Pacific Northwest
NATIONAL LABORATORY

PNNL-36768, Rev 0

Materials Characterization, Prediction, and Control Project

Summary Report on Material Characterization, Part 1

September 2024

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U.S. DEPARTMENT OF ENERGY

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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

Contents

Figures

Tables

Attachments

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 utilizing across 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 1 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:

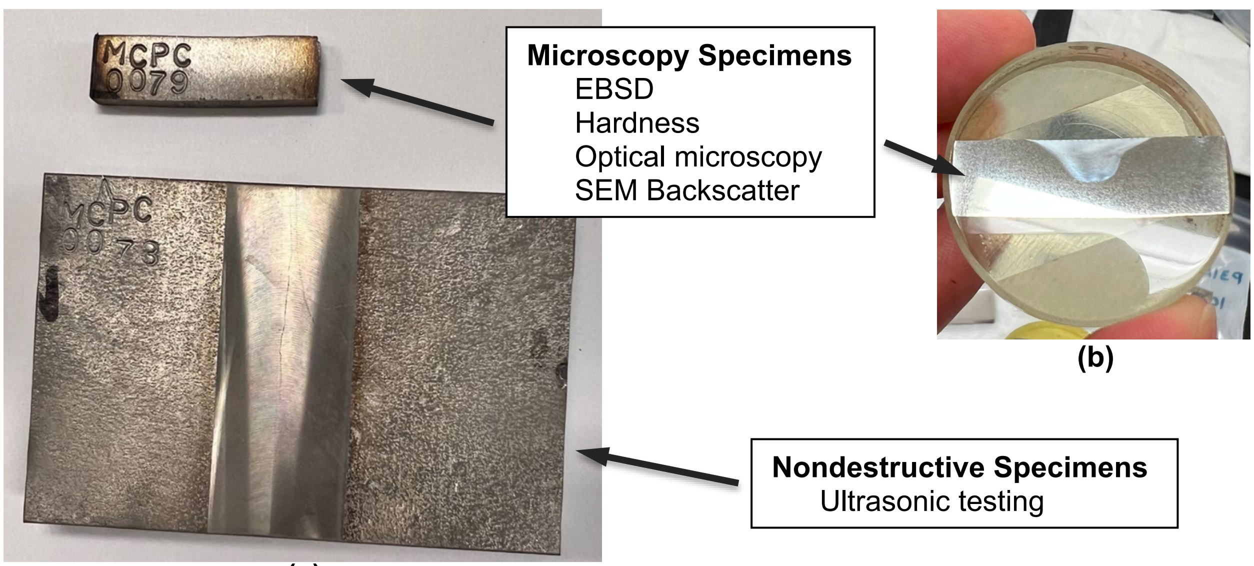
- Section 3: Sample identification and naming
- Section 4: Material characterization modalities
- Section 5: Example product metrics from material characterization
- Section 6: Structure of electronic modality data
- Attachment A: Material Characterization Results for Modality EBSD
- Attachment B: Material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



(a) (b)

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	Optical and Hardness Specimen ID	SEM and EBSD Specimen ID	UT Specimen ID
C00	SS36	MCPC0176	MCPC0177	MCPC0178
C01	SS31	MCPC0151	MCPC0152	MCPC0153
C02	SS28	MCPC0136	MCPC0137	MCPC0138
C03	SS01	MCPC0001	MCPC0002	MCPC0003
C04	SS32	MCPC0156	MCPC0157	MCPC0158
C05	SS02	MCPC0006	MCPC0007	MCPC0008
C06	SS03	MCPC0011	MCPC0012	MCPC0013
C07	SS04	MCPC0016	MCPC0017	MCPC0018
C08	SS05	MCPC0021	MCPC0022	MCPC0023
C09	SS06	MCPC0026	MCPC0027	MCPC0028
C10	SS07	MCPC0031	MCPC0032	MCPC0033
C11	SS08	MCPC0036	MCPC0037	MCPC0038

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 1 was based on obtaining a wide-view of data with measurements across a large region of each sample to produce a broad understanding of the nature of the samples. And in the case of optical microscopy, different etching was applied to select samples to confirm the most favorable approach. 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 primary optical microscopy result was obtained for etched samples using a combined ocular and magnification level of 100x that focused on the FSP-affected region. A single montaged image was provided. Additionally, wider view imagery with 50x magnification was provided for most samples. In select cases, Differential Interference Contrast imagery was also obtained.
- Microhardness testing was performed for all samples using the hardness tester typically used for testing. Data from a new tester was obtained for one repeat measurement to demonstrate the equivalent performance of the new and existing instruments. The manufacturer and model number were captured in metadata associated with individual datasets. Measurements were performed on a 0.5 × 0.5 mm grid for most samples. Data for two samples was obtained on a finer 0.2 × 0.2 mm grid.
- Measurements with SEM were performed on a relatively low magnification that revealed the gross view of the FSP-affected region. The magnification level resolved grain structure in unprocessed material but was not sufficient to resolve individual grains within the FSP-affected region. Results were provided in a montaged image as well as individual image tiles used in the construction of the montage.
- A total of eleven different measurements (e.g., panels) labeled as L01-L11 were obtained for EBSD. Five of the locations were well inside the boundary of the FSP-affected region. Some were within or near the base material, and several were in the transition region between the processed region and the base material. An example of the grid of locations is shown in Figure 2. The values in parentheses in this image indicate the distance in millimeters from the estimated center of the FSP-affected region (with negative values occurring in the upper left quadrant). See Attachment A for a discussion of numerical data for the spatial location. The primary focus of MCPC Project efforts was within the FSP-affected region; therefore, location L02 is generally used as the key location for sharing and interpreting results. Data from this location is the focus of this report.
- 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 Guo et al. (2024) describing this work for details.

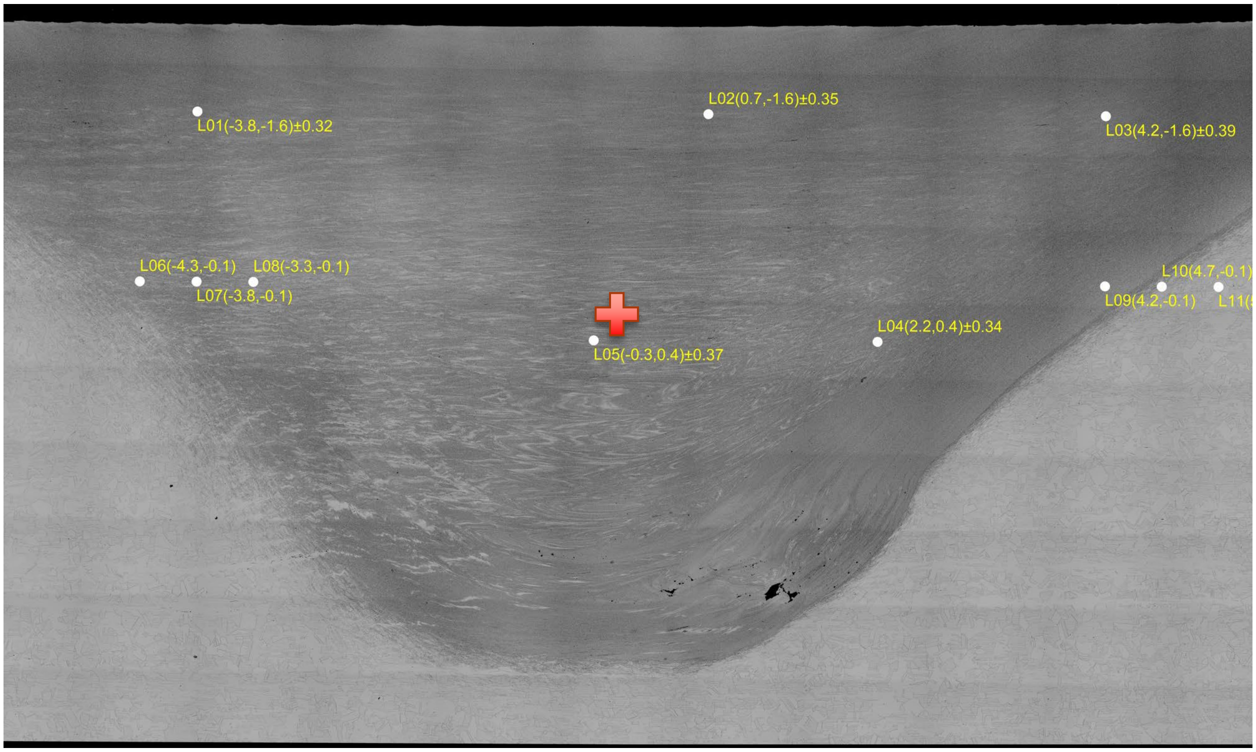


Figure 2. Example EBSD measurement locations for Sample ID SS32.

Spatial registration information was provided in metadata across several modalities for individual datasets sufficient to locate the approximate center of the FSP-affected region (approximated with a red "+" in Figure 2) and describe the location within the region in millimeters. The center location was defined from the geometric center in width (x) and height (y). Negative x- and y-locations correspond to the upper left quadrant of the figure, consistent with the ascending arrangement of pixels in a typical optical image.

Note that the location of the FSP-affected region reported for hardness data was obtained through a Gaussian fitting process that discriminates the FSP-affected region from base material. The reported center found in metadata for the vertical distance of hardness data was a weighted center value, not the geometric center illustrated in Figure 2.

Spatial registration information for Round 1 is available in electronic data for EBSD, HARDNESS, SEM, and OPTICAL modalities that allows alignment of imagery across the modalities. For example, the illustrations for EBSD locations, as drawn onto OPTICAL imagery in Figure 2, were performed via automated software scripting as a demonstration of the spatial registration.

UT data was provided in physical dimensions of millimeters, and the coordinate system origin corresponded to the starting location of the UT scan—not the approximate center of the FSP affected region in Figure 2. The center location of the FSP affected region must be estimated in relation to the UT data's coordinate system origin if spatially-aligned comparison to other modality data is desired.

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 ID[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 technical references associated with UT data analysis for more 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)
C03	SS01	720	1.0	25.4	10500	46.7
C05	SS02	720	3.0	76.2	10500	46.7
C06	SS03	750	1.0	25.4	10500	46.7
C07	SS04	750	3.0	76.2	10500	46.7
C08	SS05	800	1.0	25.4	10500	46.7
C09	SS06	800	3.0	76.2	10500	46.7
C10	SS07	850	1.0	25.4	10500	46.7
C11	SS08	850	3.0	76.2	10500	46.7
C02	***SS28***	**720**	**0.5**	**12.7**	**10500**	**46.7**
C01	SS31	720	0.5	12.7	9000	40.0
C04	SS32	720	1.0	25.4	9000	40.0
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
C03	SS01	1.0	1.6	267	0.0552
C05	SS02	1.2	2.0	264	0.0730
C06	SS03	1.6	2.2	259	0.0964
C07	SS04	1.5	2.4	243	0.1292
C08	SS05	2.1	2.8	234	0.1634
C09	SS06	2.1	3.4	231	0.2211
C10	SS07	3.0	5.7	210	0.5405
C11	SS08	2.5	4.5	205	0.3971
C02	***SS28***	***0.8***	**1.3**	***305***	***0.0671***
C01	SS31	1.2	1.4	275	0.0519
C04	SS32	1.0	1.4	273	0.0471
C00	SS36	36.5	N/A	~150	N/A

- Sample ID SS28 is highlighted in ***Bold***. Inspection of this data suggests the FSP tool has broken—but was not dispositioned as such by the FSP experts after the experiment. This is reinforced in comparing results with the same nominal processing conditions in other rounds. Numerical results for this sample are valid but not representative of the nominal processing conditions. See the next section for illustration.
- UT data is not collected for 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 eleven 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 SS28 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.

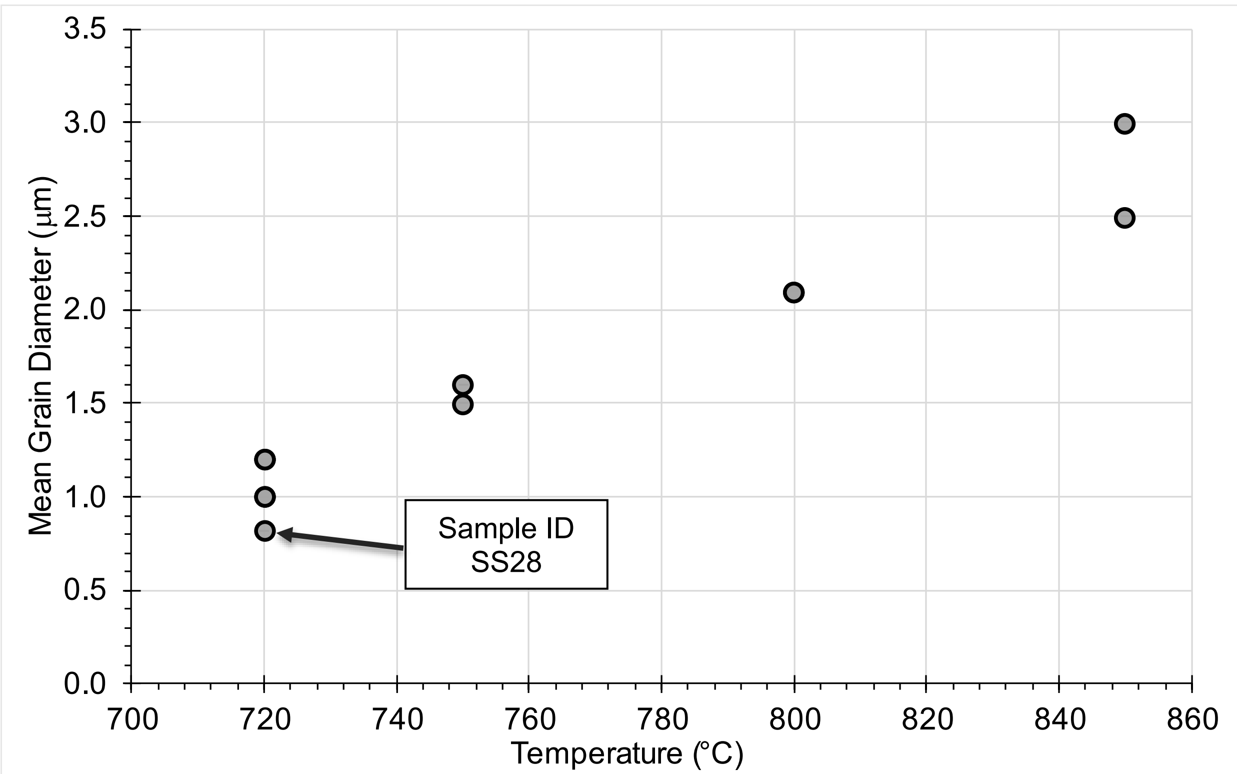


Figure 3. Circle-equivalent mean grain diameter versus nominal processing temperature.

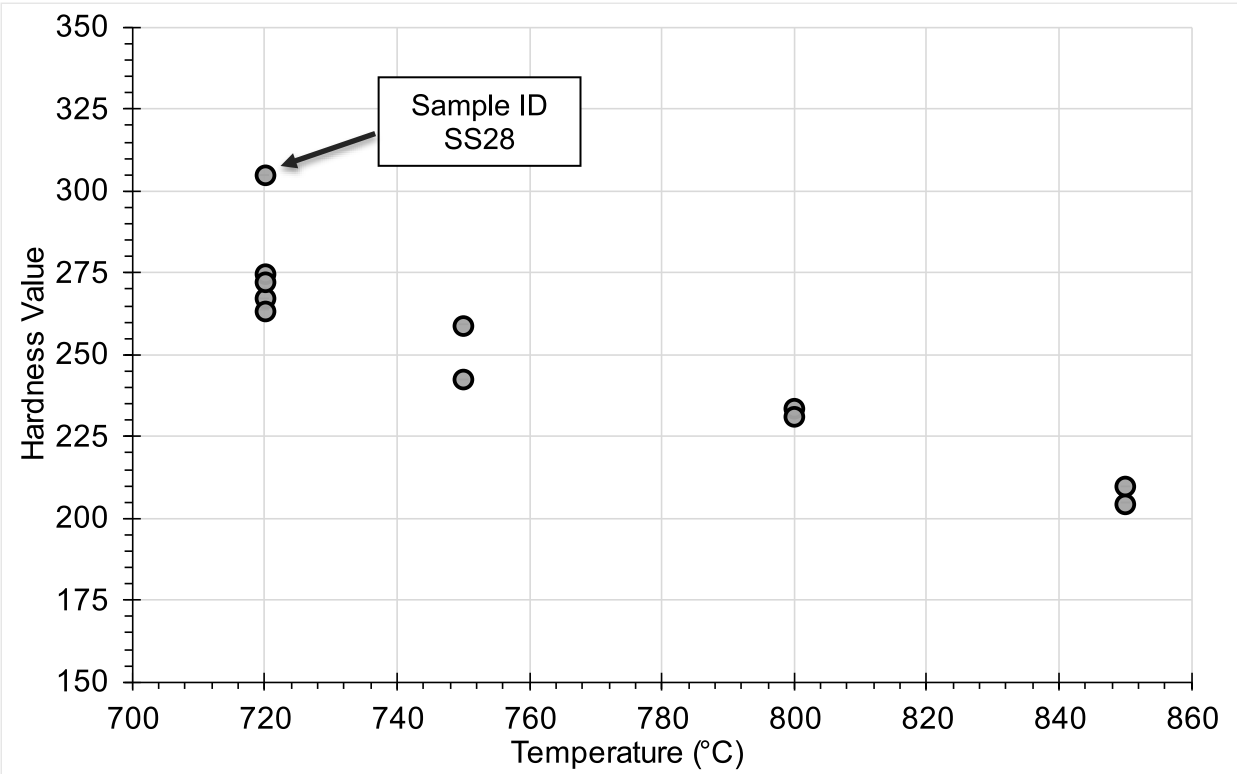


Figure 4. Hardness versus nominal processing temperature.

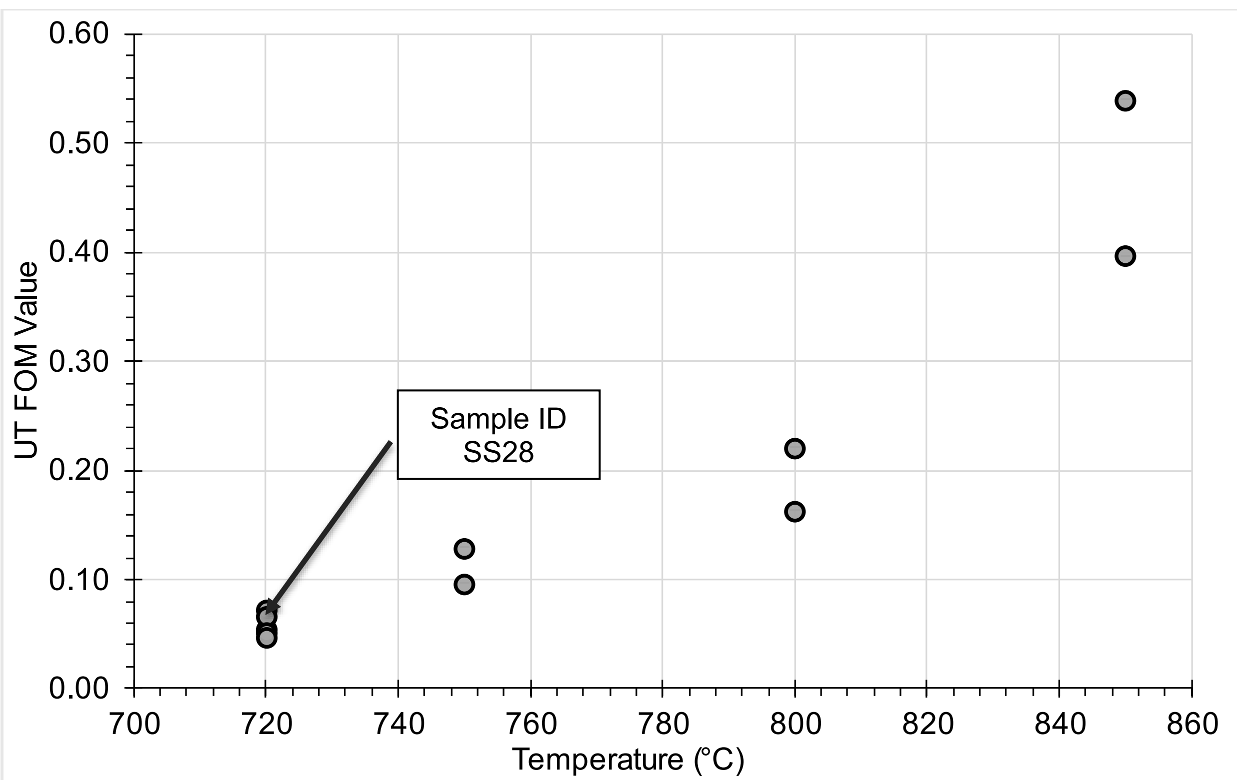


Figure 5. UT FOM nominal processing temperature.

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.

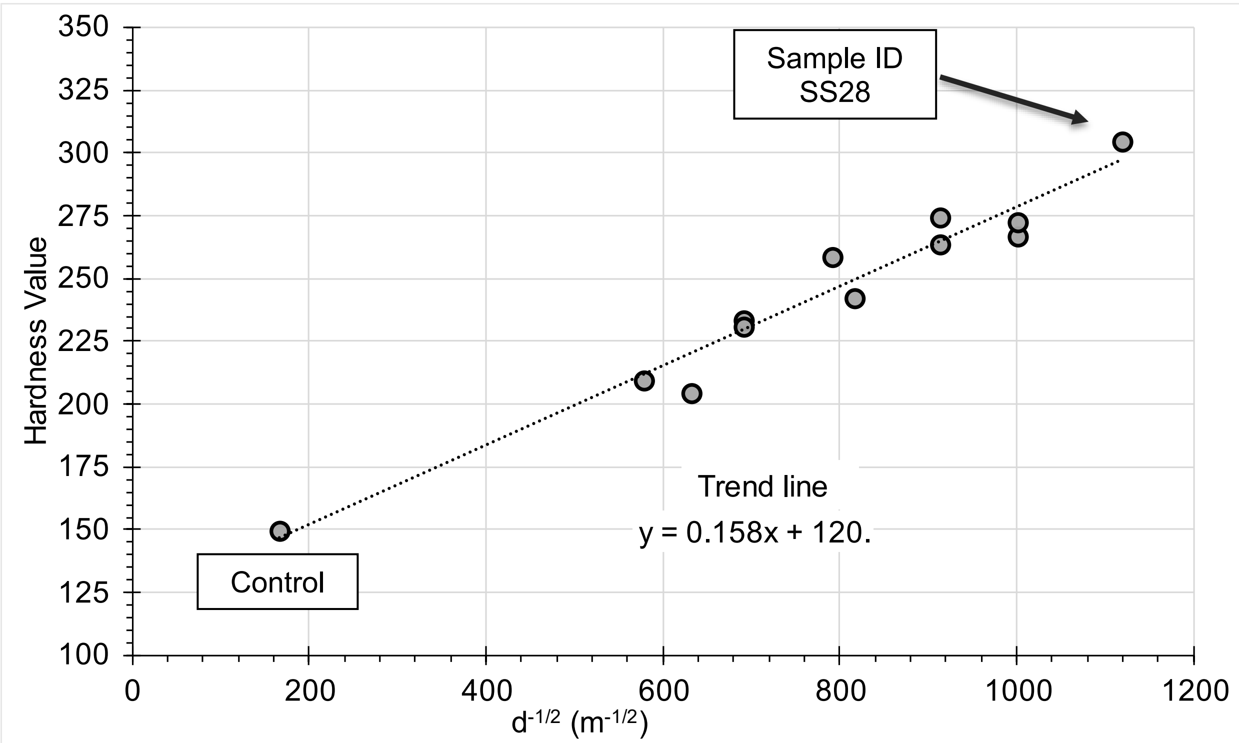


Figure 6. Hardness and mean grain size relationship.

