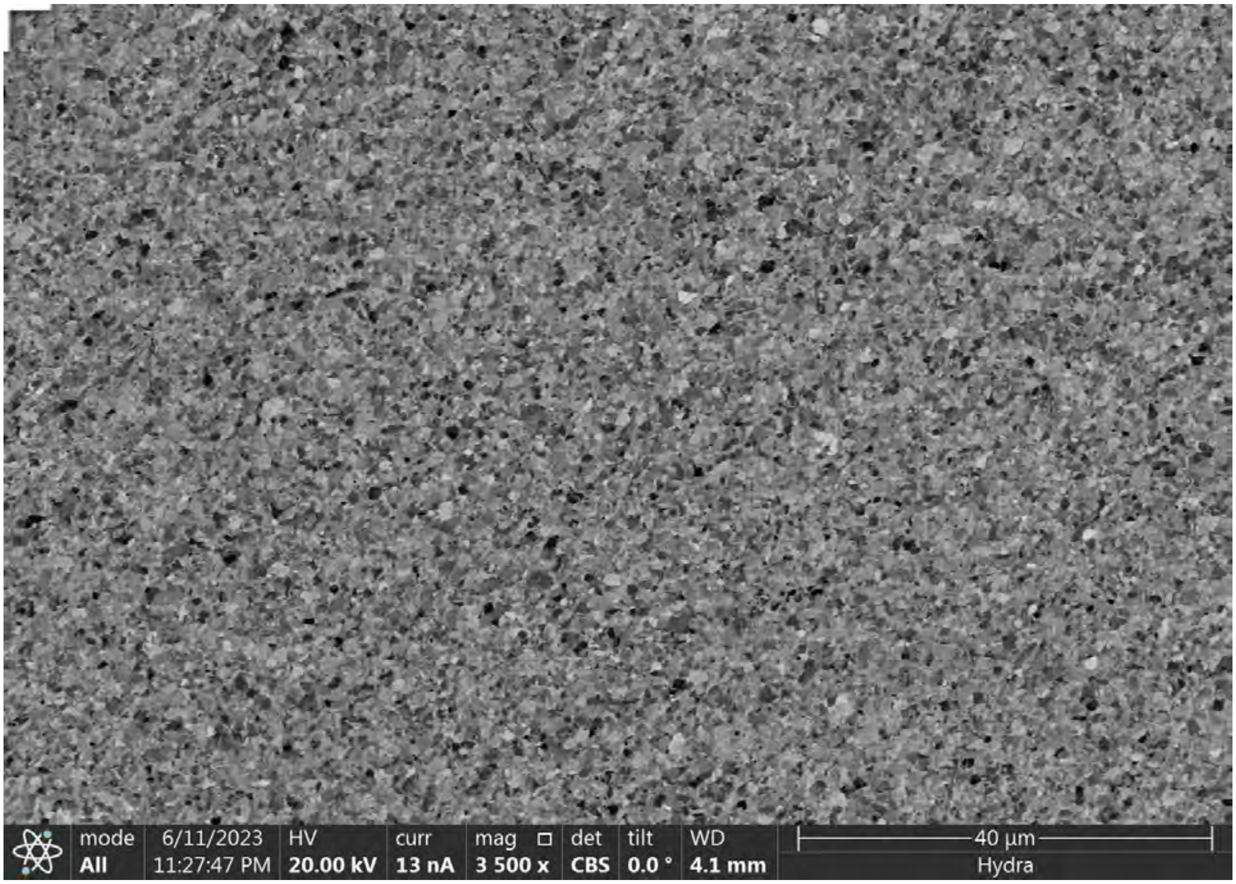

Figure 7.8. SEM Results at 3500 magnification for L02: Condition C01, Sample SS34

7.3 SEM with 8000x Magnification

SEM images at 8000x magnification are provided in this section. All available specimens were measured at this magnification.

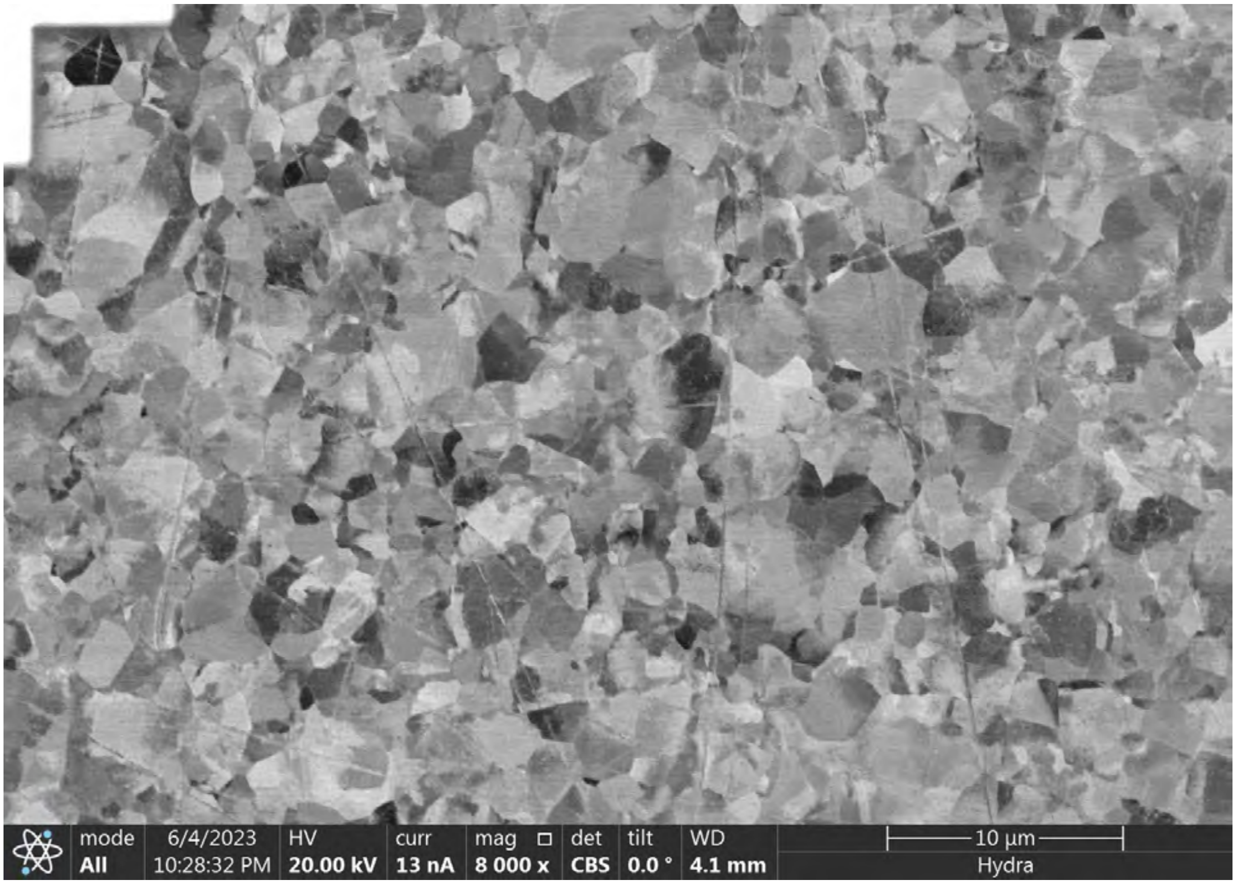

Figure 7.9. SEM Results at 8000 magnification for L02: Condition C08, Sample SS17