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.

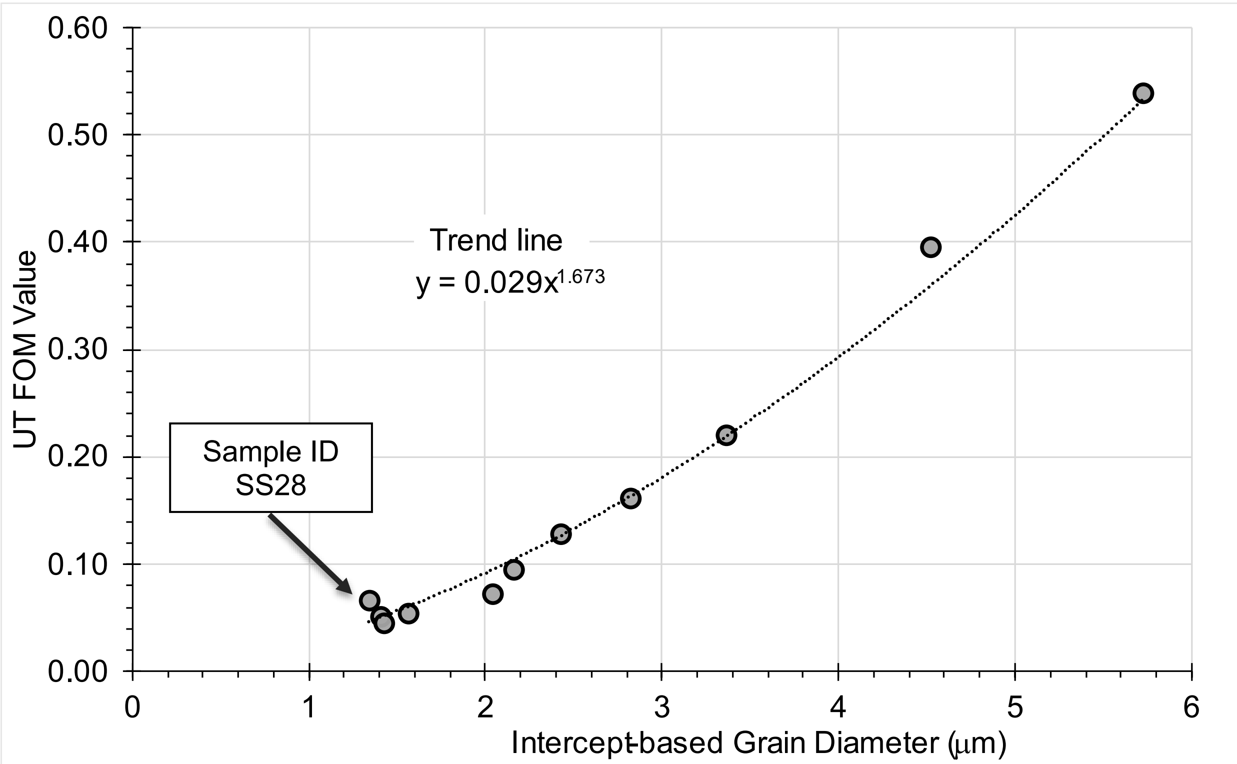


Figure 7. UT FOM versus intercept-based grain diameter.

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.

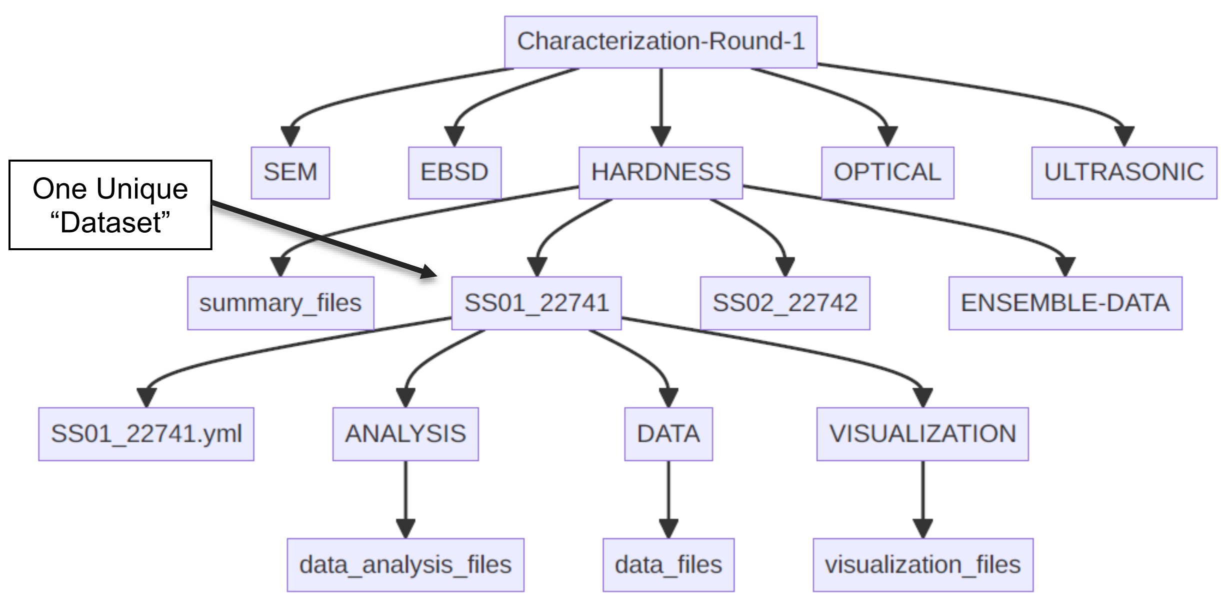


Figure 8. Illustration of typical file structure.

[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 SS01_22741). 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.

```
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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Attachment A: MCPC Round 1 Material Characterization Results for Modality EBSD

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K Nwe

Attachment A contains 42 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)

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 in the main report. The distinct locations were labeled L01 through L11. Each interrogation produced results files labeled with the related location code. The locations were listed in file ENSEMBLE-INFO/EBSD-Measurement-Locations and numerical values were provided in the parameter file for each dataset relative to the stir zone center. The locations were illustrated in optical imagery that is displayed in the section titled ”EBSD Collection Locations” in Attachment C.

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 (VISUALIZATION Directory, TIF Image): EBSD results image showing the band contrast image overlaid with grain boundaries identified through segmentation.
- 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.

5.0 Notes and Comments

Analysis of grain size and orientation is only provided for L01 through L05 in the ANALYSIS Directory. Other locations are not within the stir zone (they are located near interfaces with base metal) and were not analyzed in detail for grain information.

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

1. Data associated with MCPC0007_22734_L01.h5oina was collected at a different resolution than other data. This can be seen by inspecting step size information reported from metadata in file ENSEMBLE-INFO/EBSD-Metadata_All.xlsx archived with the data.

2. The grain boundary threshold angle used to calculate the reported circle equivalent diameters in the next section was set to 10°. However, the threshold angle used to calculate the grain boundary images shown later was 5°, or less in two cases. Therefore, any analysis to extract circle equivalent diameters from the grain boundary images will yield slightly smaller values than summarized in the tabular data.

3. Data for L06 through L11 are not carefully scrutinized for validity. At some locations the grain sizes are so large that only a few are visible. Refer to data for the control (SS36) for valid data of the unprocessed material structure.

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 for L01-L05.

Sample ID	L01	L02	L03	L04	L05
SS01	1.2	1.0	1.2	1.3	1.3
SS02	1.2	1.2	1.3	1.1	1.0
SS03	1.6	1.6	1.3	1.4	1.5
SS04	1.5	1.5	1.2	1.1	1.7
SS05	1.6	2.1	1.7	1.9	2.3
SS06	1.8	2.1	2.1	1.9	1.9
SS07	2.8	3.0	2.7	2.8	2.5
SS08	2.1	2.5	2.3	2.6	2.1
SS28	0.9	0.8	0.9	0.8	0.7
SS31	1.1	1.2	1.1	1.0	1.1
SS32	1.1	1.0	1.0	1.0	1.1
SS36	36.5				

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 figures show 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 for each location. The other results are available in file EBSD_Grain-Data-Results.xlsx. The figures show the grain boundaries identified through segmentation at the misorientation angle of 10° as indicated next to ”Threshold Angle” in each figure.

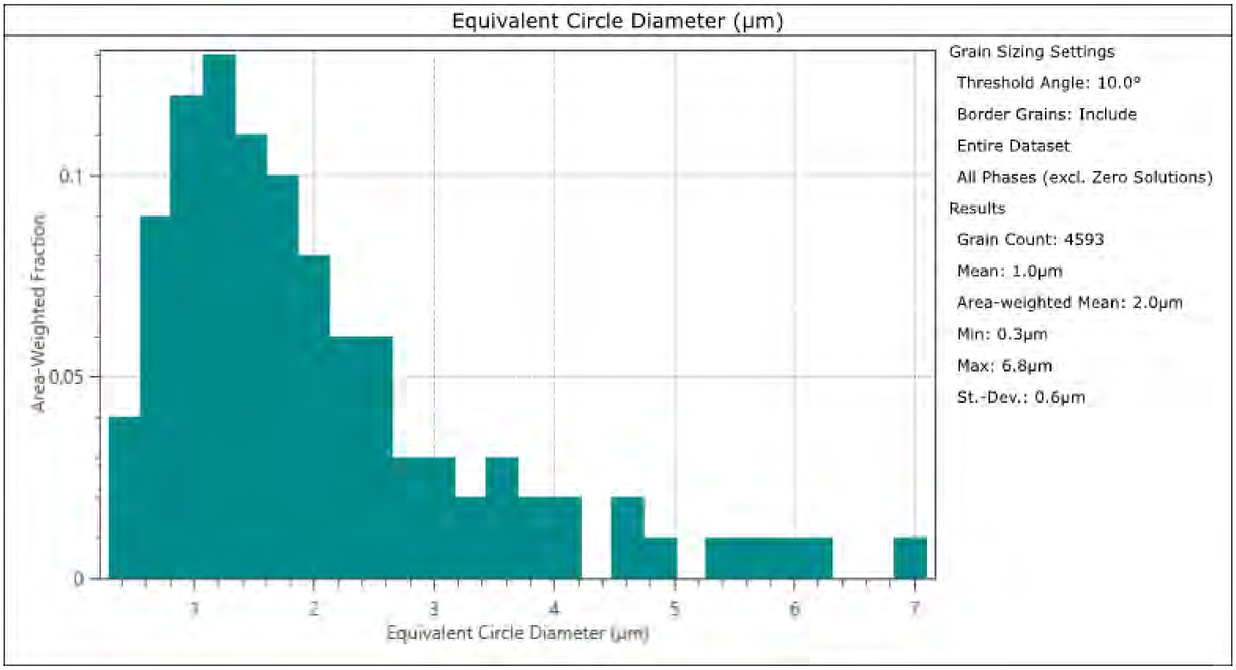

Figure 7.1. CED Grain Size and Other Data for L02: Condition C03, Sample SS01

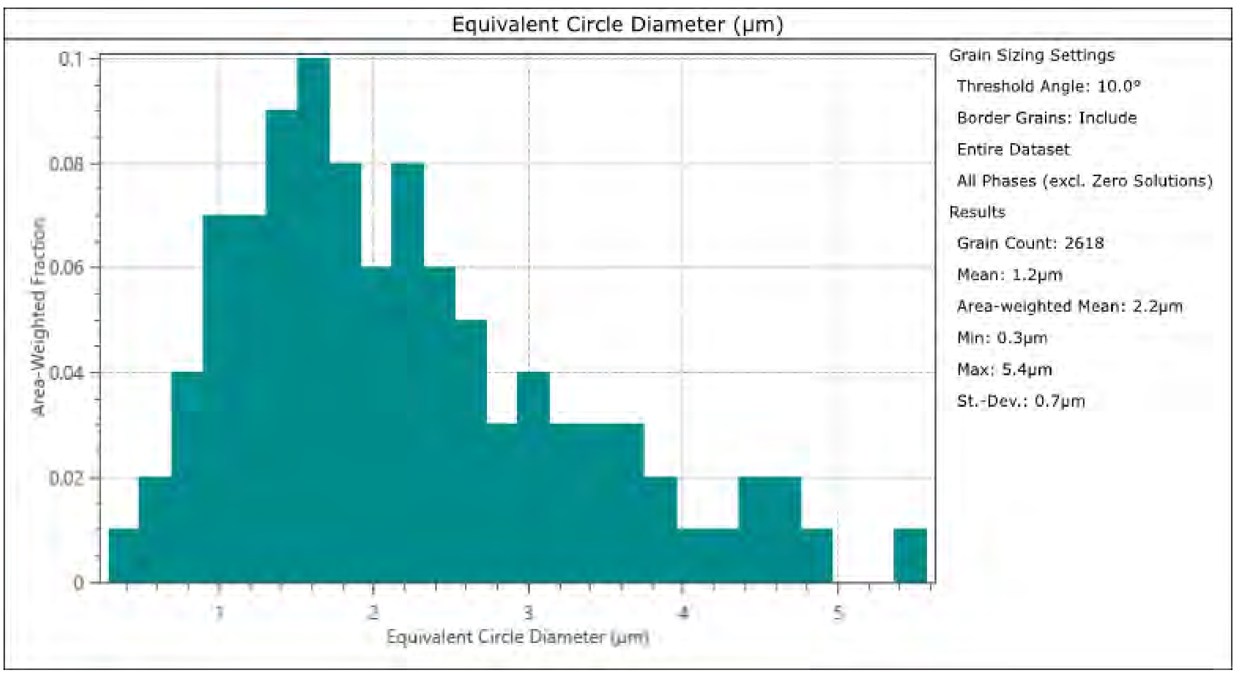