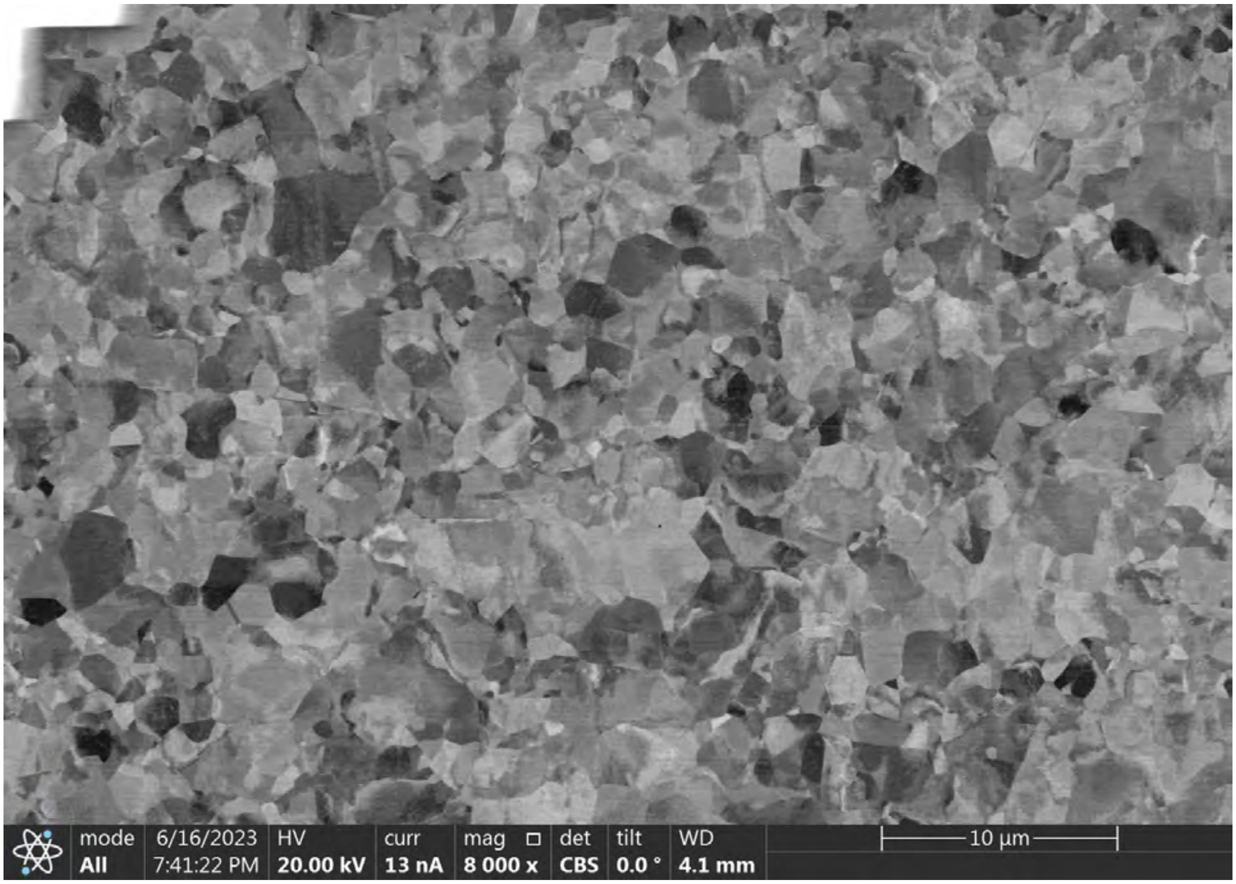

Figure 7.10. SEM Results at 8000 magnification for L02: Condition C09, Sample SS18

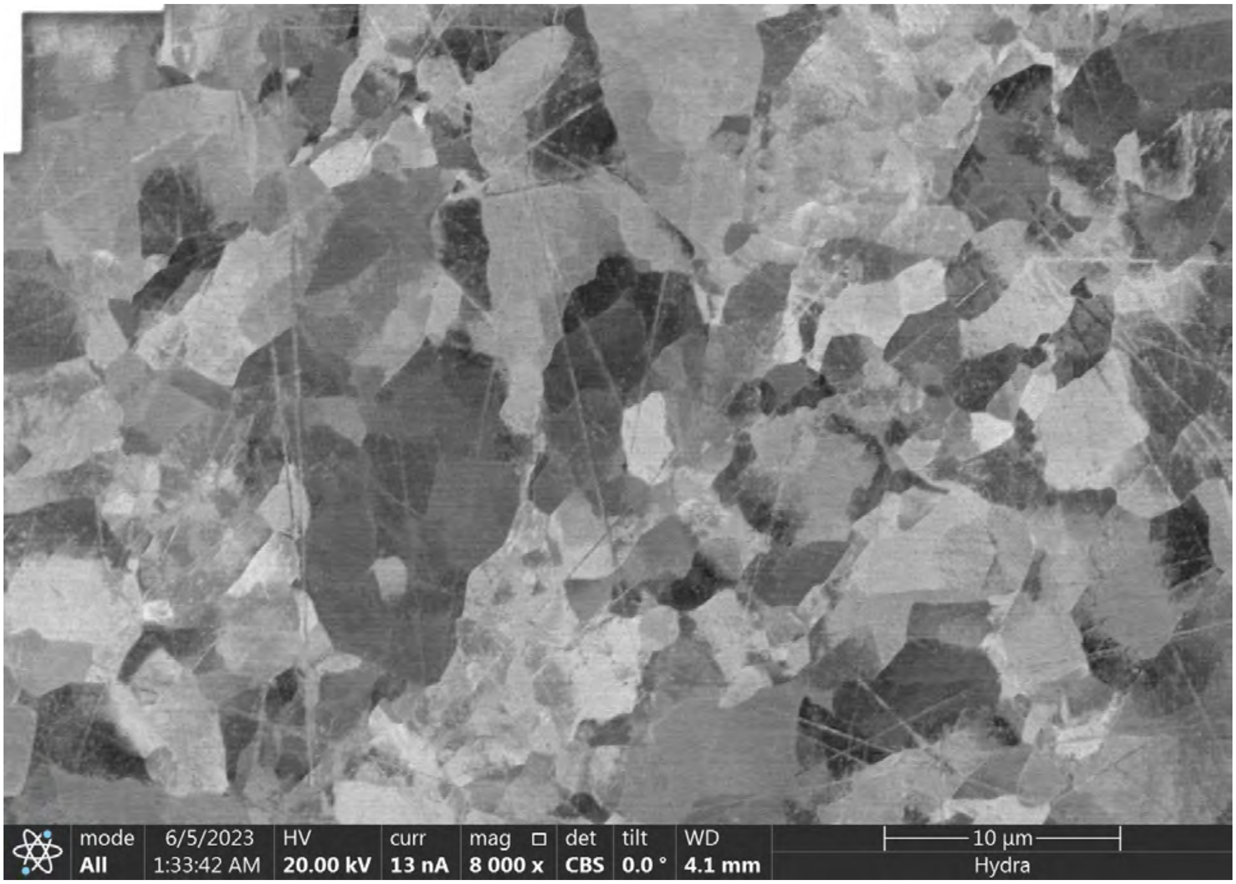

Figure 7.11. SEM Results at 8000 magnification for L02: Condition C10, Sample SS19