Figure 7.2. CED Grain Size and Other Data for L02: Condition C05, Sample SS02

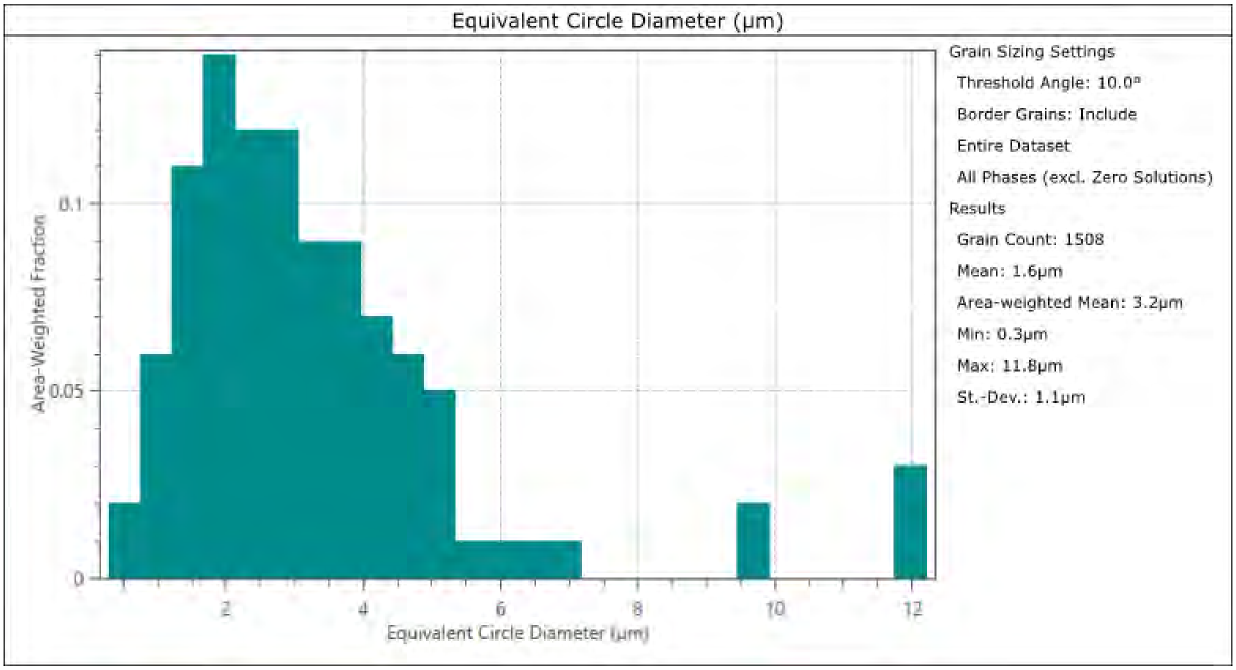

Figure 7.3. CED Grain Size and Other Data for L02: Condition C06, Sample SS03

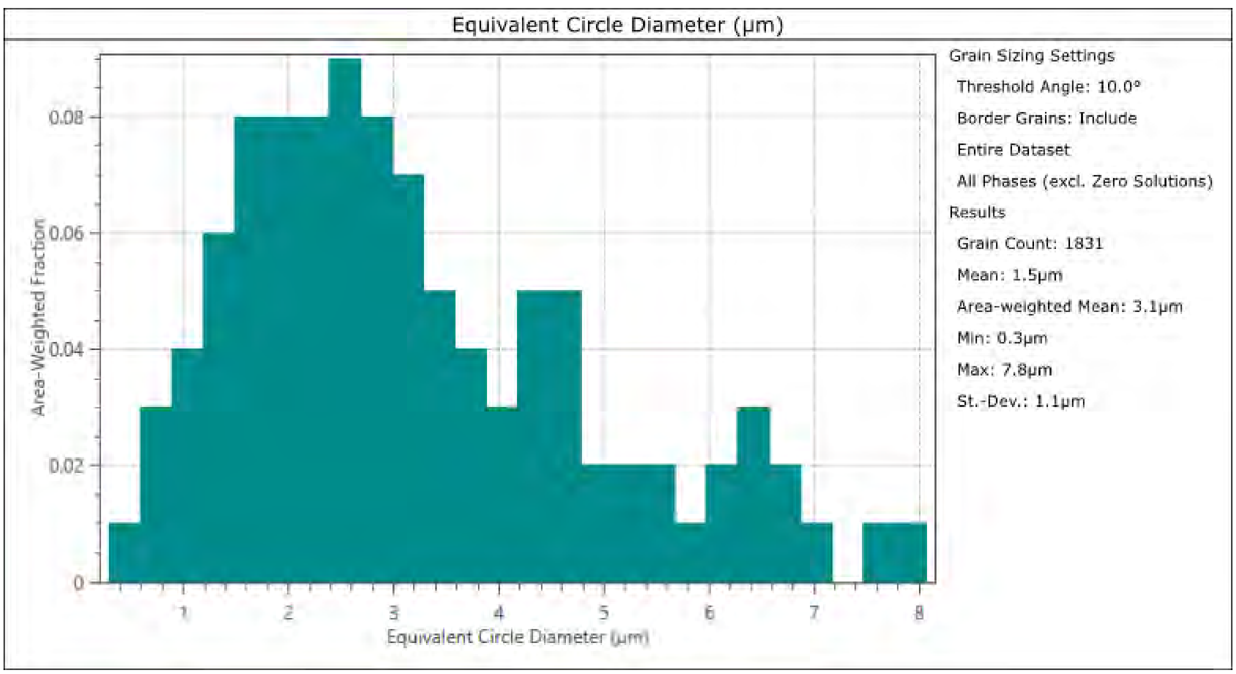

Figure 7.4. CED Grain Size and Other Data for L02: Condition C07, Sample SS04

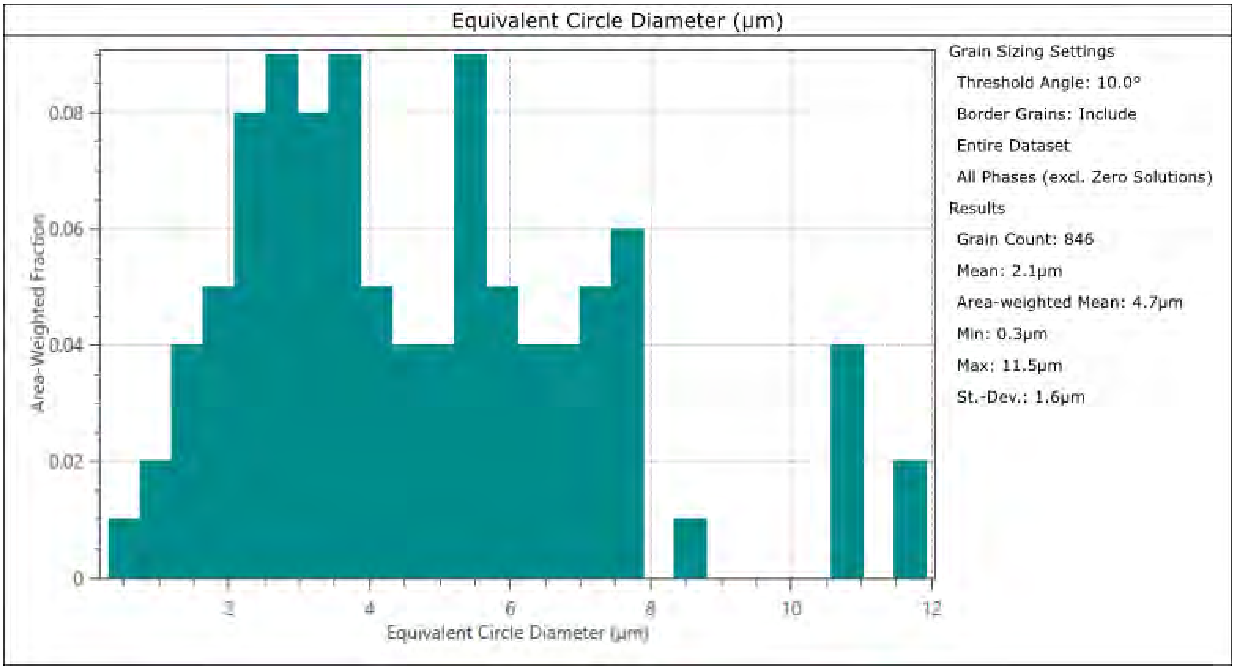

Figure 7.5. CED Grain Size and Other Data for L02: Condition C08, Sample SS05

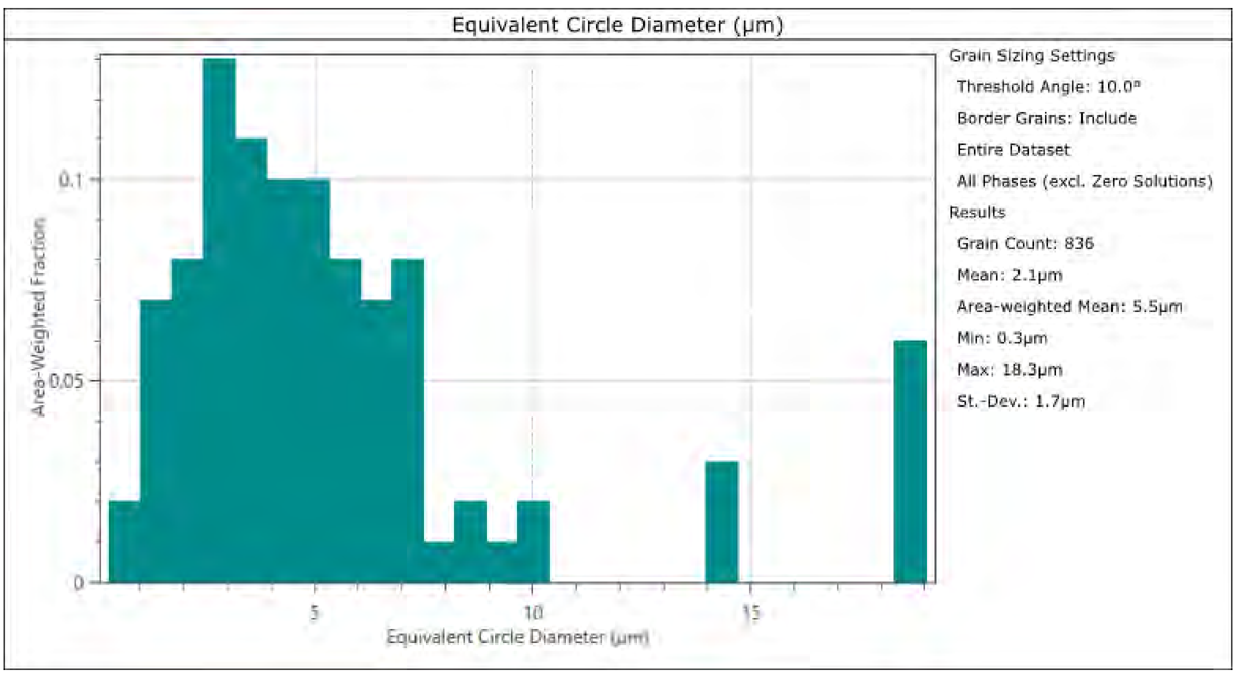

Figure 7.6. CED Grain Size and Other Data for L02: Condition C09, Sample SS06

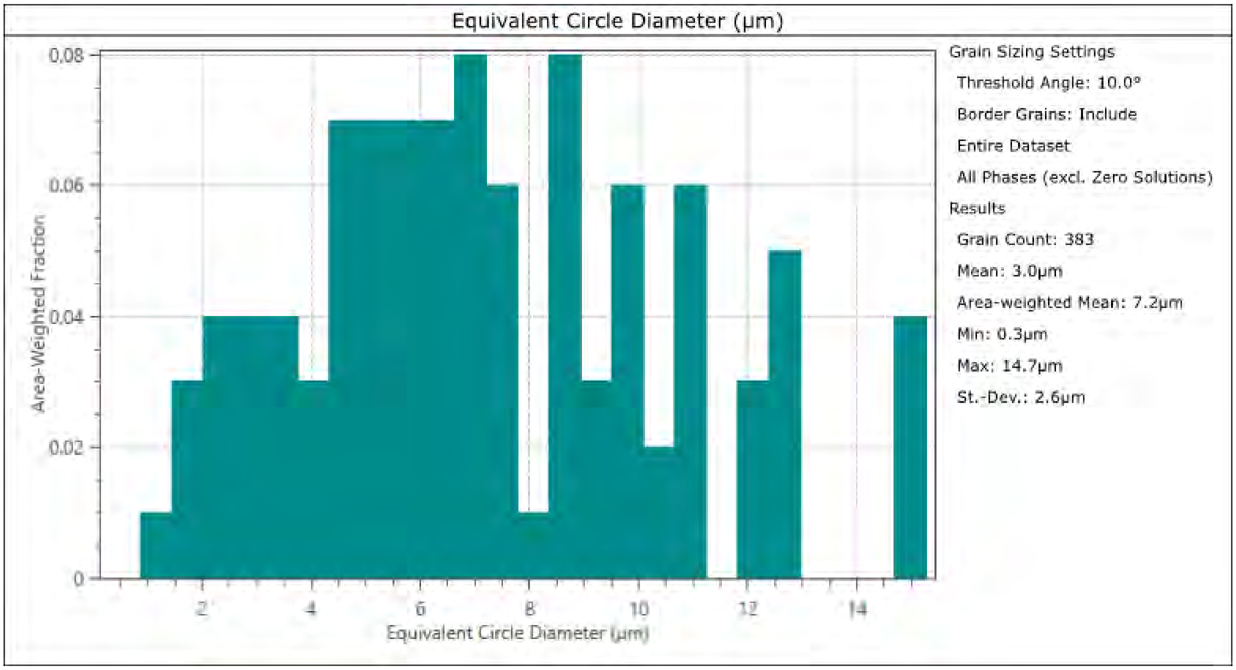

Figure 7.7. CED Grain Size and Other Data for L02: Condition C10, Sample SS07

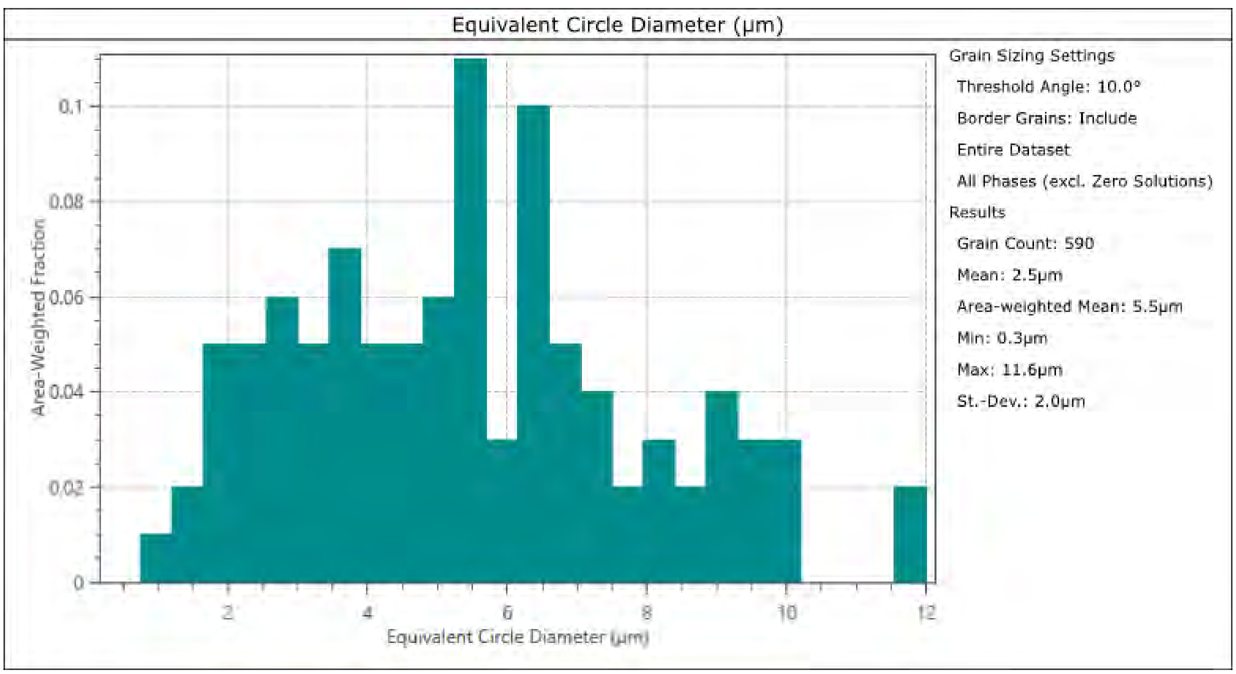

Figure 7.8. CED Grain Size and Other Data for L02: Condition C11, Sample SS08

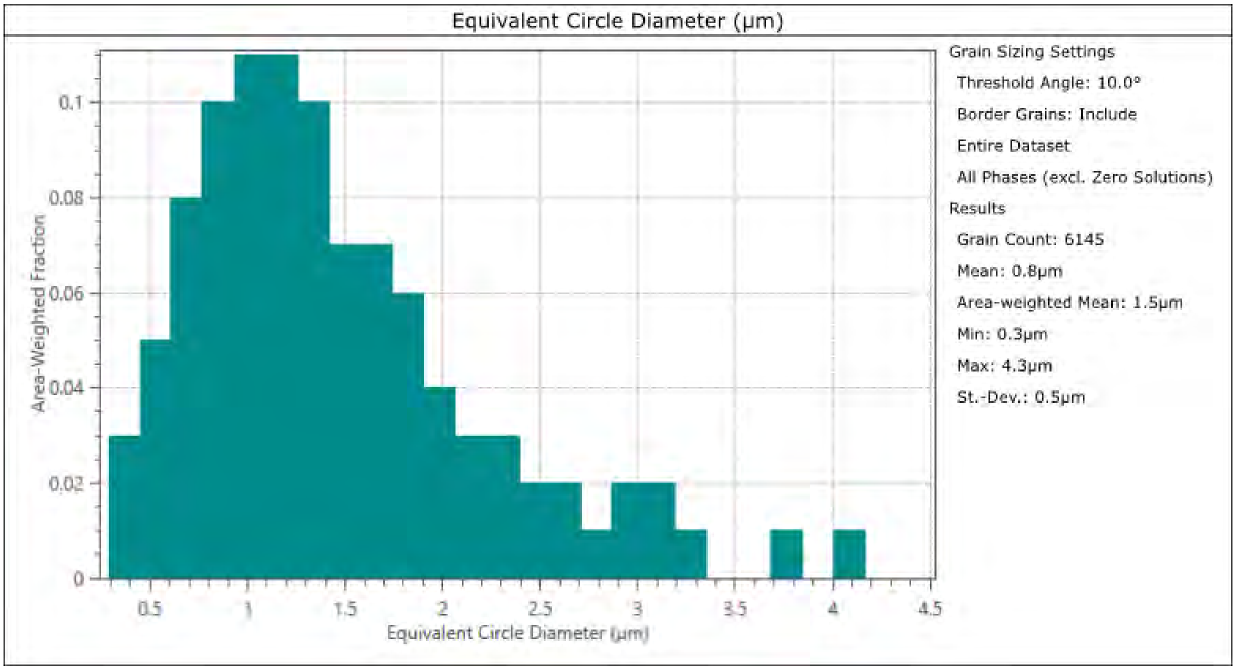

Figure 7.9. CED Grain Size and Other Data for L02: Condition C02, Sample SS28

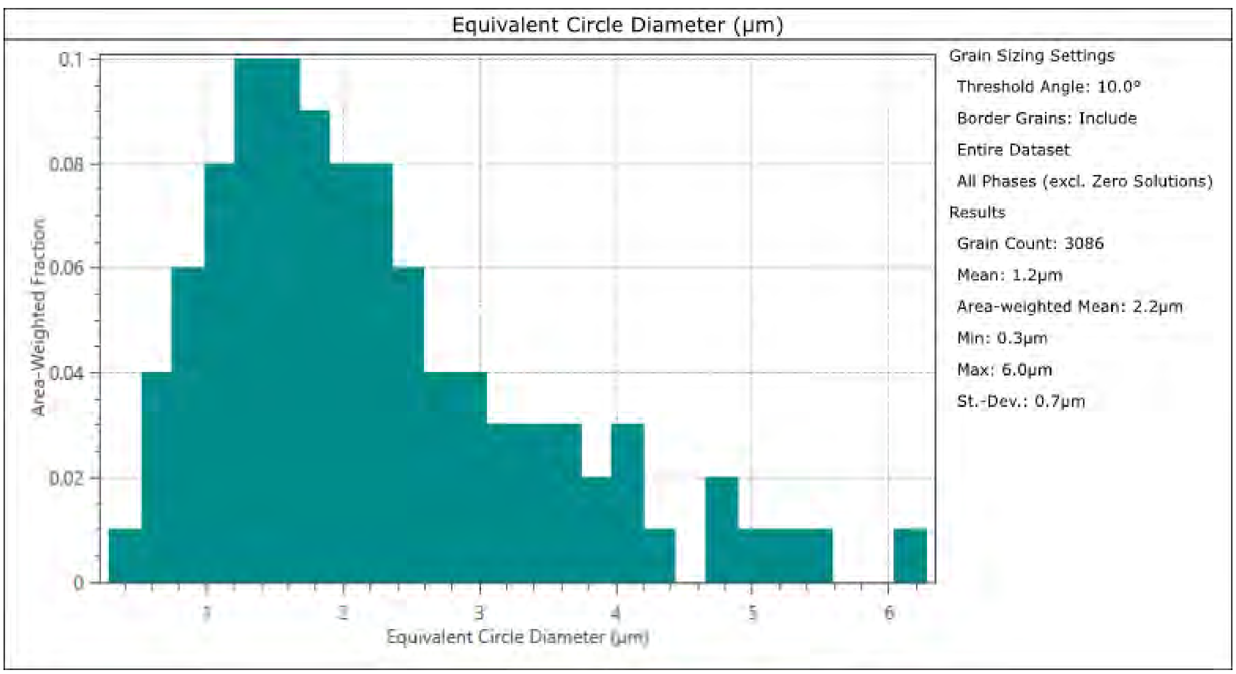

Figure 7.10. CED Grain Size and Other Data for L02: Condition C01, Sample SS31

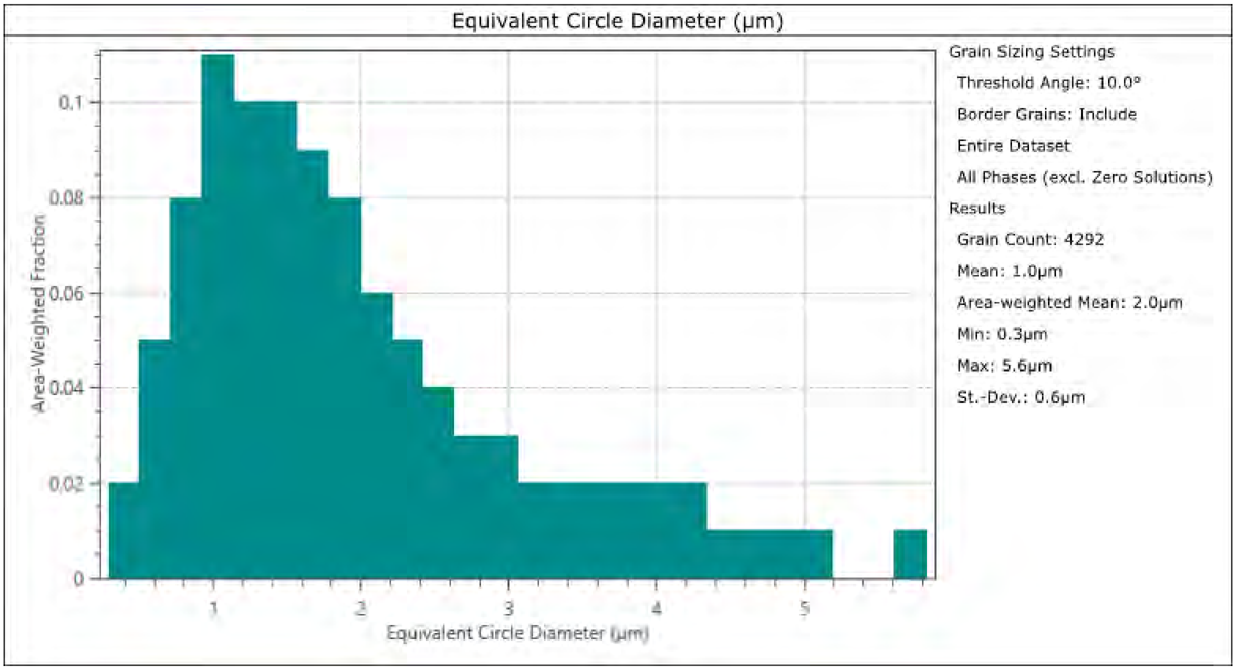

Figure 7.11. CED Grain Size and Other Data for L02: Condition C04, Sample SS32

7.2 Band Contrast + Grain Boundaries

The following figures show the grain boundaries identified through segmentation at the misorientation angle indicated below ”Grain Boundaries” in each figure (5° except for SS08 and SS28), 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.

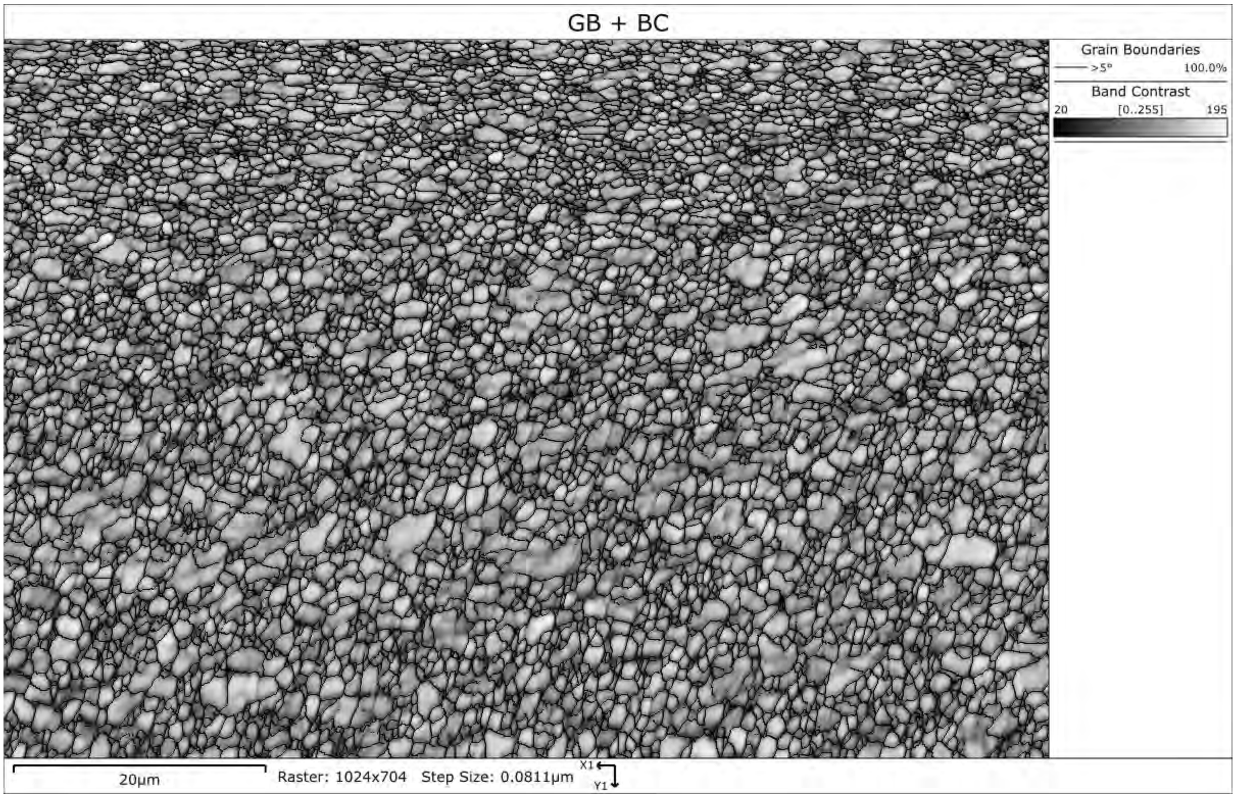

Figure 7.12. Grain Boundary + Band Contrast for L02: Condition C03, Sample SS01

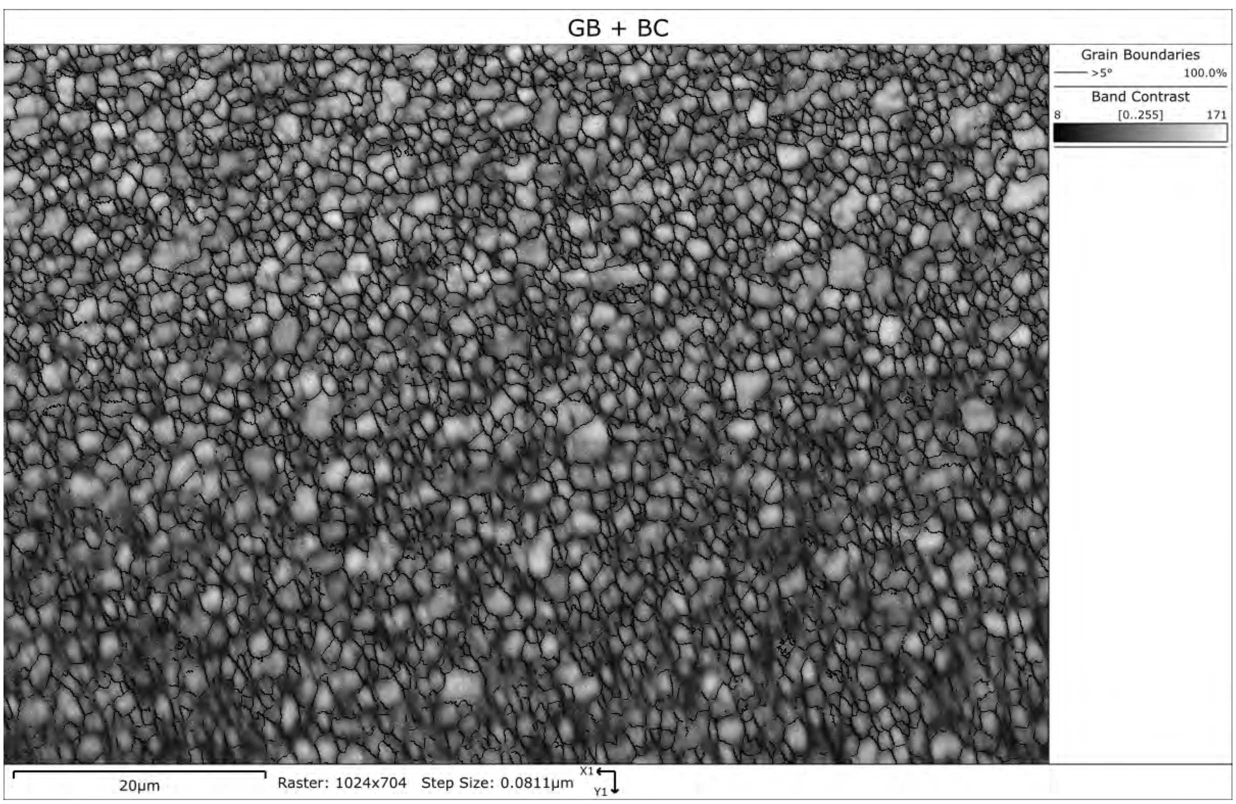

Figure 7.13. Grain Boundary + Band Contrast for L02: Condition C05, Sample SS02

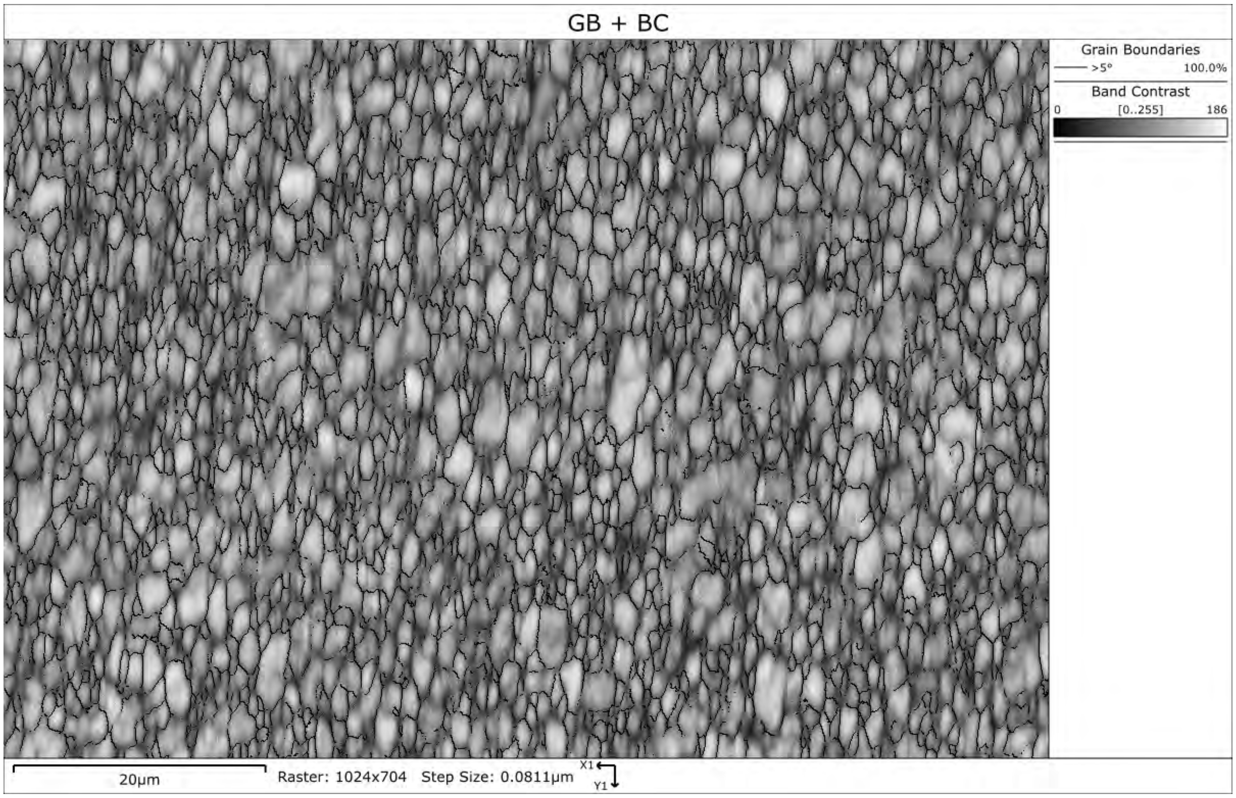

Figure 7.14. Grain Boundary + Band Contrast for L02: Condition C06, Sample SS03

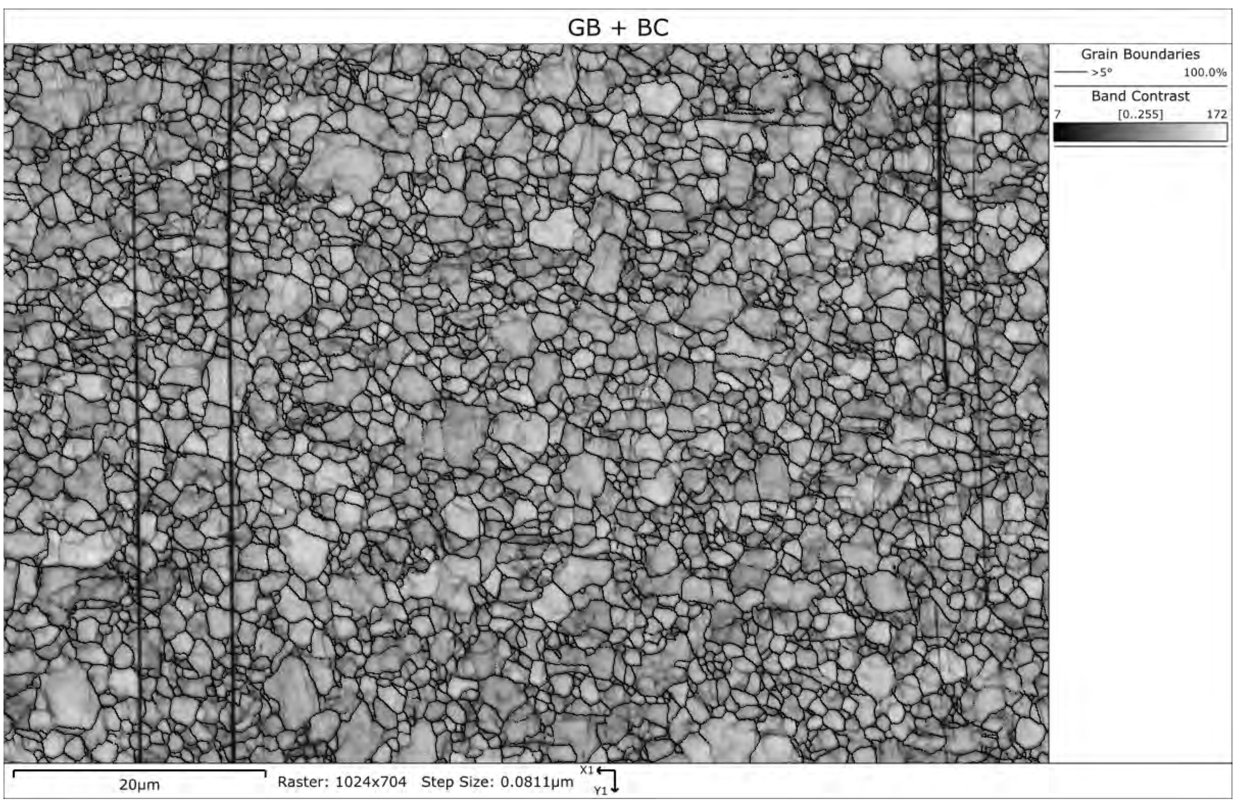

Figure 7.15. Grain Boundary + Band Contrast for L02: Condition C07, Sample SS04

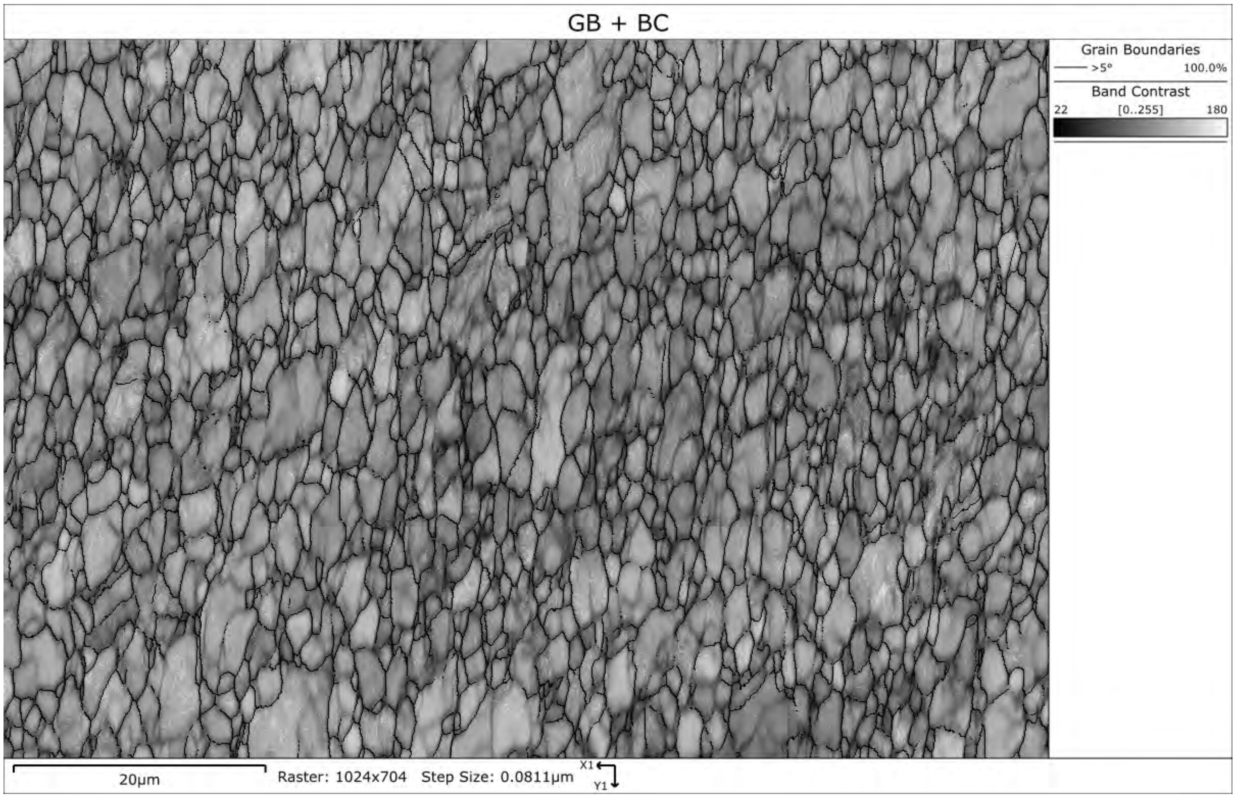

Figure 7.16. Grain Boundary + Band Contrast for L02: Condition C08, Sample SS05

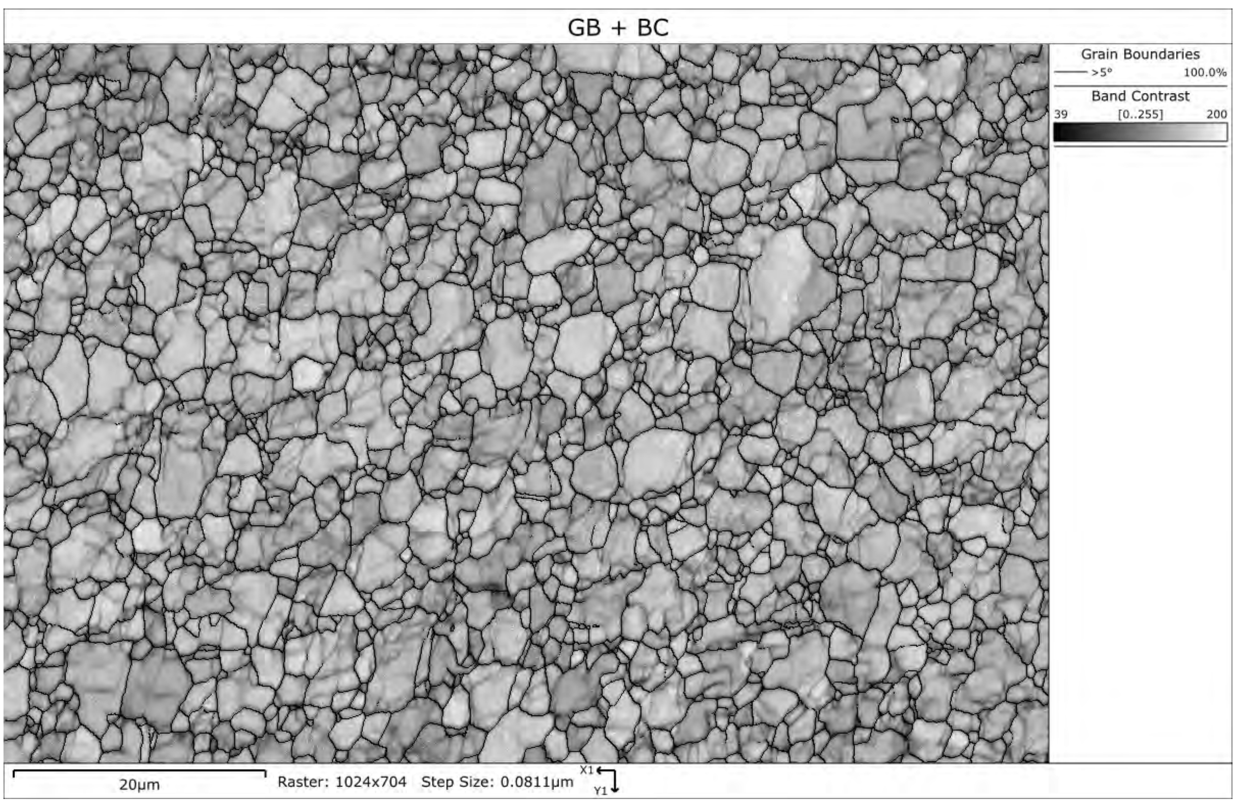

Figure 7.17. Grain Boundary + Band Contrast for L02: Condition C09, Sample SS06

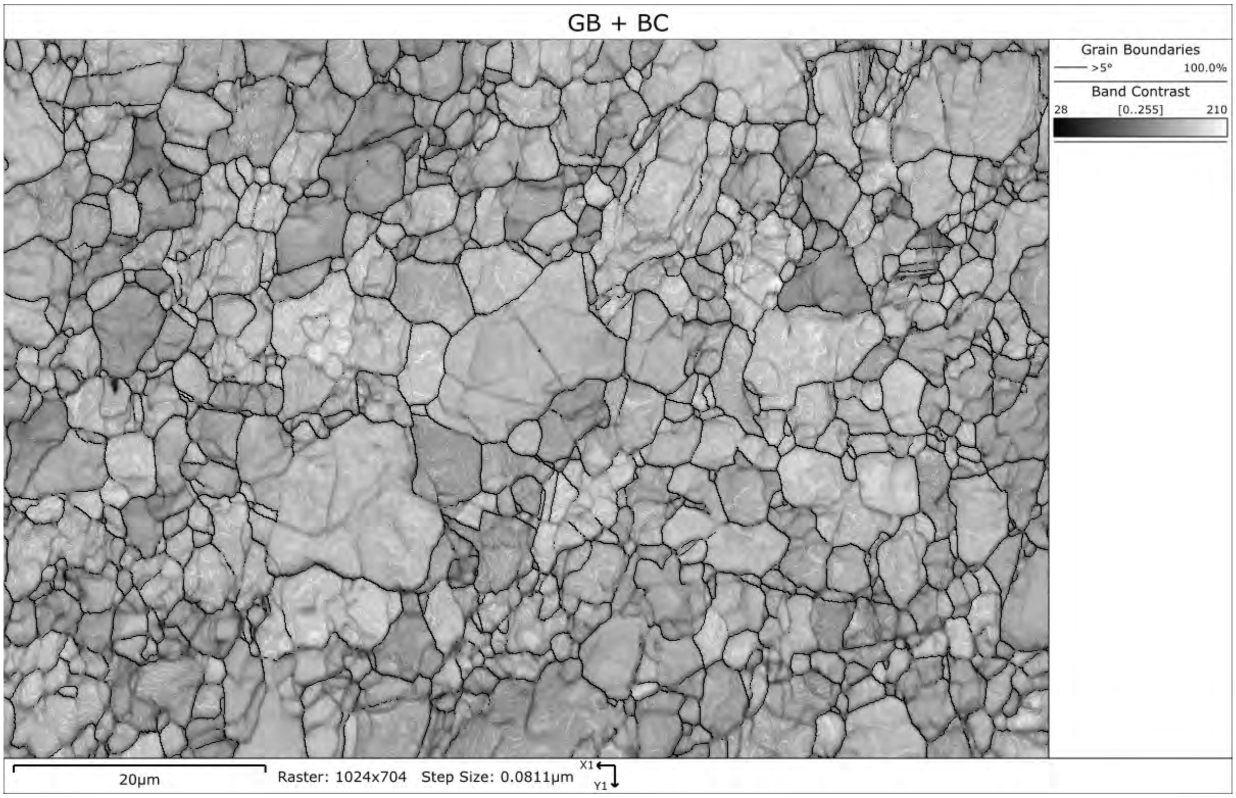

Figure 7.18. Grain Boundary + Band Contrast for L02: Condition C10, Sample SS07

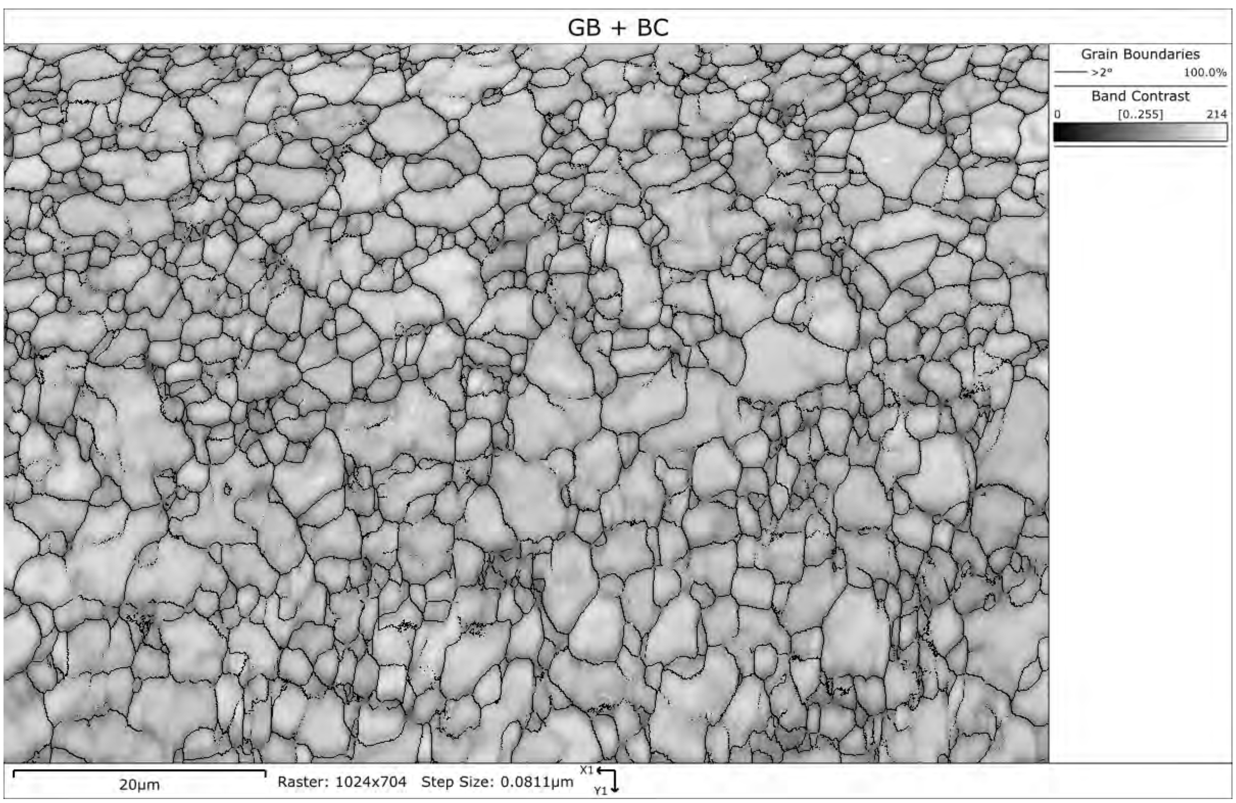

Figure 7.19. Grain Boundary + Band Contrast for L02: Condition C11, Sample SS08

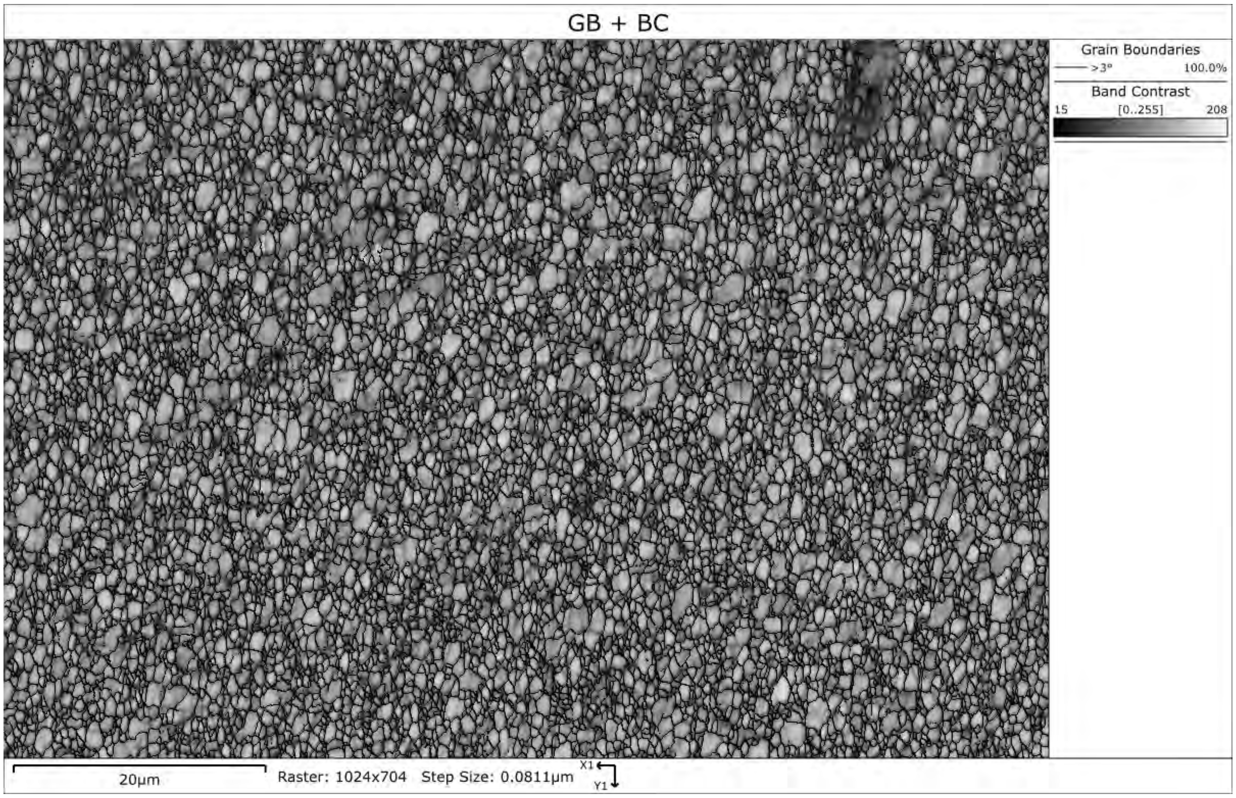

Figure 7.20. Grain Boundary + Band Contrast for L02: Condition C02, Sample SS28

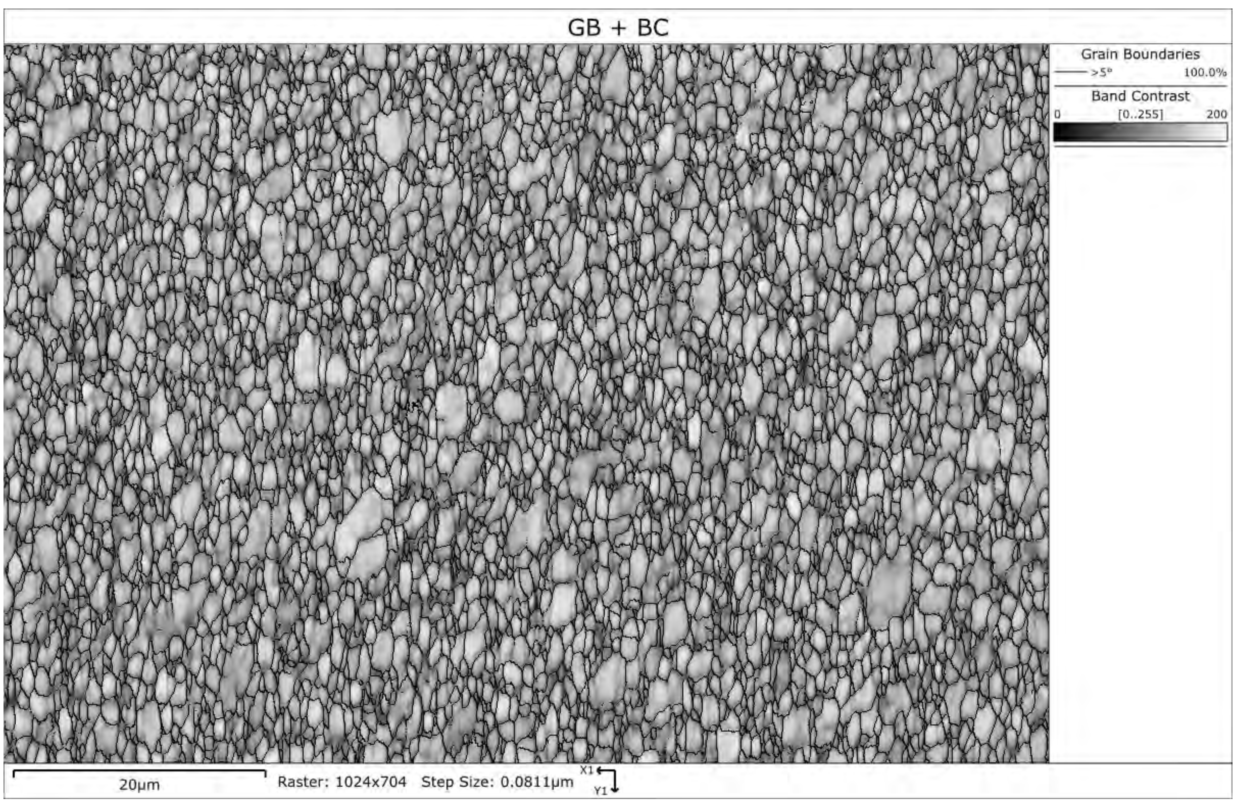

Figure 7.21. Grain Boundary + Band Contrast for L02: Condition C01, Sample SS31

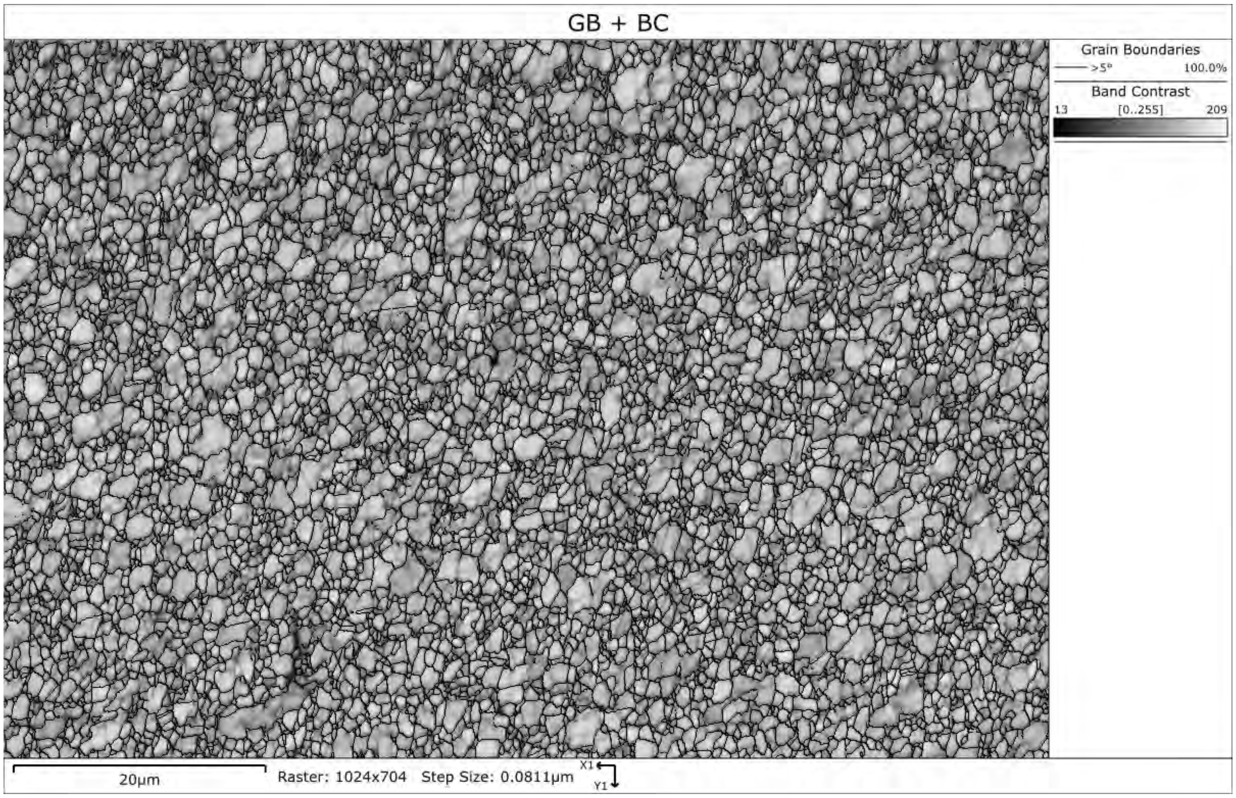

Figure 7.22. Grain Boundary + Band Contrast for L02: Condition C04, Sample SS32

7.3 Inverse Pole Function - X direction

The following figures show the IPF map in the x-direction with grain boundaries identified through segmentation at the misorientation angle indicated below ”Grain Boundaries” in each figure (5° except for SS08 and SS28), overlaid on the image.

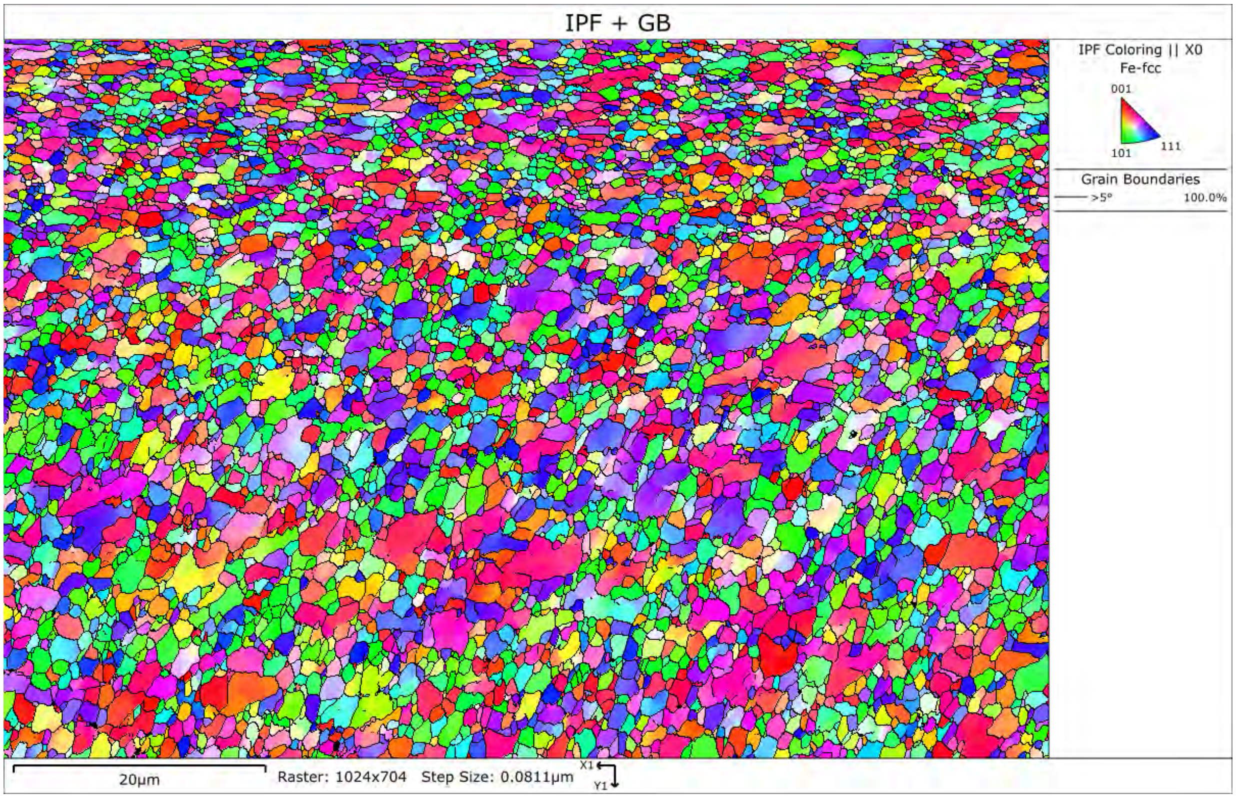

Figure 7.23. Inverse Pole Function - X Images for L02: Condition C03, Sample SS01

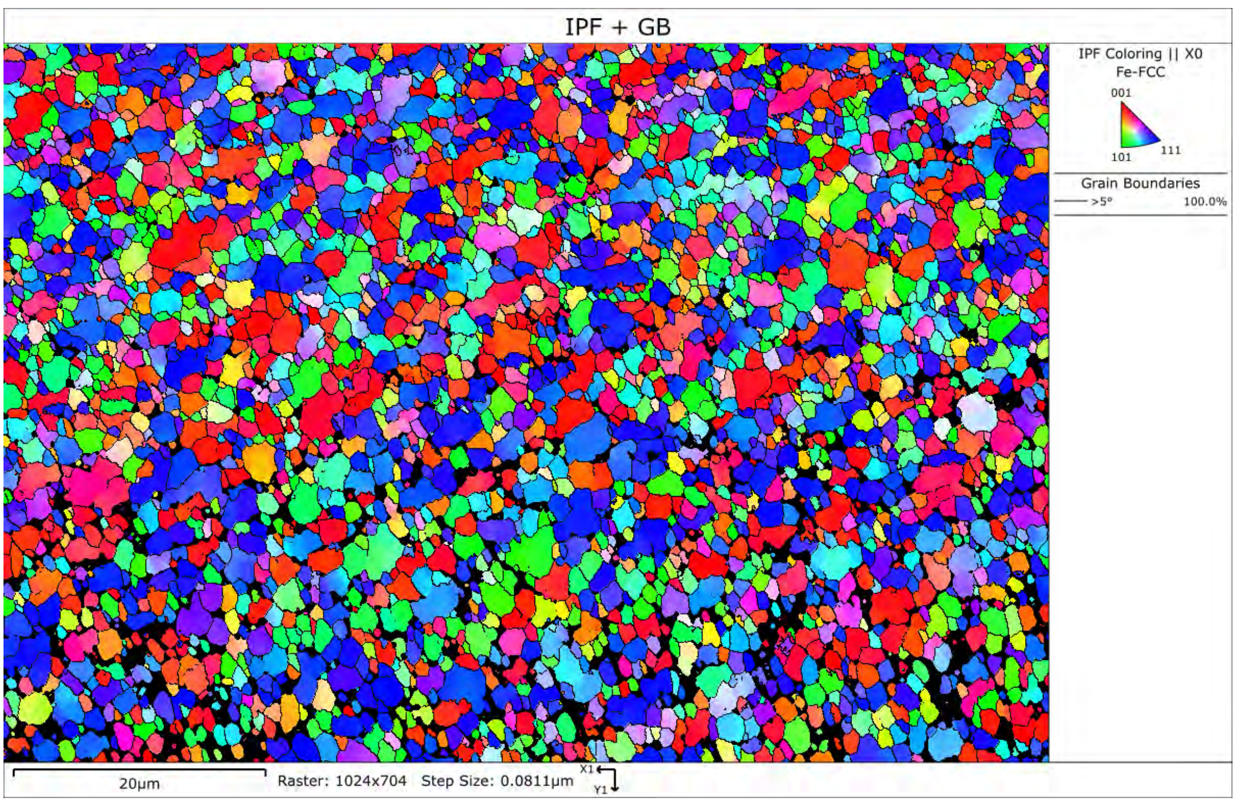

Figure 7.24. Inverse Pole Function - X Images for L02: Condition C05, Sample SS02

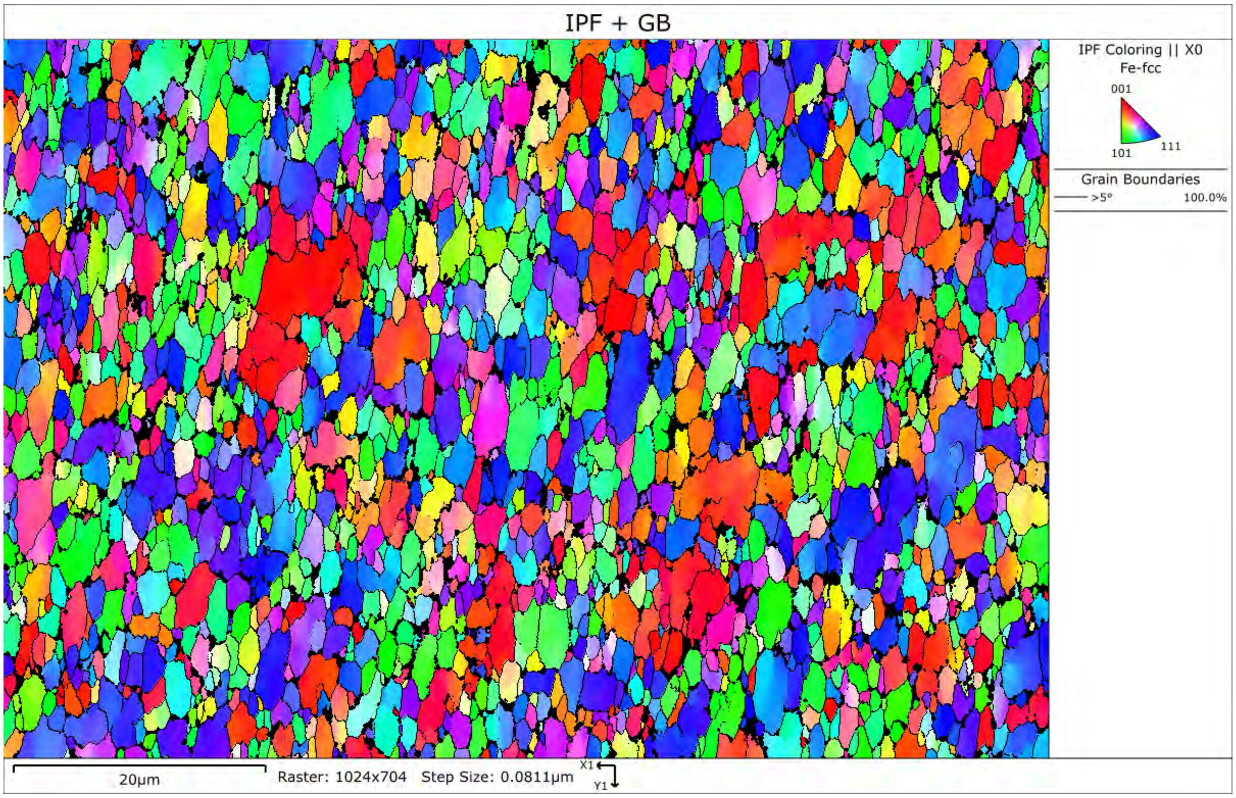