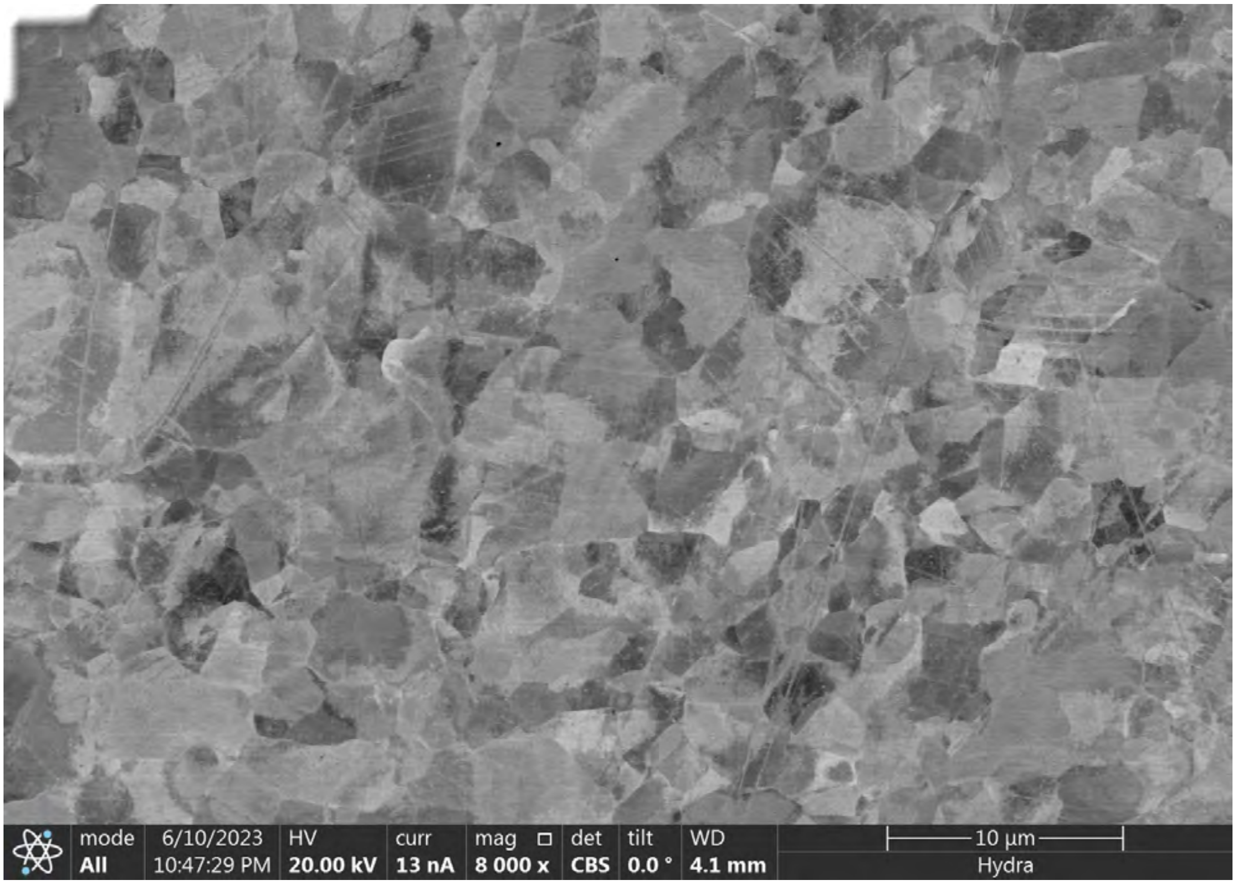

Figure 7.12. SEM Results at 8000 magnification for L02: Condition C11, Sample SS20

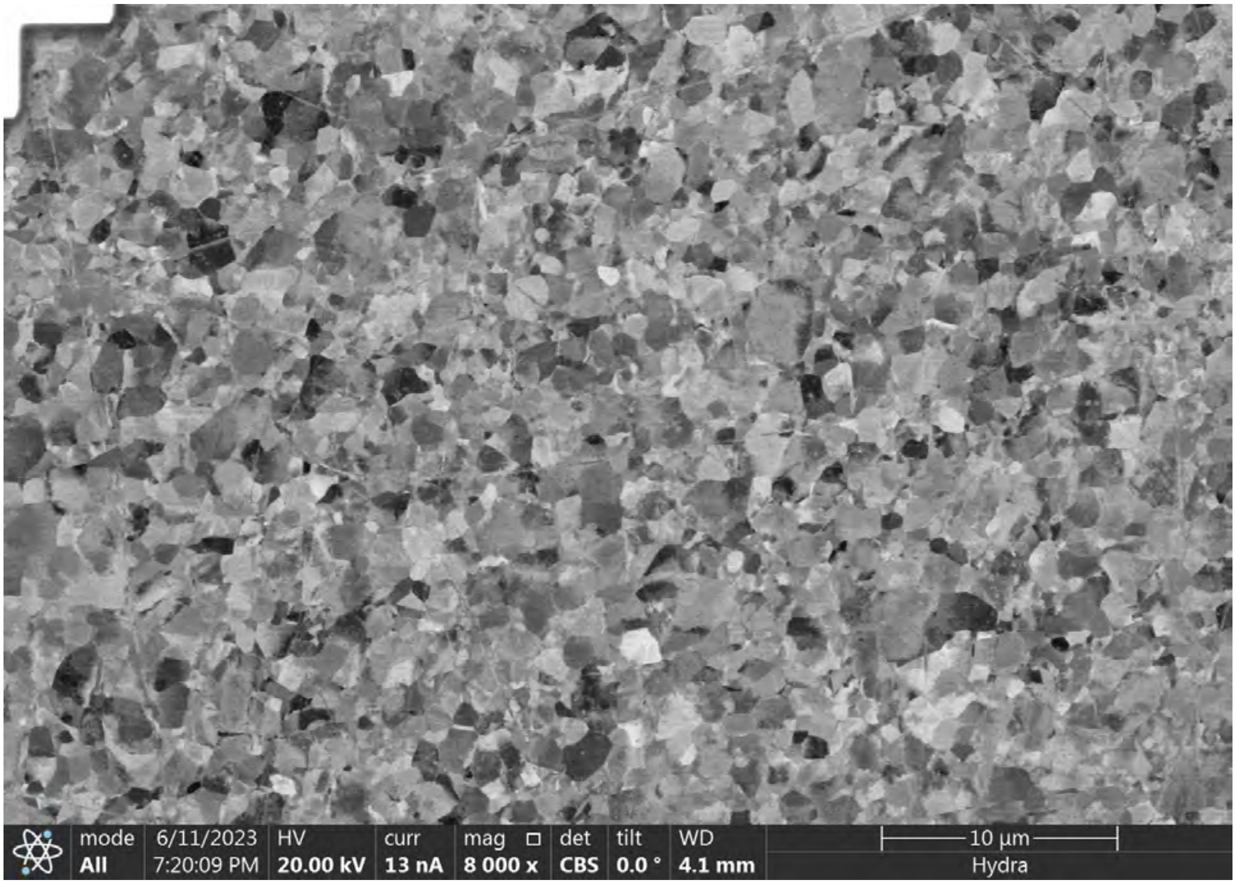

Figure 7.13. SEM Results at 8000 magnification for L02: Condition C06, Sample SS21

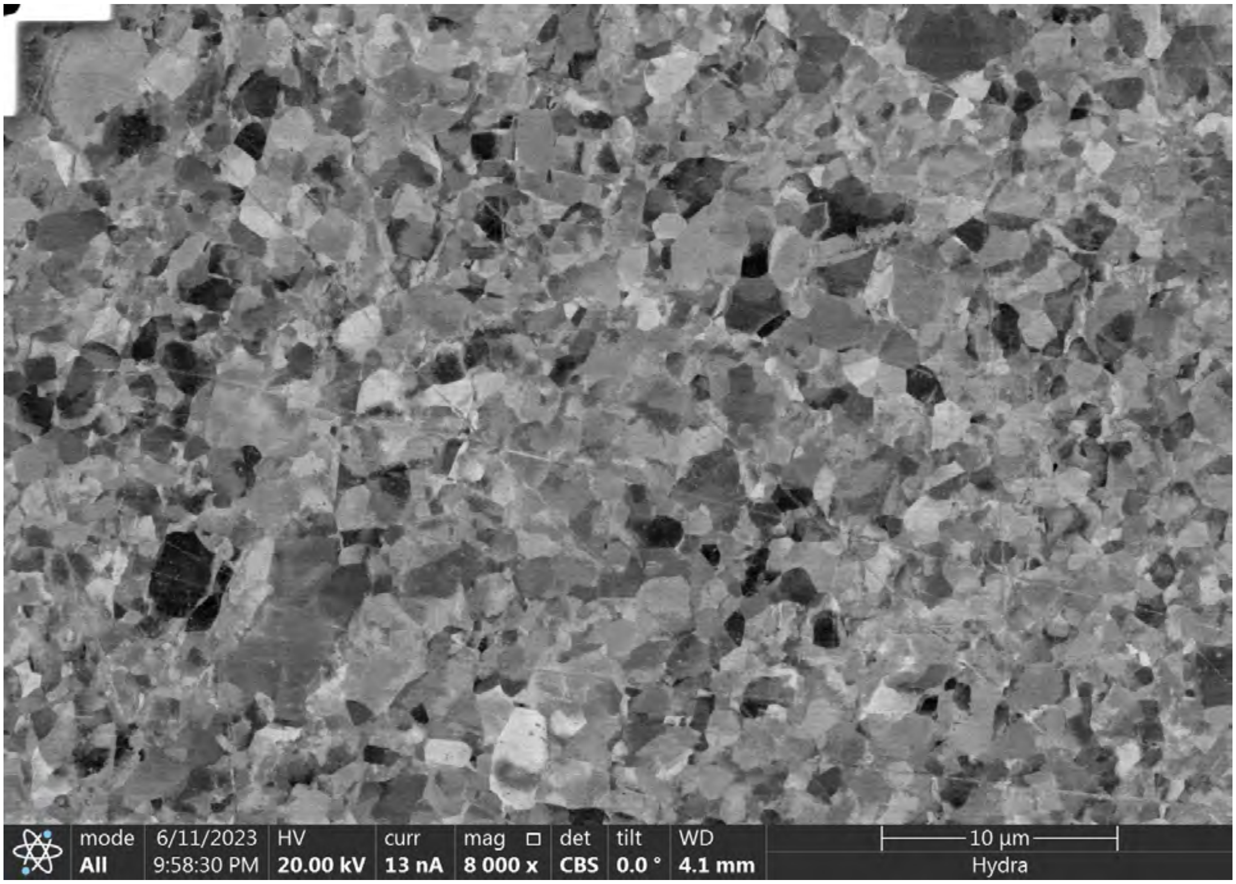

Figure 7.14. SEM Results at 8000 magnification for L02: Condition C07, Sample SS22

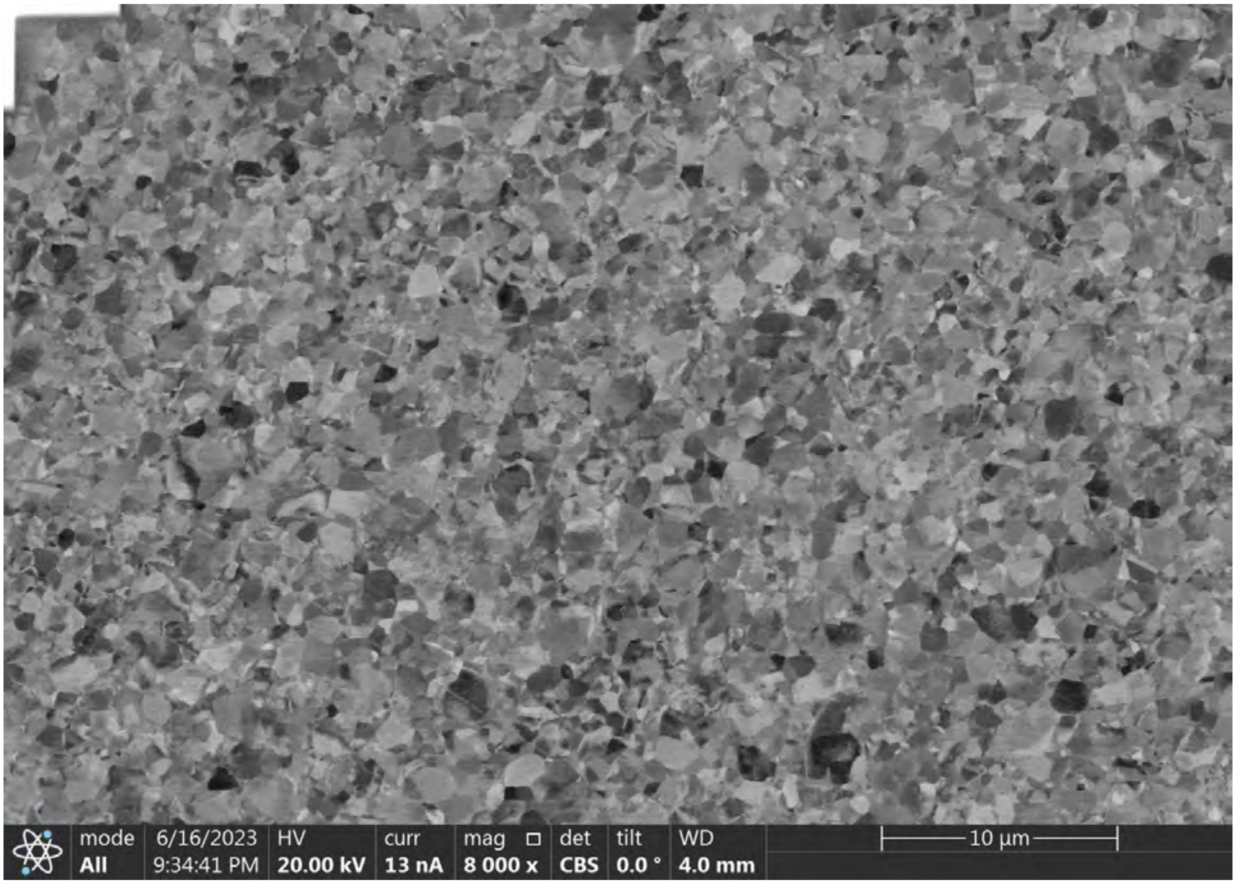

Figure 7.15. SEM Results at 8000 magnification for L02: Condition C03, Sample SS23