Figure 7.25. Inverse Pole Function - X Images for L02: Condition C06, Sample SS03

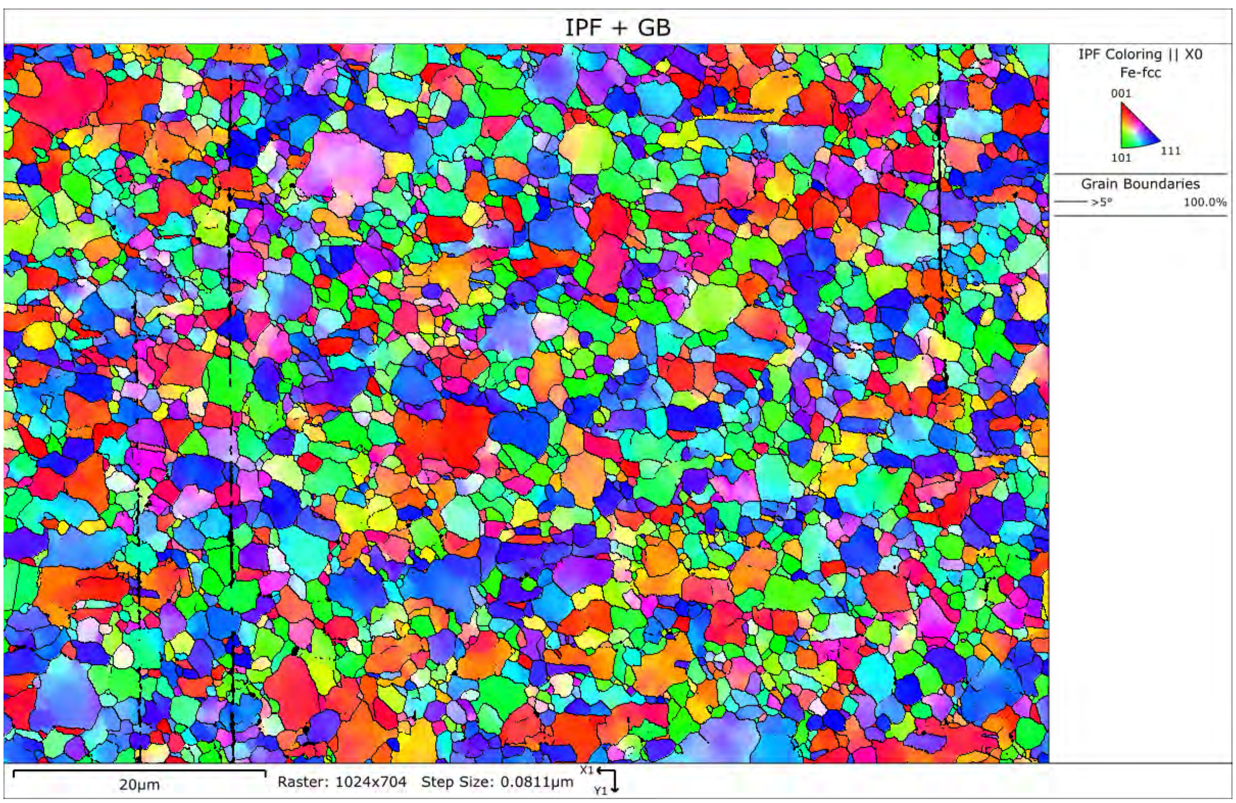

Figure 7.26. Inverse Pole Function - X Images for L02: Condition C07, Sample SS04

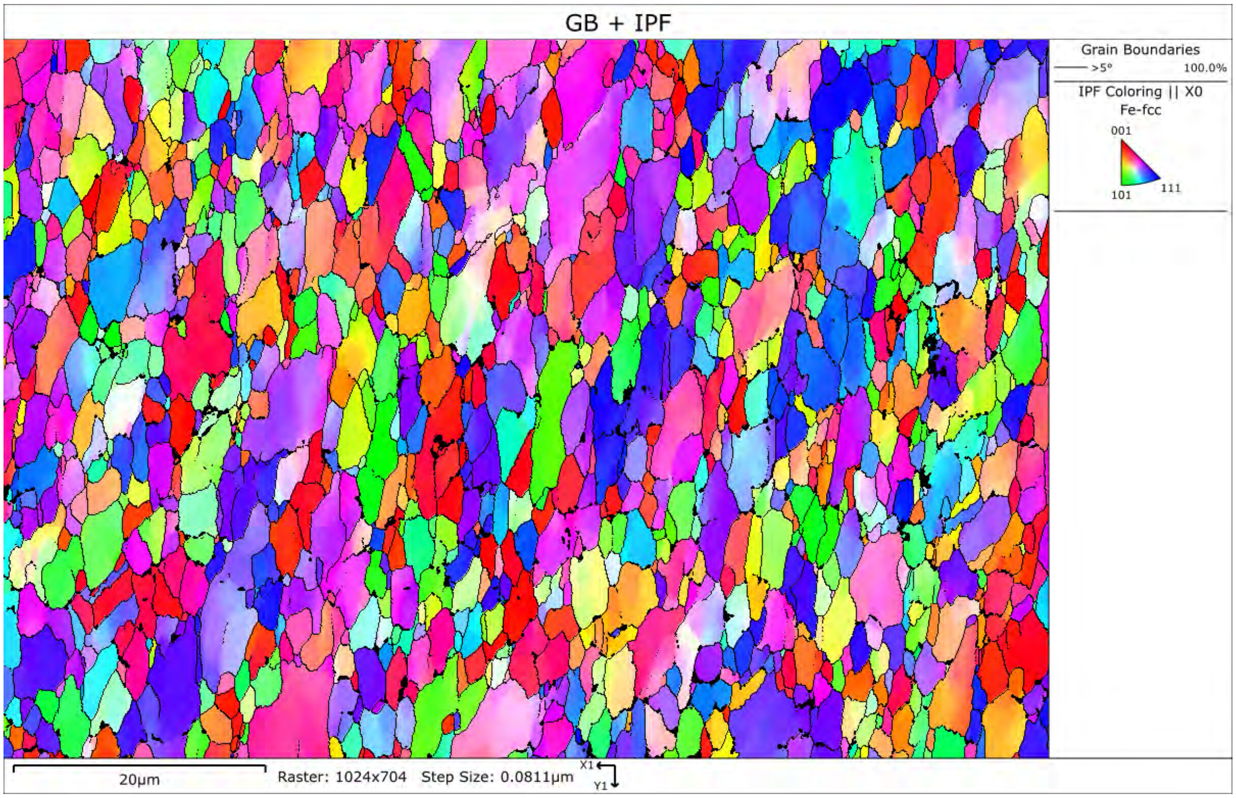

Figure 7.27. Inverse Pole Function - X Images for L02: Condition C08, Sample SS05

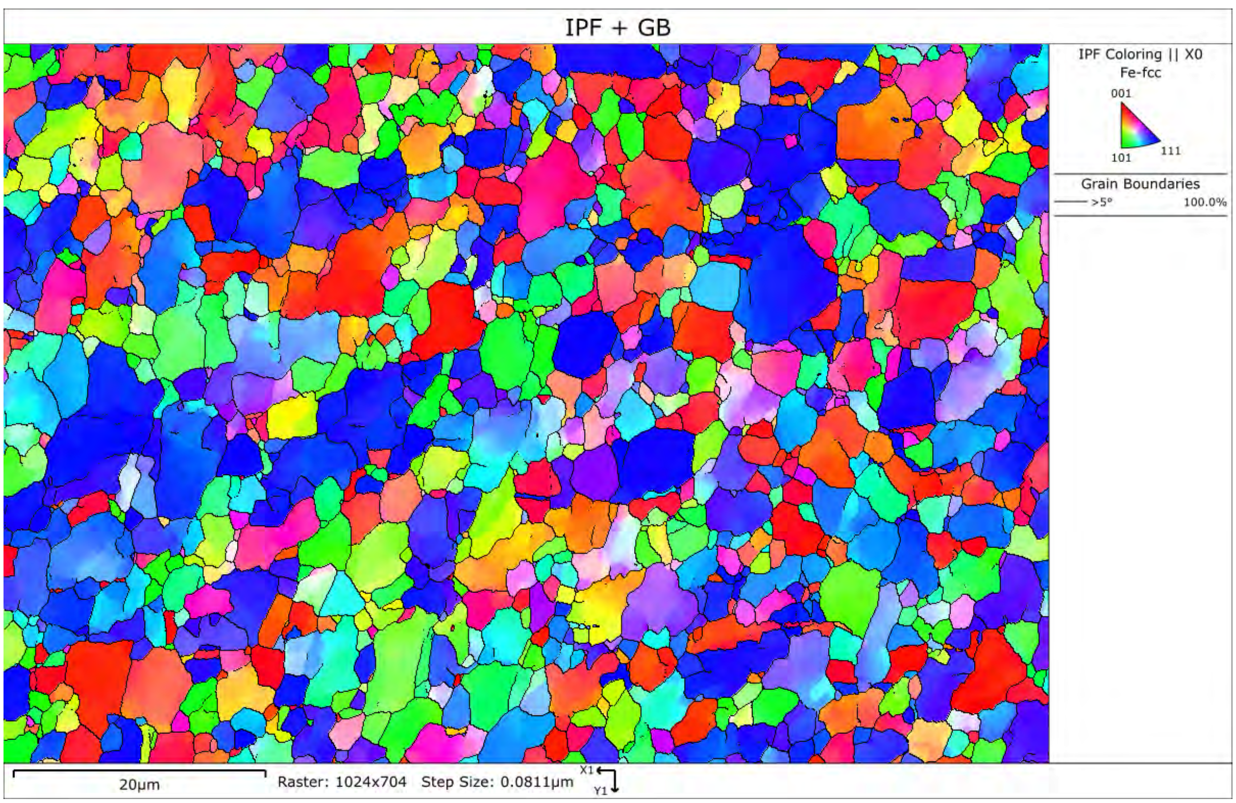

Figure 7.28. Inverse Pole Function - X Images for L02: Condition C09, Sample SS06

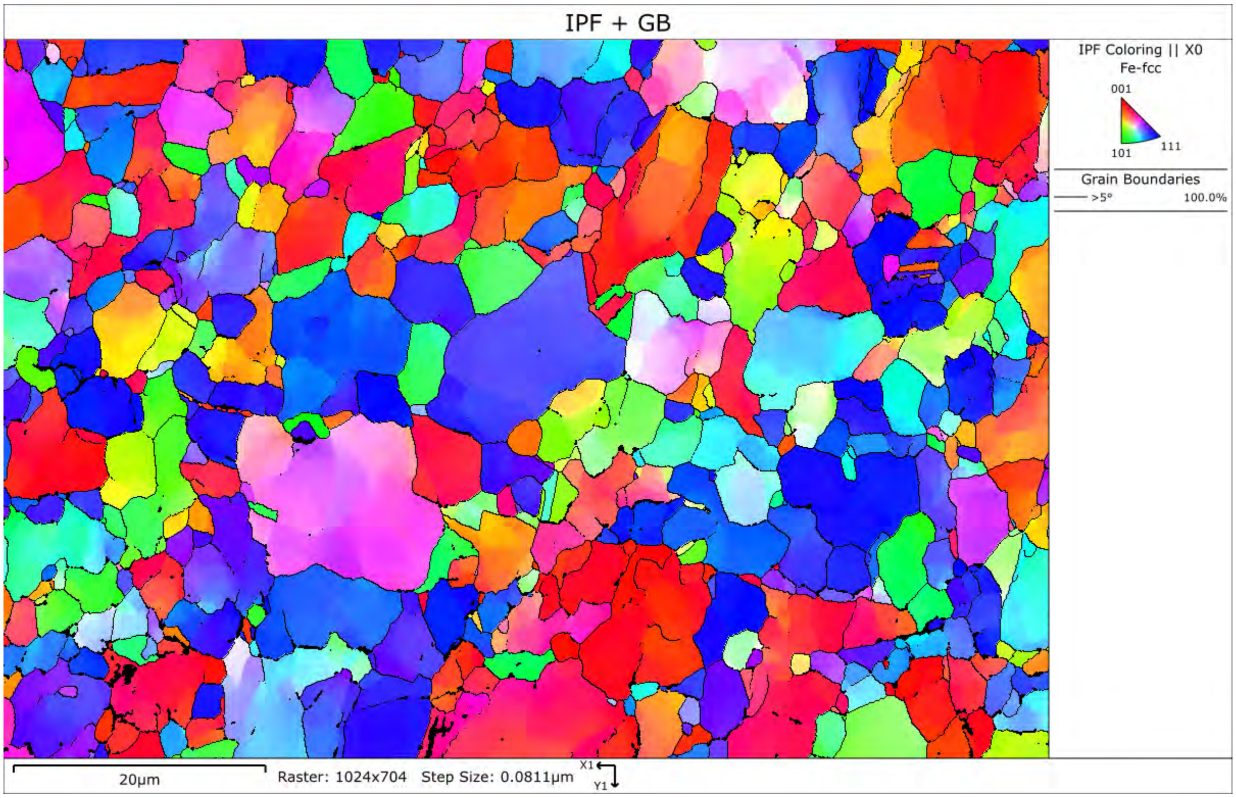

Figure 7.29. Inverse Pole Function - X Images for L02: Condition C10, Sample SS07

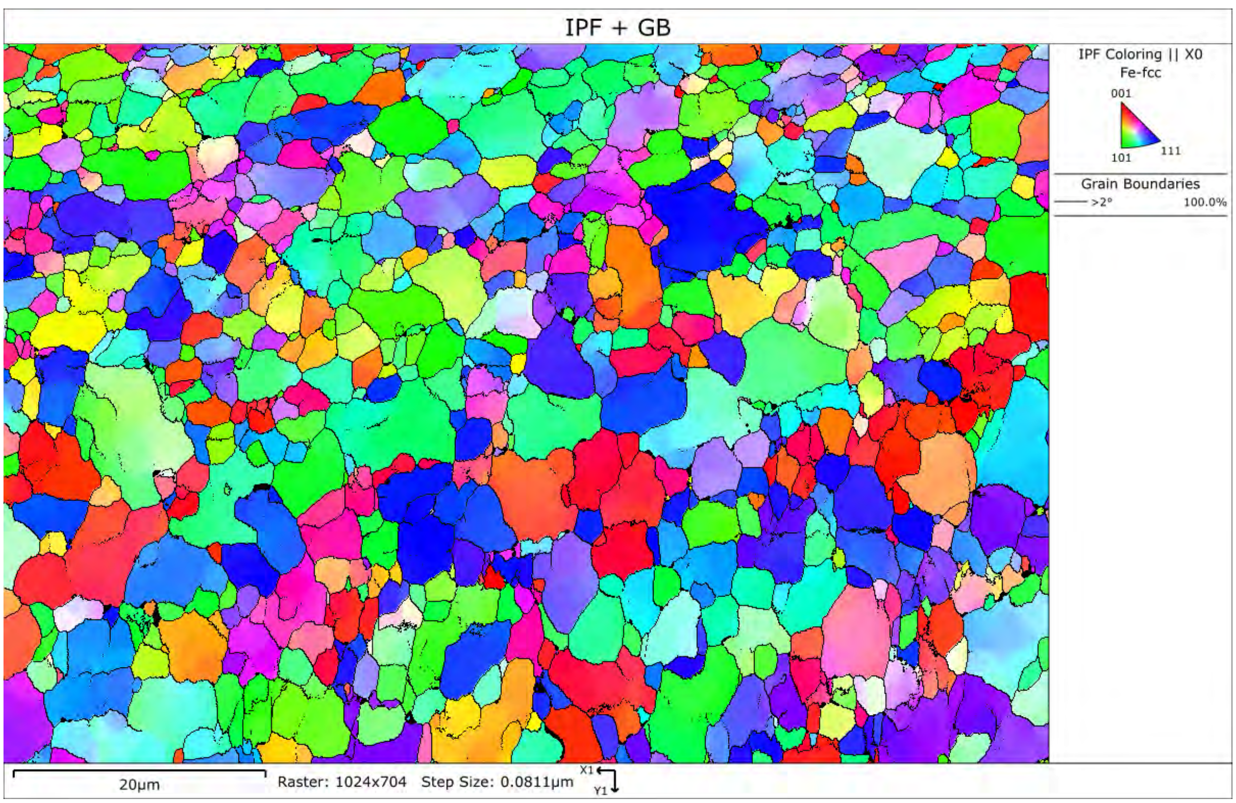

Figure 7.30. Inverse Pole Function - X Images for L02: Condition C11, Sample SS08

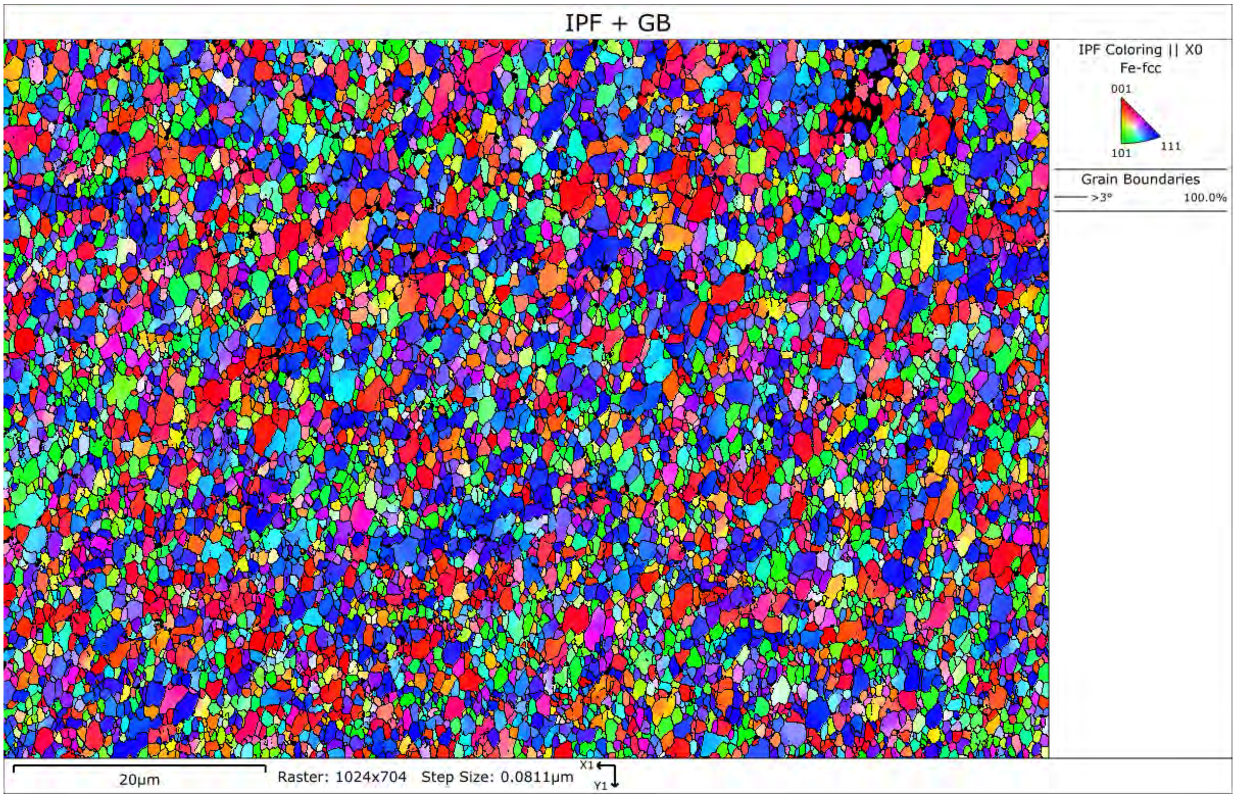

Figure 7.31. Inverse Pole Function - X Images for L02: Condition C02, Sample SS28

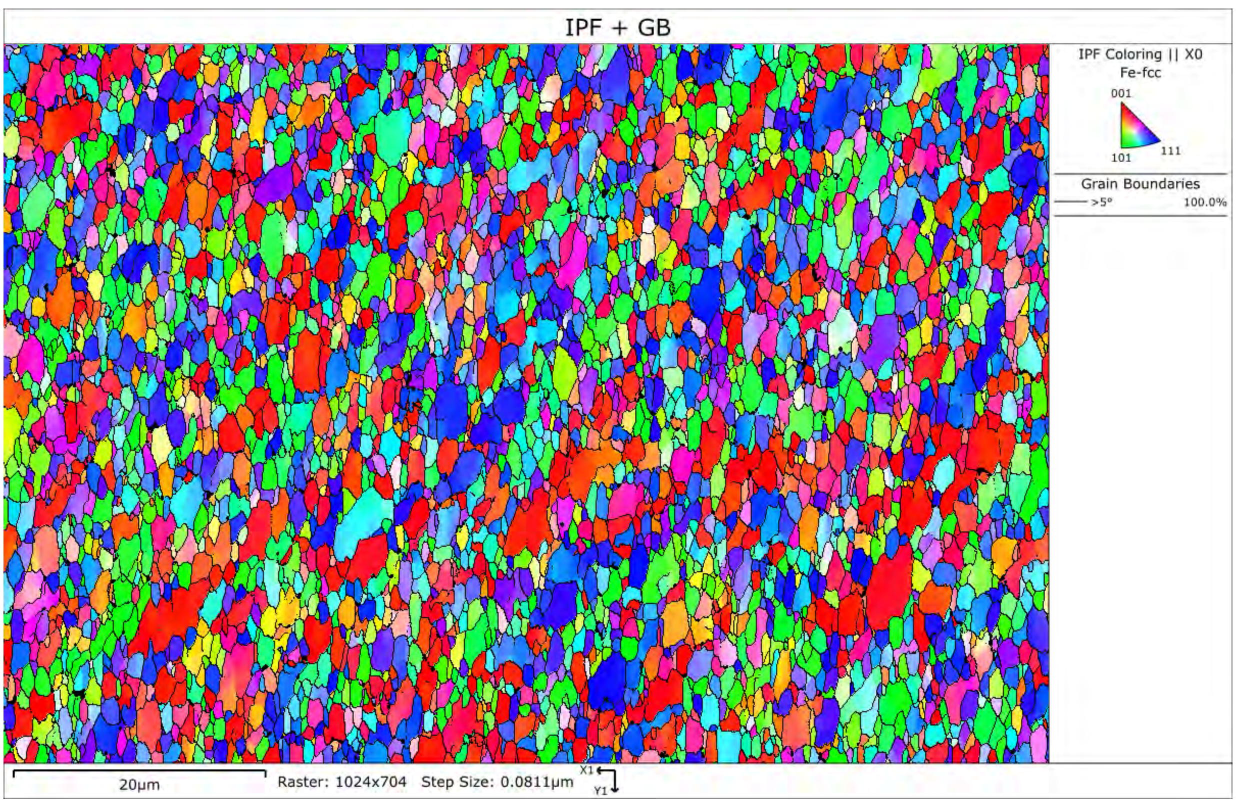

Figure 7.32. Inverse Pole Function - X Images for L02: Condition C01, Sample SS31

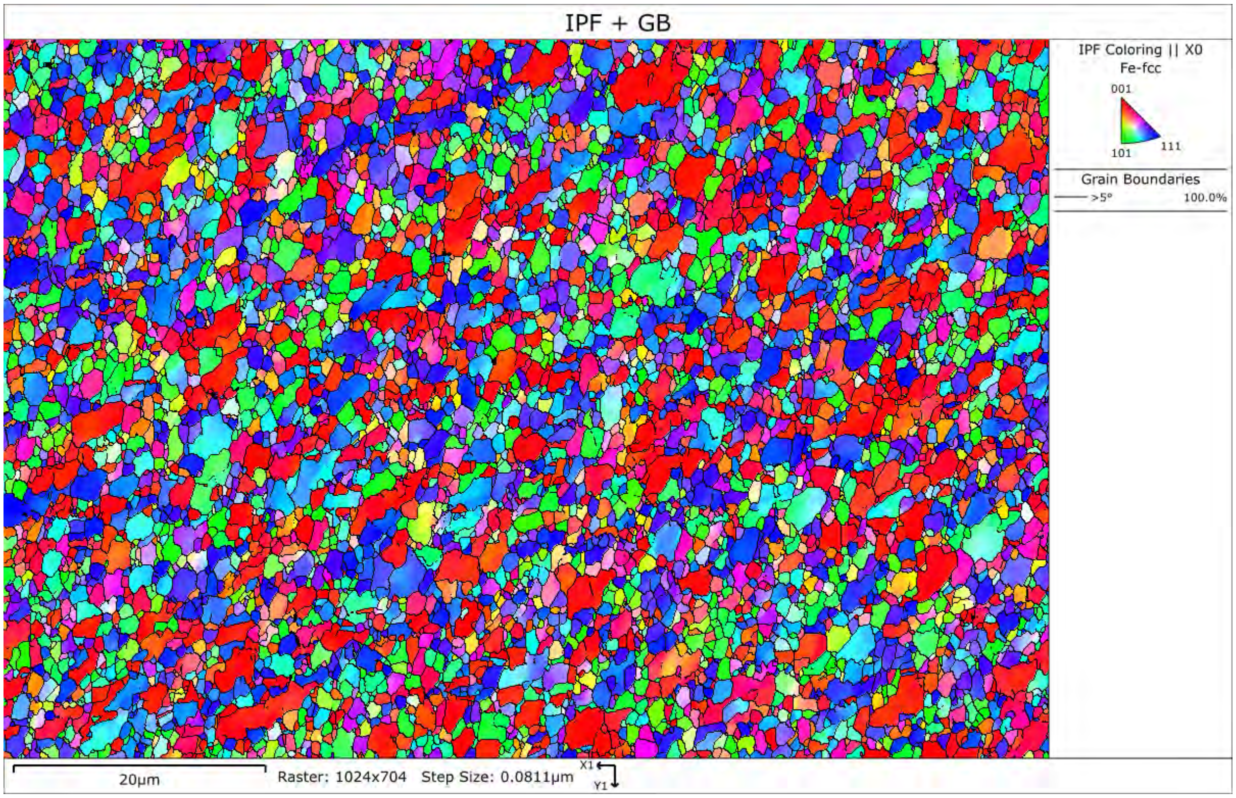

Figure 7.33. Inverse Pole Function - X Images for L02: Condition C04, Sample SS32

7.4 Inverse Pole Function - Y direction

The following figures show the IPF map in the y-direction with grain boundaries identified through segmentation at the misorientation angle indicated below "Grain Boundaries" in each figure (5° except for SS08 and SS28), overlaid on the image.

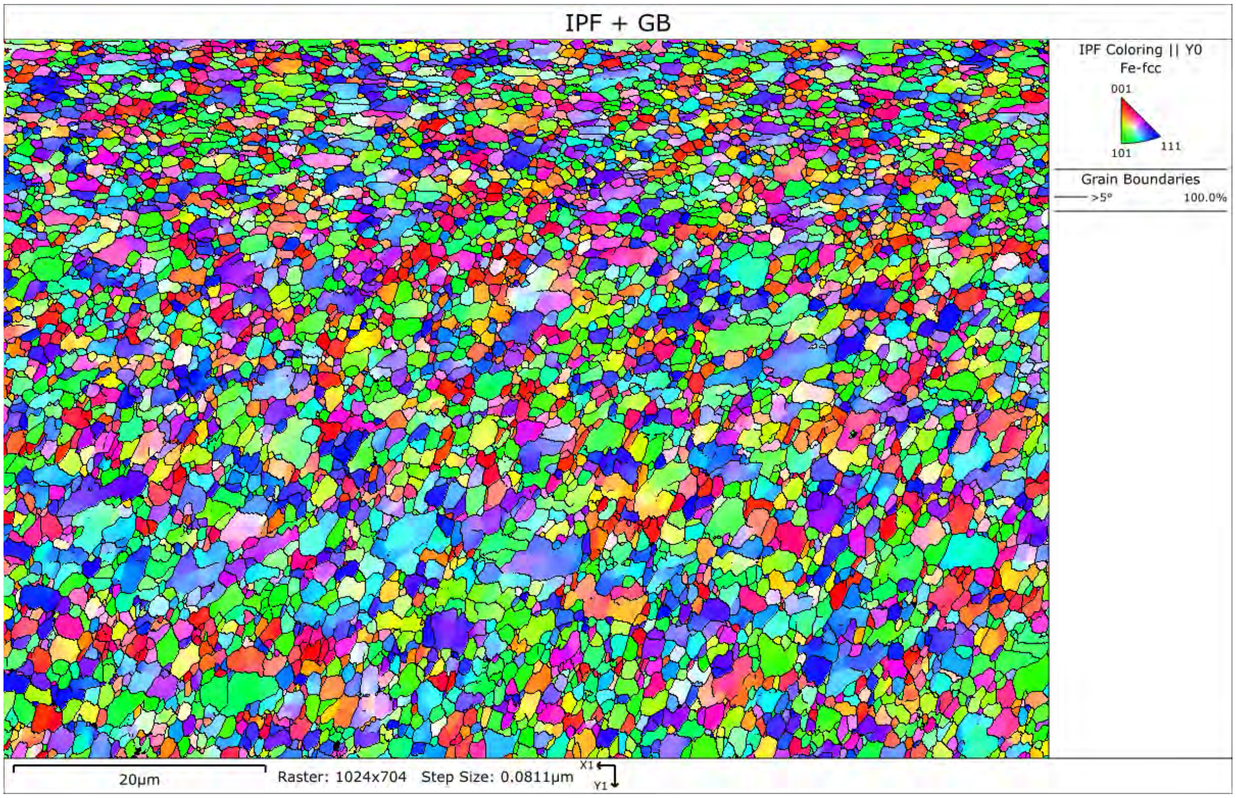

Figure 7.34. Inverse Pole Function - Y Images for L02: Condition C03, Sample SS01

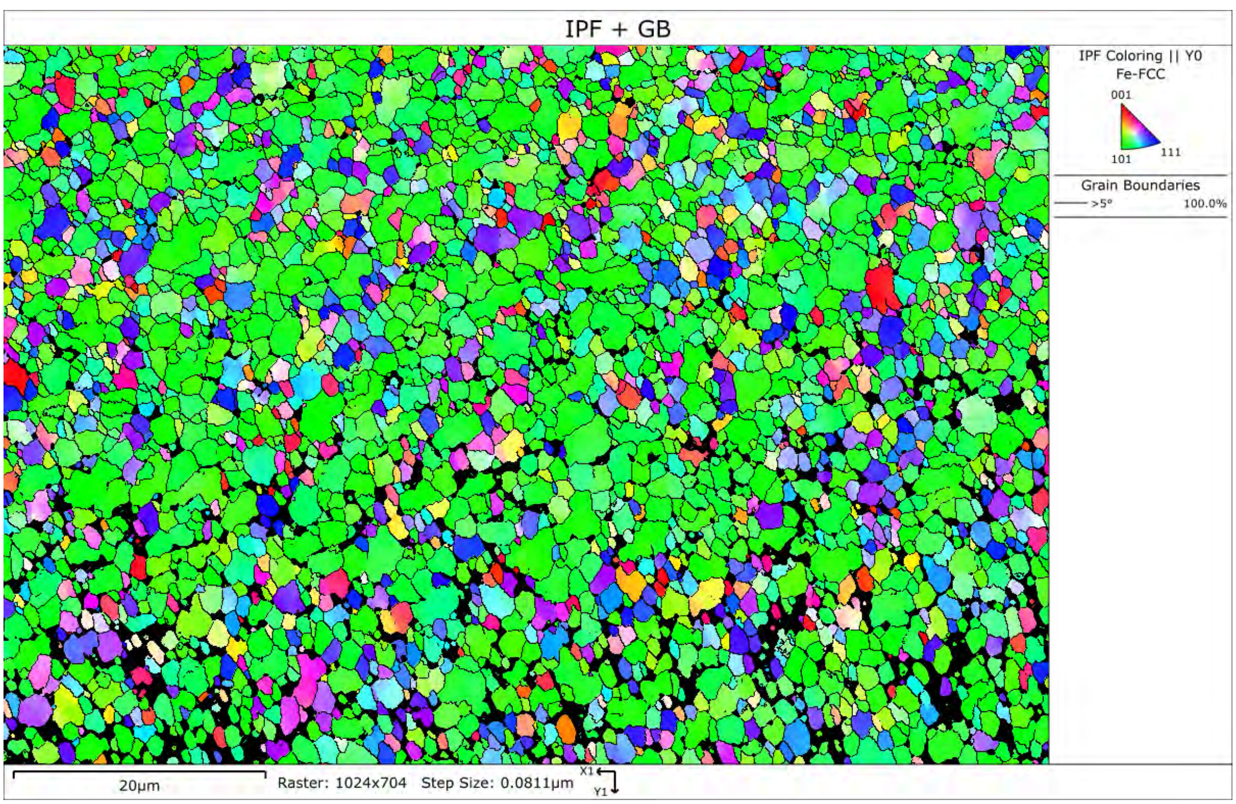

Figure 7.35. Inverse Pole Function - Y Images for L02: Condition C05, Sample SS02

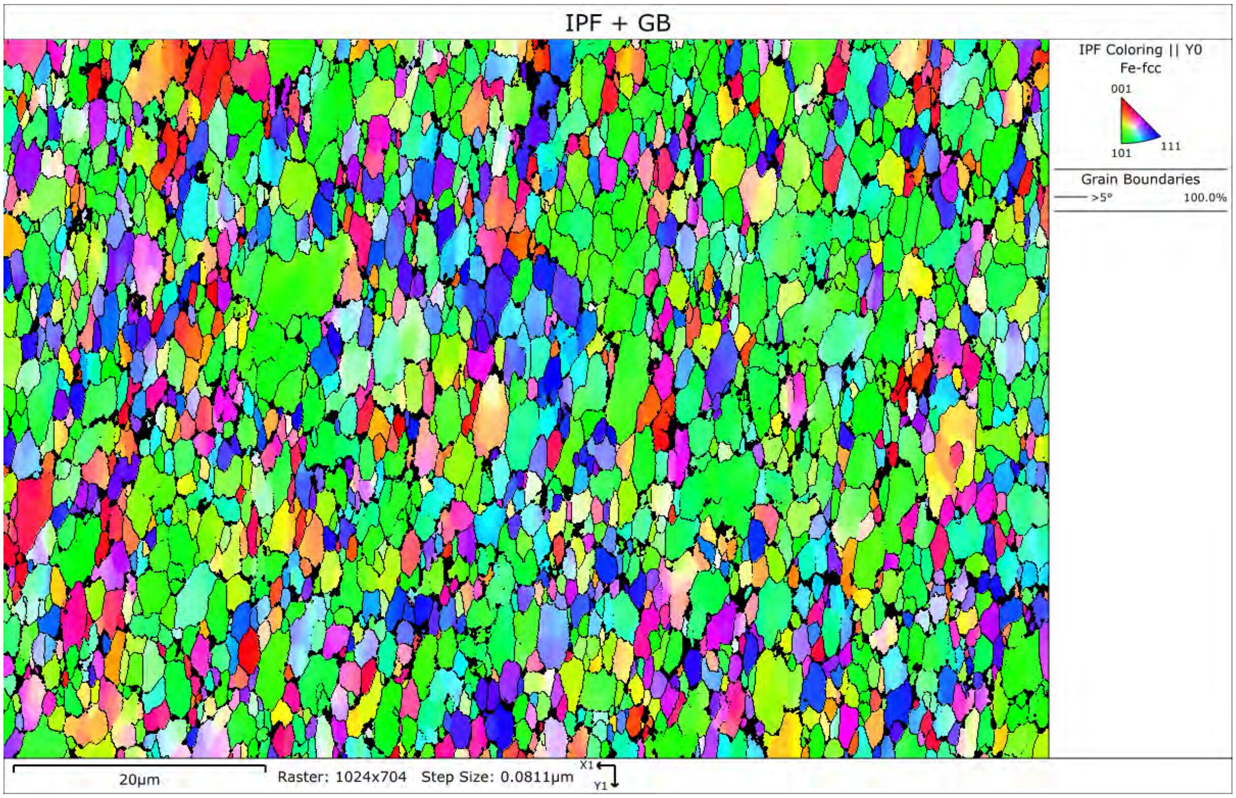

Figure 7.36. Inverse Pole Function - Y Images for L02: Condition C06, Sample SS03