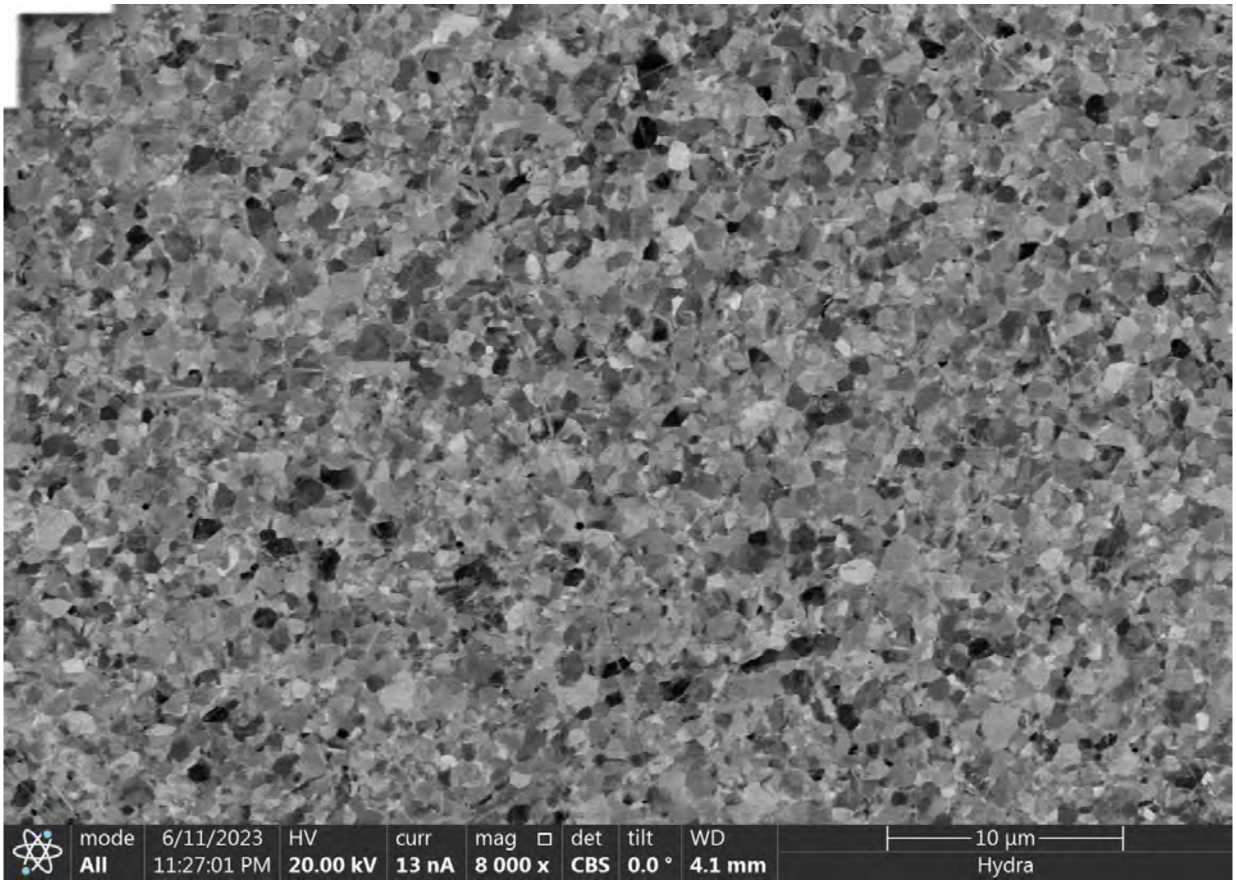

Figure 7.16. SEM Results at 8000 magnification for L02: Condition C01, Sample SS34

7.4 SEM with 15000x Magnification

SEM images at 15000x magnification are provided in this section. Two specimens were measured at this magnification.

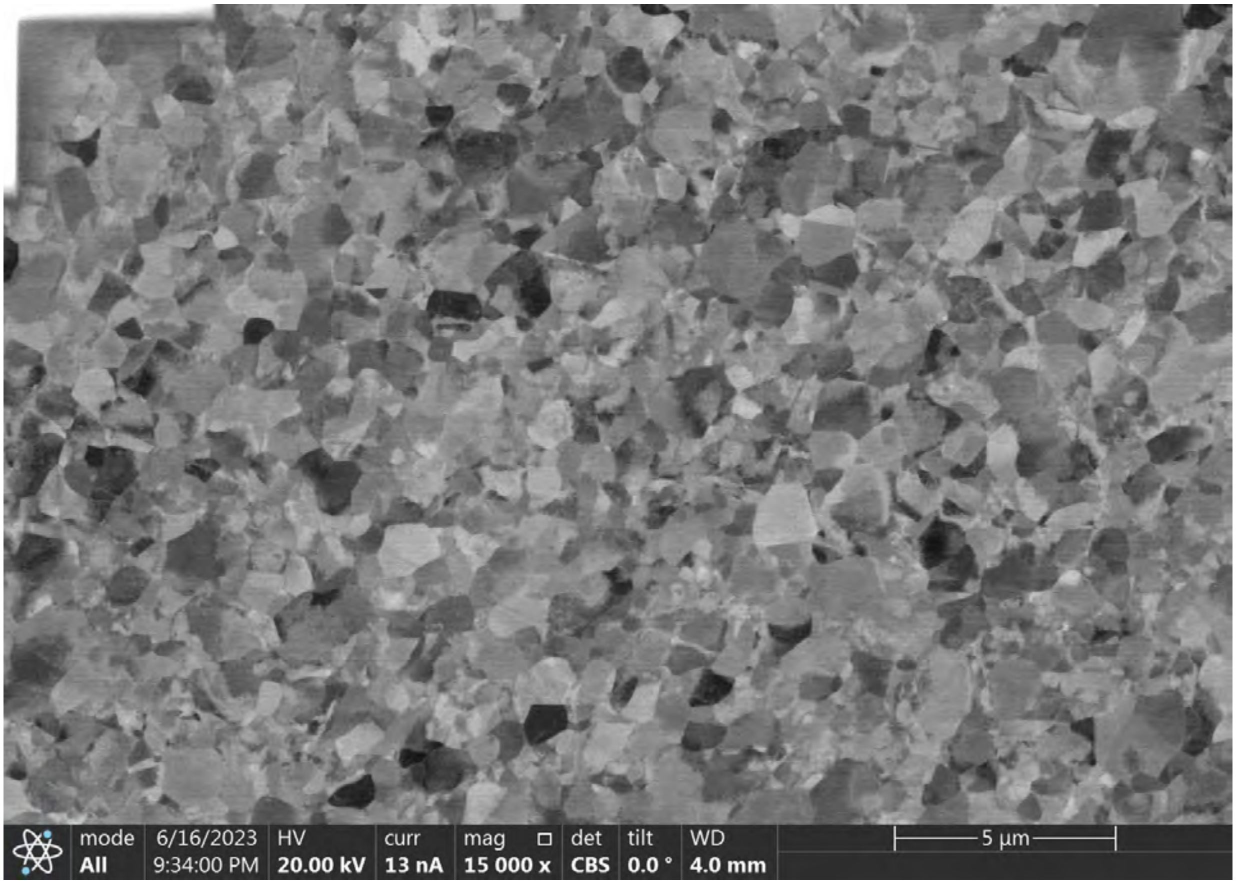

Figure 7.17. SEM Results at 15000 magnification for L02: Condition C03, Sample SS23

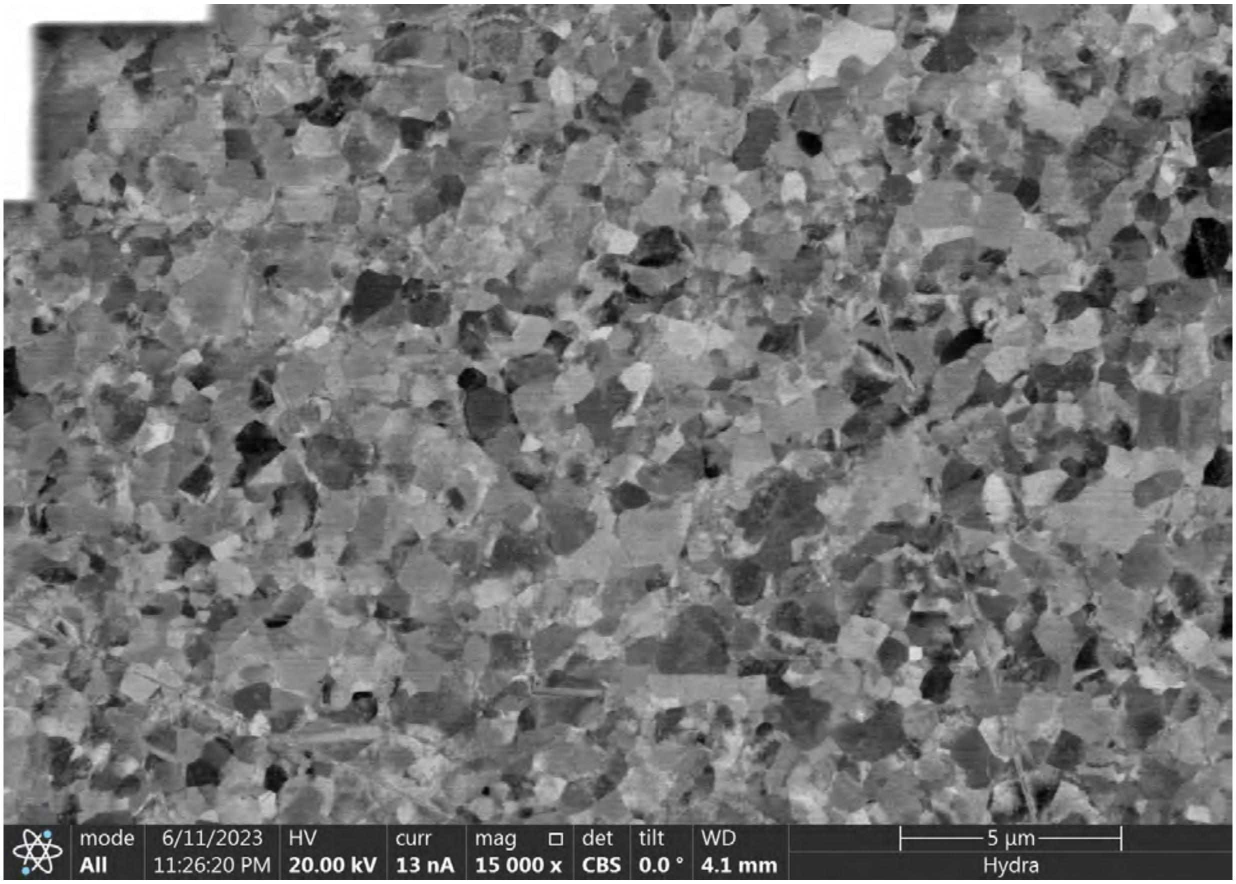

Figure 7.18. SEM Results at 15000 magnification for L02: Condition C01, Sample SS34

Pacific Northwest National Laboratory

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99354

1-888-375-PNNL (7665)

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Attachment E: MCPC Round 3 Material Characterization Results for Modality ULTRASONIC

Y Guo K Nwe
DR Todd N Conway

Attachment E contains 17 pages

Acknowledgments

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Contents

Figures

Tables

1.0 Introduction

The UT measurement technique uses ultrasonic waves to measure and analyze the properties of a medium. Ultrasonic attenuation and backscattering are used to infer material microstructure (specifically grain sizes) in the polycrystalline steel specimens through sensing changes in speed of sound of the anisotropic crystals. UT scans performed here produce two text-based file for each measurement. Data is copied manually from the instrument. The files contain ASCII text in comma separated values (CSV) format. The collected data represents time-averaged (over many pulses) amplitude versus measured time after each pulse. This collection is repeated at numerous discrete scan and index locations on the specimen. The process applied here is effectively a raster scan applied to the specimen top surface with information at each discrete location about potential defects and grain sizes collected for different depths at each discrete location. Measurements are made in two configurations.

The following sections provide a summary of results for this modality to enhance dissemination of the large volume of similar data that are made available via the PNNL DataHub (https://data.pnnl.gov) platform.

2.0 Key Information and Results

The main portion of this report provides a summary of key information related to the modality, including processing conditions, identification information, and the numerical results of most focus in the project. Where to find the information and results in the main report is described below (Section, Table, and Figure numbers listed below are found in the main report):

- Nominal process conditions for the FSP experiments are defined in Table 1 and Table 3 (data is in two different sorting orders).
- The Condition, Sample, and Specimen ID matrix is defined in Table 2 that is necessary to decode the nominal conditions applied to a particular specimen.
- Details about the modality data collection approach is defined in Section 4.0. This includes information about where ULTRASONIC is performed.
- Tabulation of the modality results per Sample ID is listed in Table 4 with discussion of what the particular result means discussed earlier in the section.
- UT figure of merit value versus nominal processing temperature listed in Table 3 and Table 4 is displayed graphically in Figure 5. This is shown to highlight consistency of results across a range of conditions.

3.0 Instrumentation

See Guo et al. (2024) (Reference provided in the main report) for details.

4.0 Summary of Results

Three types of data are archived; the raw ultrasonic scan data, the visualization of the raw data, and the ultrasonic figure-of-merit (FOM) results calculated from the grain noise data. Under the folder for each specimen, the raw ultrasonic scan data is in the DATA folder, the visualization is in the VISUALIZATION folder, and the FOM results are in the RESULTS folder. The DATA folder contains two CSV files for the front surface (FS) reference signal and the grain noise (GN) data, respectively. The VISUALIZATION folder contains image files visualizing the grain noise data in the form of B-Scan images. The RESULTS folder contains a CSV file that lists FOM values for a few different frequencies. See Guo, et al. (2024) for more details (the reference is provided in the main report).

5.0 Results Files

5.1 DATA Directory Contents

Data files obtained for each dataset from the instrumentation are saved in the associated DATA directory. For ultrasound, two data files are generated for each specimen: a CSV text file containing an ultrasonic waveform later used as a reference signal in data analysis, and a CSV text file containing all the ultrasonic grain noise waveforms for the raster scan later used to calculate ultrasonic FOM.

- **(name)_FS.csv** (CSV Text type): When the probe is in the normal incidence configuration (oriented directly downward at the specimen), only a single front surface (FS) echo is acquired that is a reference signal for later analysis.

- **(name)_GN.csv** (CSV Text type): When the probe is in the transverse wave grain noise (GN) configuration, a full raster scan is performed. This orientation is at roughly 22 degrees from the top surface of the specimen to produce a transverse wave.

5.2 VISUALIZATION Directory Contents

Visualizations are derived directly from data to illustrate or interpret results. Different colors, scales, magnifications, file formats (images vs animation) etc. may be present. The following visualizations are provided:

- **(name)_GN** (TIFF Image type, MP4 Movie type, Directory): A collection of imagery and animations of the grain noise data. The TIFF is a multipage object. The embedded imagery in the TIFF is also provided in the directory for easy import of a single image into reports and presentations.

The most useful data across multiple datasets is captured in directory ENSEMBLE-INFO for this modality. The information is provided for convenience and is directly replicated from the related datasets. Refer to the individual datasets for details. In the case of conflicting information between items found in this directory and with individual datasets, the individual dataset information takes precedence

- ULTRASONIC_Values.csv: Contains a summary of all ultrasonic data from across all the datasets as a convenience. This data is assembled from the individual dataset files.

6.0 Notes and Comments

The following observations are made about data and files for this modality:

- None.

7.0 Tabular Results

Figure of Merit (FOM) is listed Table 7.1 along with the intercept-based grain size in microns (these are the same values shown in Table 4 of the main report). This data is obtained from the (name)_FOM.csv file. The file contains additional tabular FOM values at different frequencies obtained by analyzing the grain noise data.

Table 7.1. Listing of all ultrasonic FOM data.

Sample ID	Condition ID	Mean Grain Size (um)	FOM at 20.5 MHz
SS17	C08	3.94	0.2824
SS18	C09	3.04	0.2136
SS19	C10	9.62	0.5772
SS20	C11	5.94	0.4249
SS21	C06	1.92	0.0855
SS22	C07	2.50	0.1099
SS23	C03	1.38	0.0504
SS34	C01	1.04	0.0443

8.0 Graphics

The following sub sections display key graphics for convenience:

8.1 Grain Noise at Stir Zone and Its Immediate Transition Area

A grain noise B-Scan image of the transverse cross-section at index 25, selected from the {name}_GN-Color folder, is shown below for each condition and sample as an example.

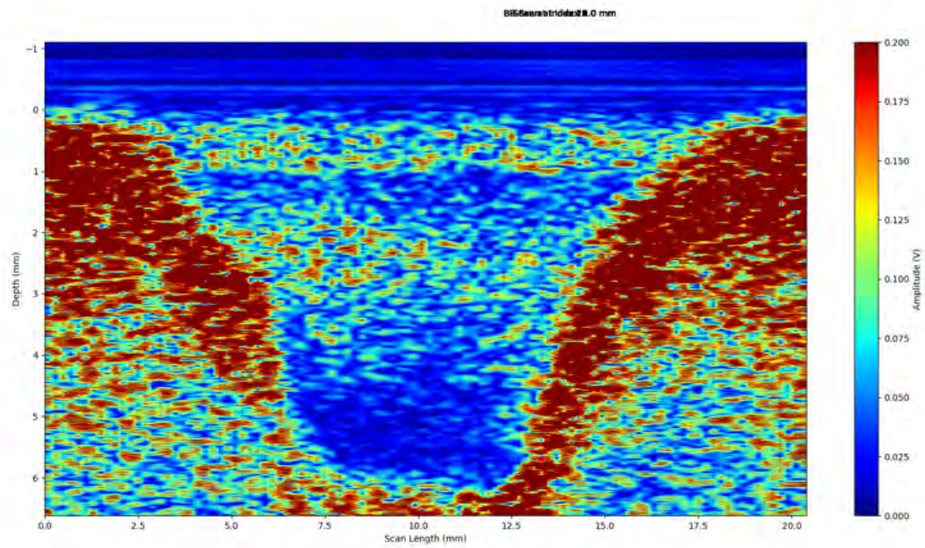

Figure 8.1. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C08, Sample SS17

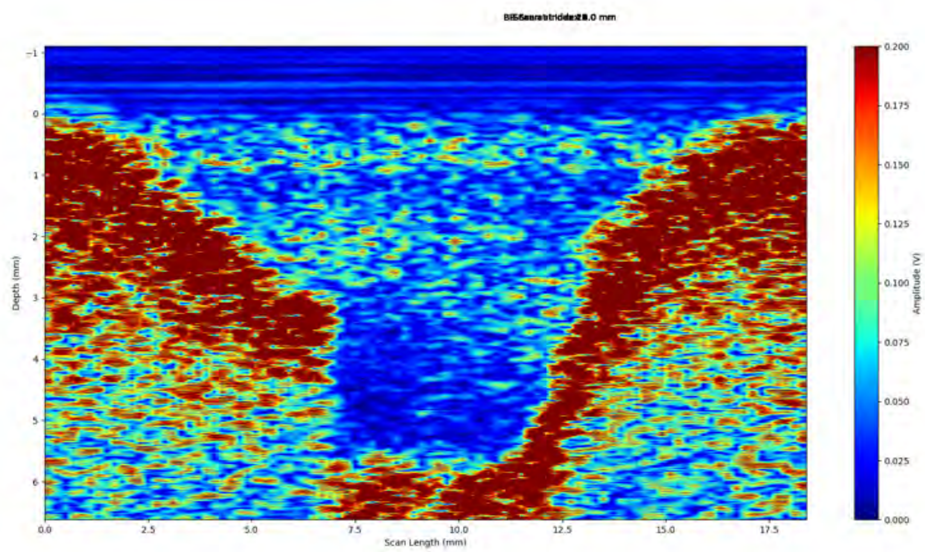

Figure 8.2. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C09, Sample SS18

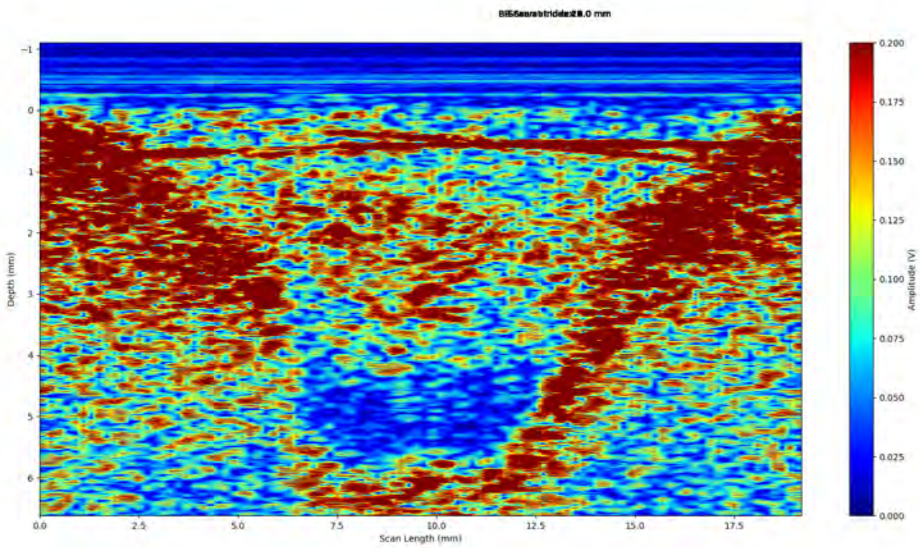

Figure 8.3. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C10, Sample SS19

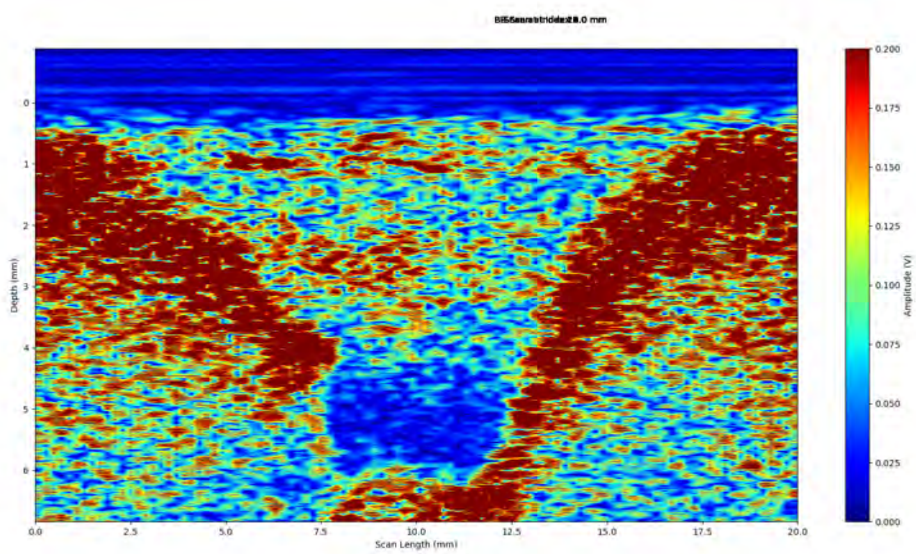

Figure 8.4. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C11, Sample SS20

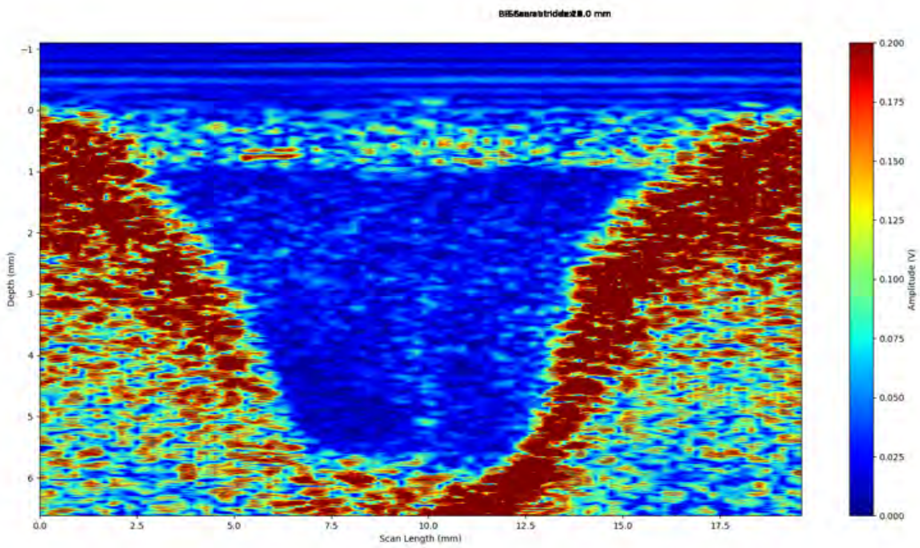

Figure 8.5. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C06, Sample SS21

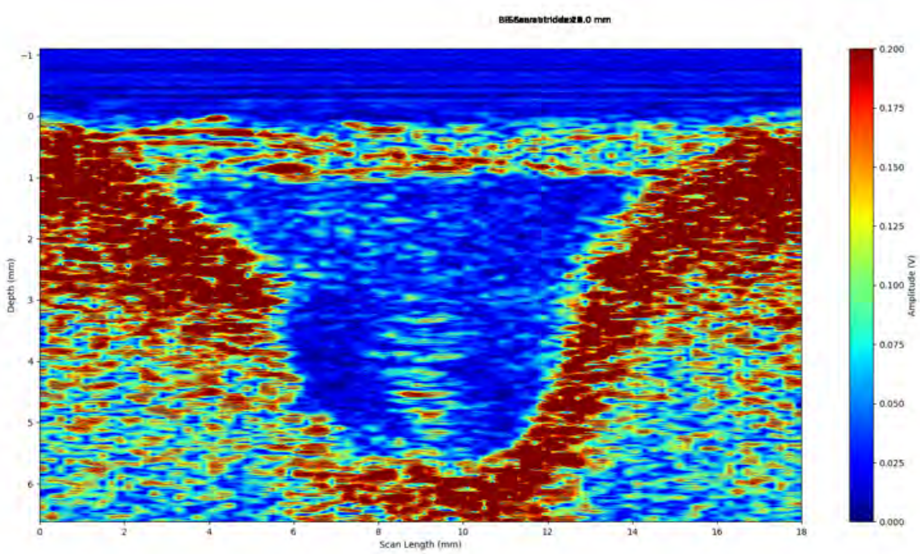

Figure 8.6. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C07, Sample SS22

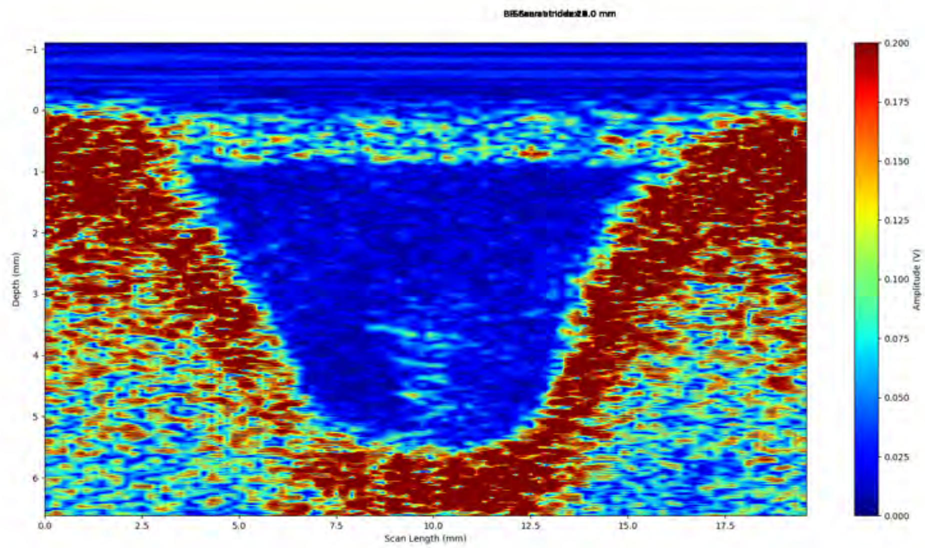

Figure 8.7. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C03, Sample SS23

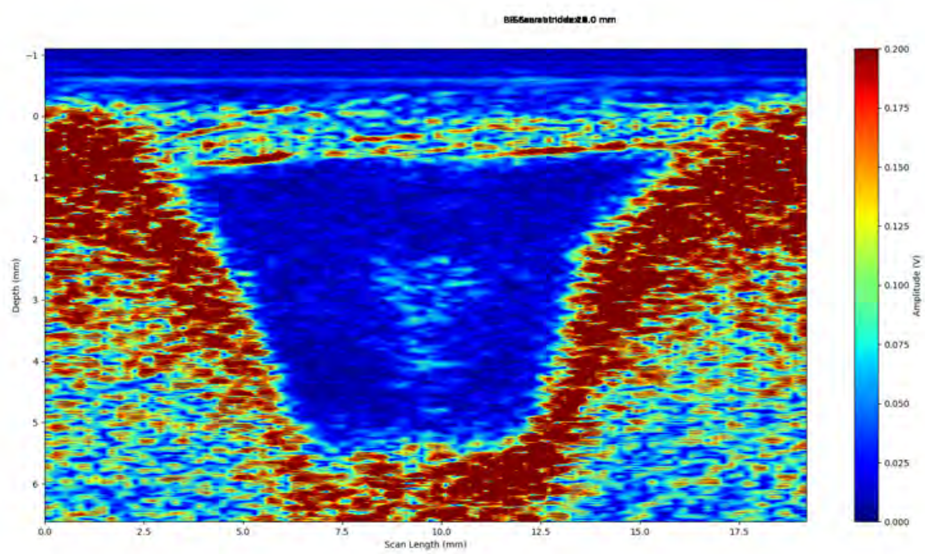

Figure 8.8. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C01, Sample SS34

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P.O. Box 999
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