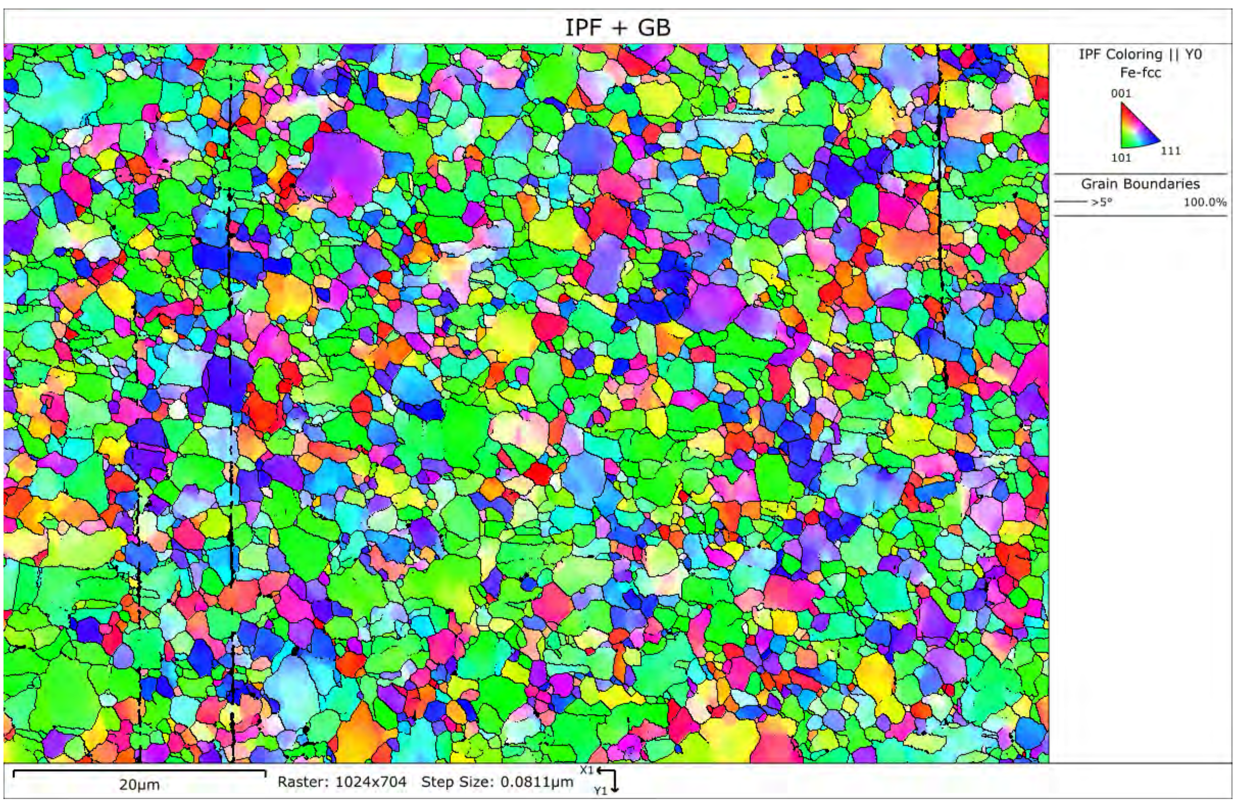

Figure 7.37. Inverse Pole Function - Y Images for L02: Condition C07, Sample SS04

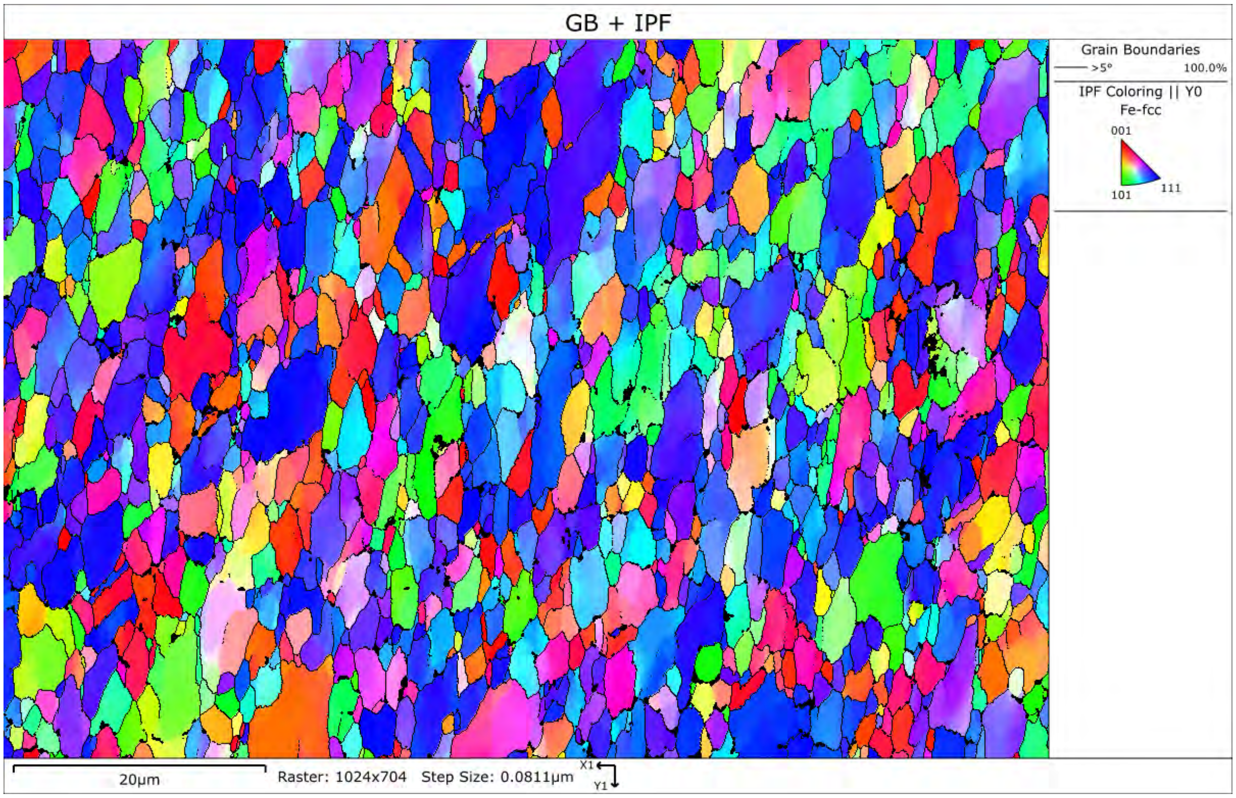

Figure 7.38. Inverse Pole Function - Y Images for L02: Condition C08, Sample SS05

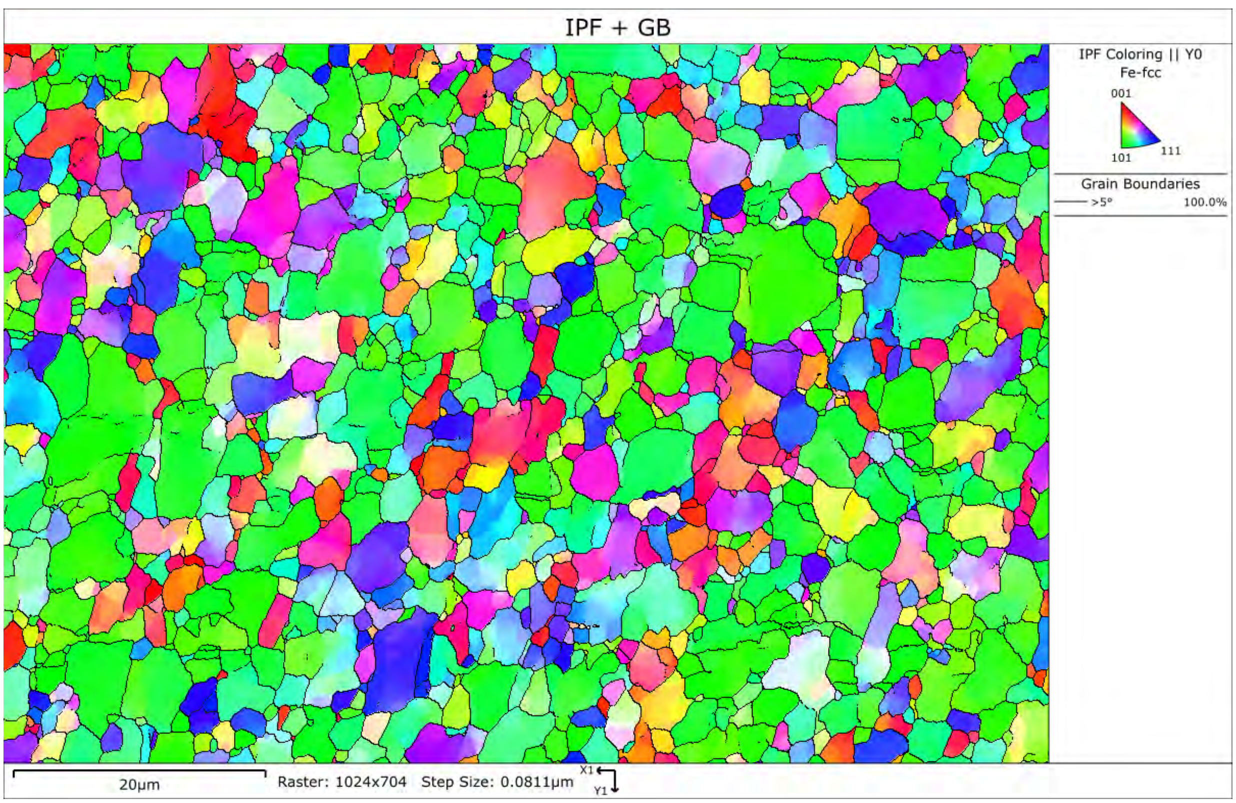

Figure 7.39. Inverse Pole Function - Y Images for L02: Condition C09, Sample SS06

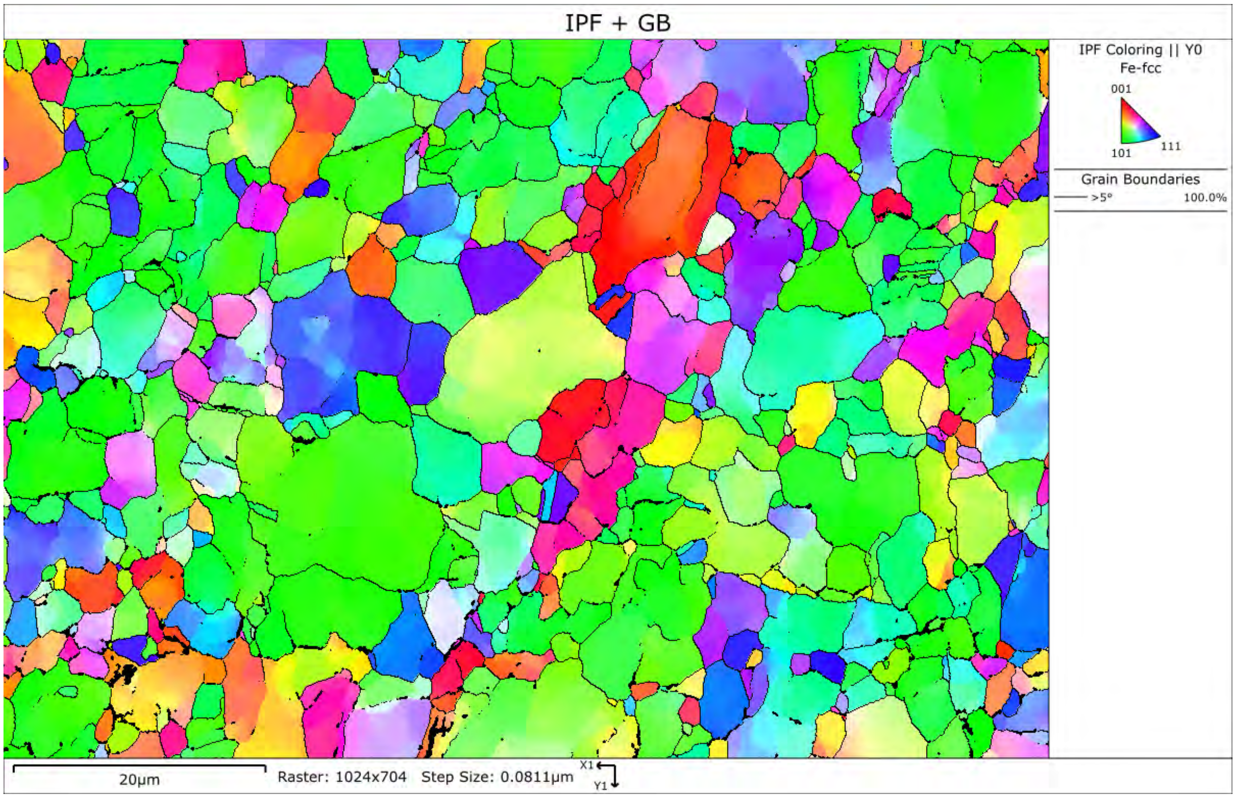

Figure 7.40. Inverse Pole Function - Y Images for L02: Condition C10, Sample SS07

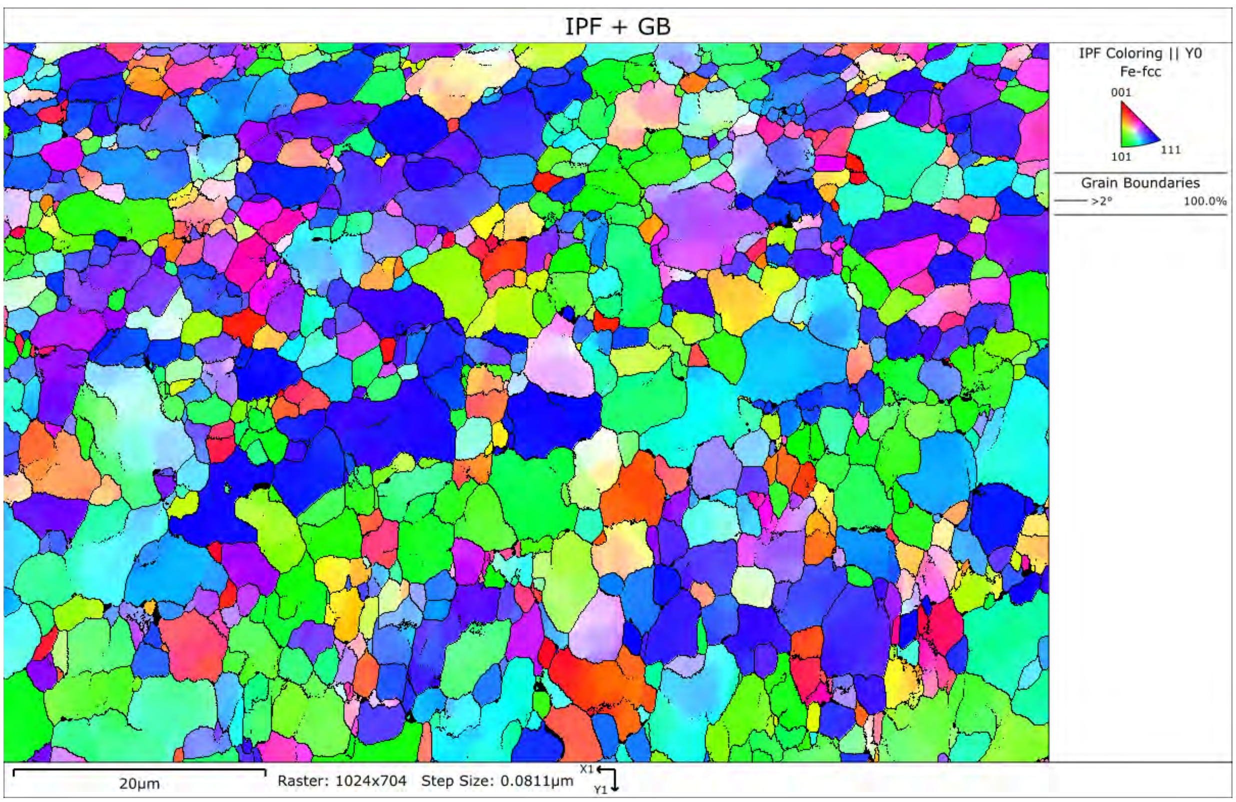

Figure 7.41. Inverse Pole Function - Y Images for L02: Condition C11, Sample SS08

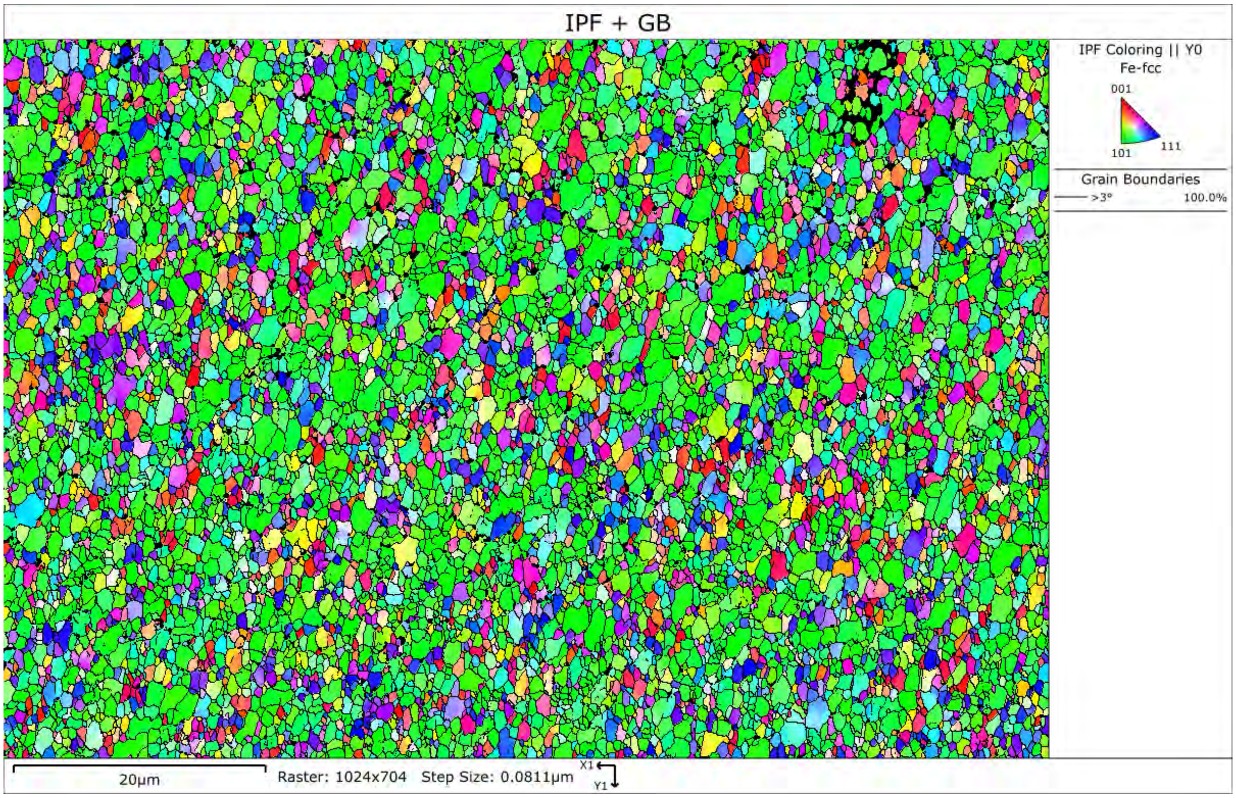

Figure 7.42. Inverse Pole Function - Y Images for L02: Condition C02, Sample SS28

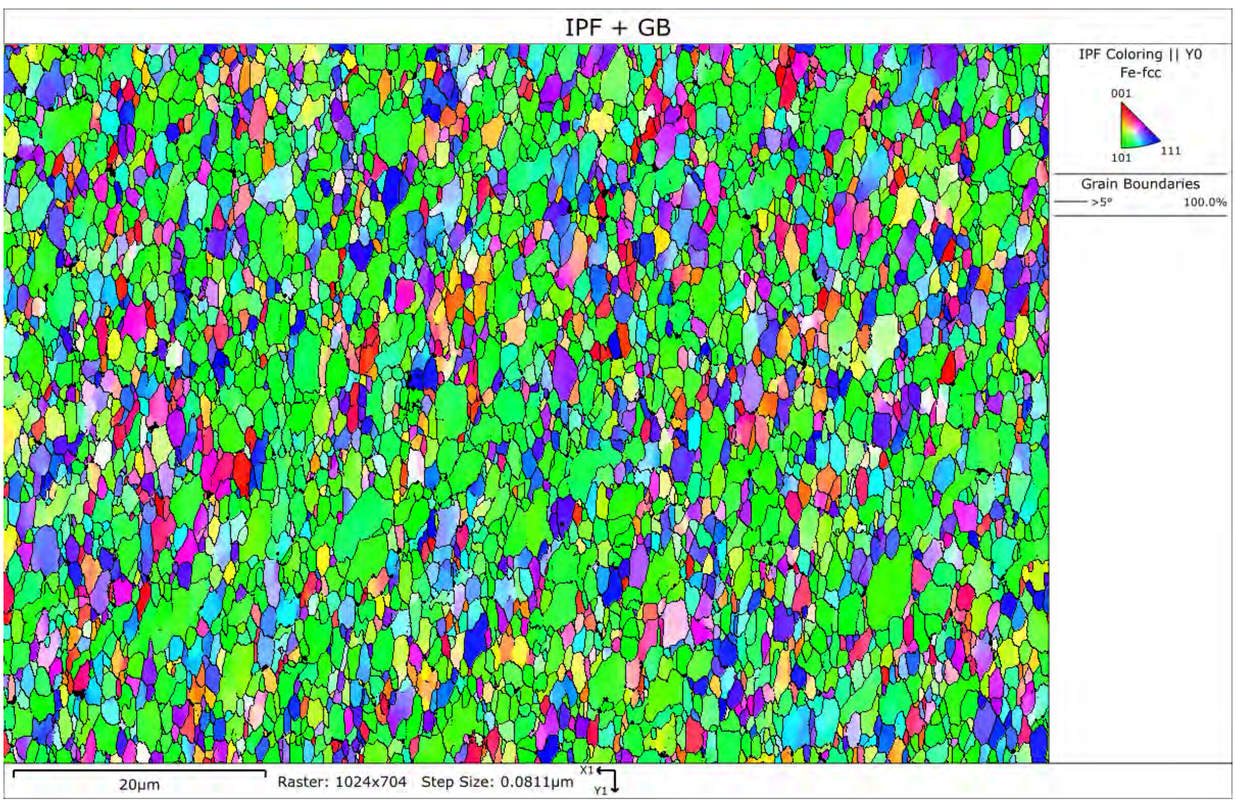

Figure 7.43. Inverse Pole Function - Y Images for L02: Condition C01, Sample SS31

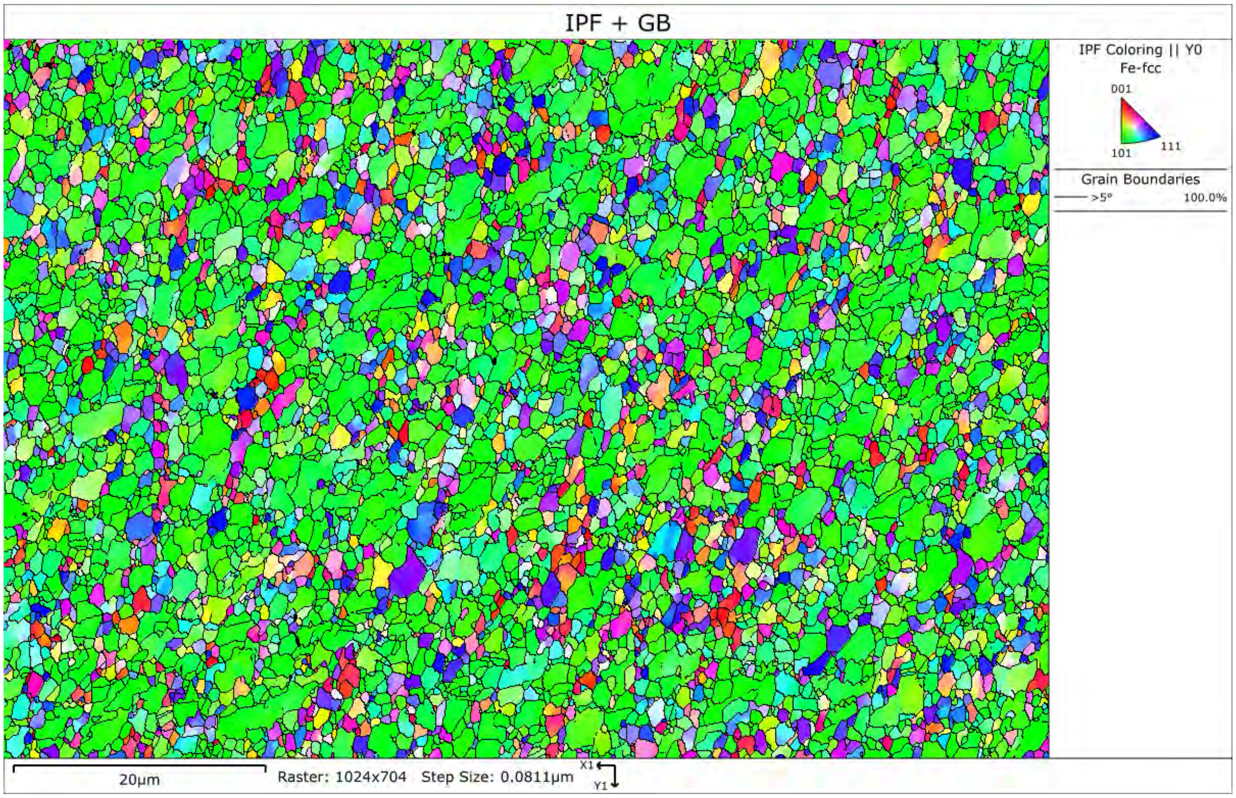

Figure 7.44. Inverse Pole Function - Y Images for L02: Condition C04, Sample SS32

7.5 Inverse Pole Function - Z direction

The following figures show the IPF map in the z-direction with grain boundaries identified through segmentation at the misorientation angle indicated below ”Grain Boundaries” in each figure (5° except for SS08 and SS28), overlaid on the image.

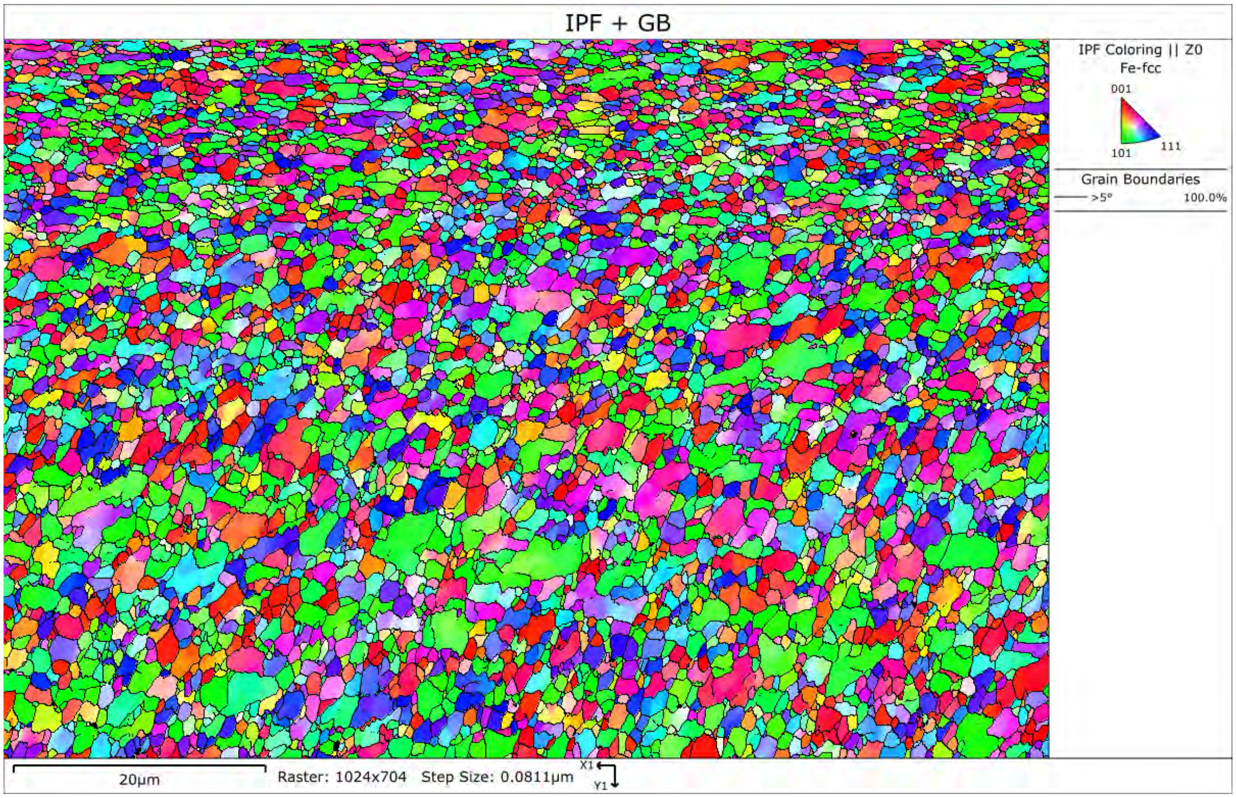

Figure 7.45. Inverse Pole Function - Z Images for L02: Condition C03, Sample SS01

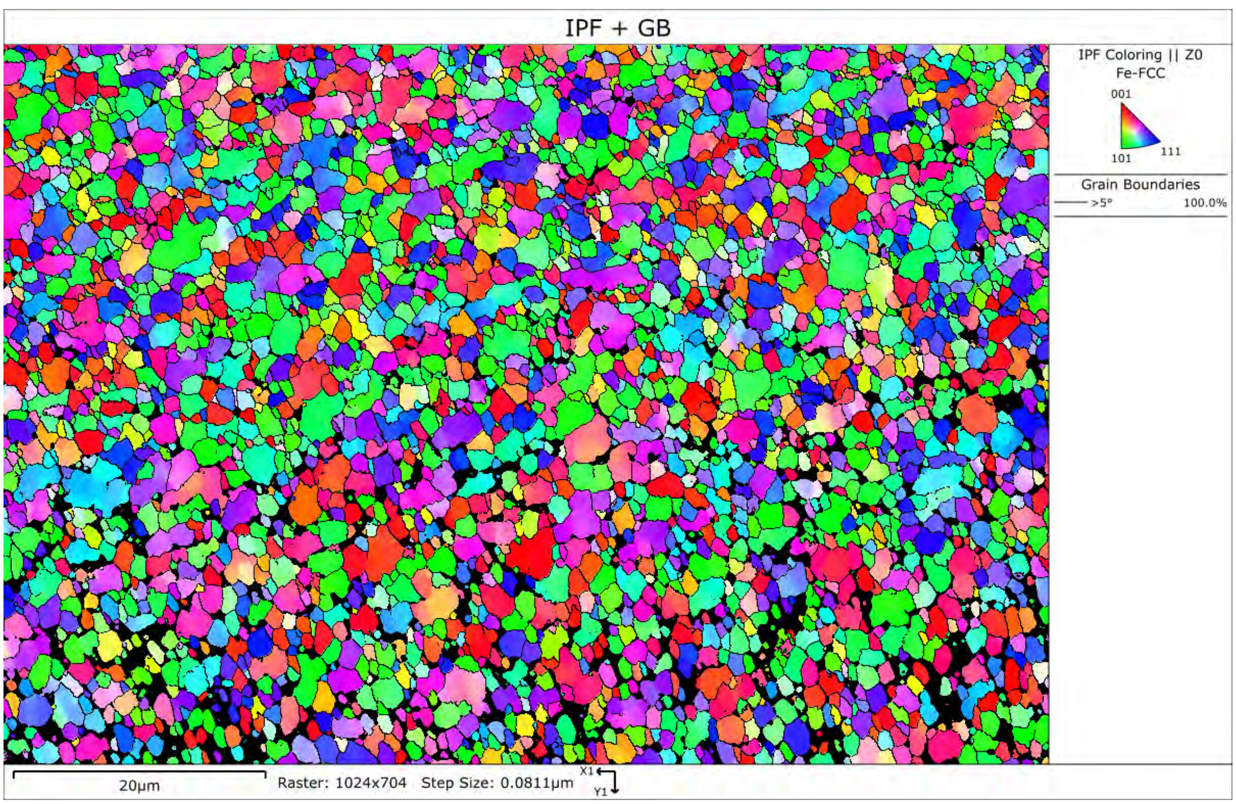

Figure 7.46. Inverse Pole Function - Z Images for L02: Condition C05, Sample SS02

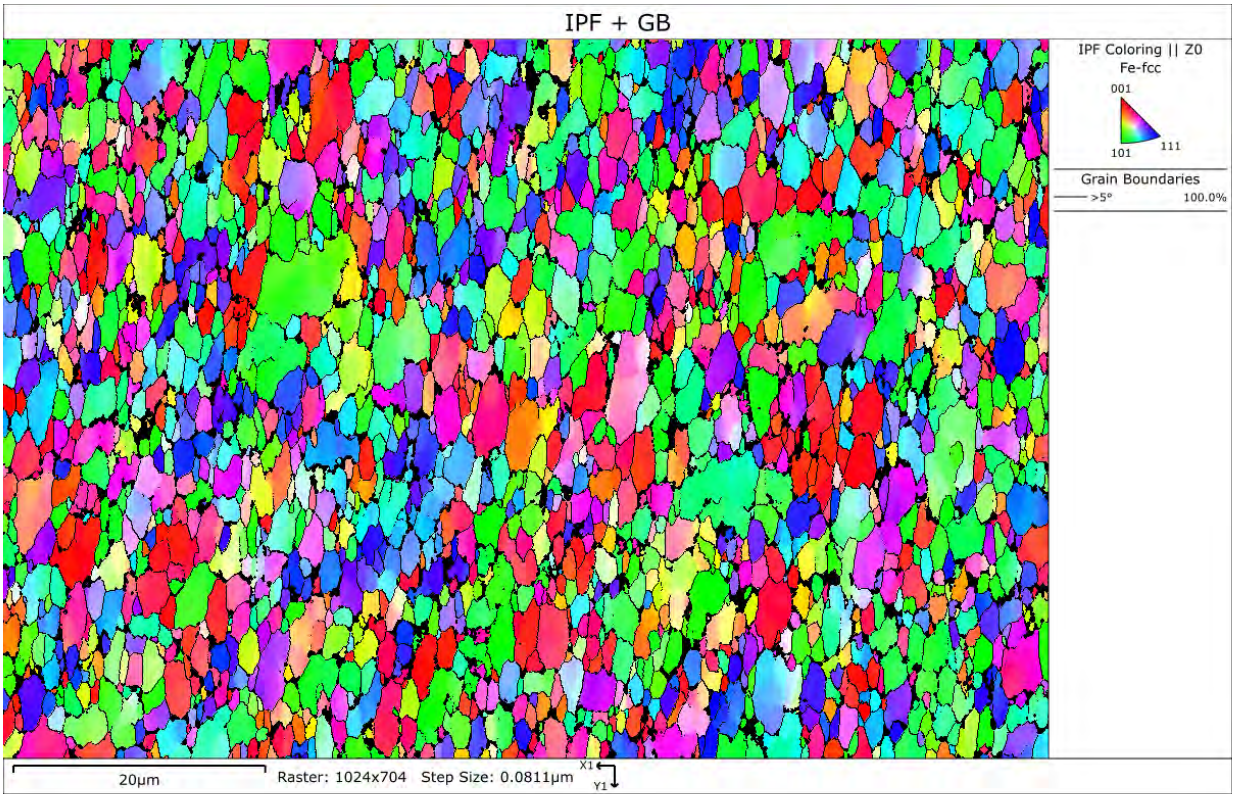

Figure 7.47. Inverse Pole Function - Z Images for L02: Condition C06, Sample SS03

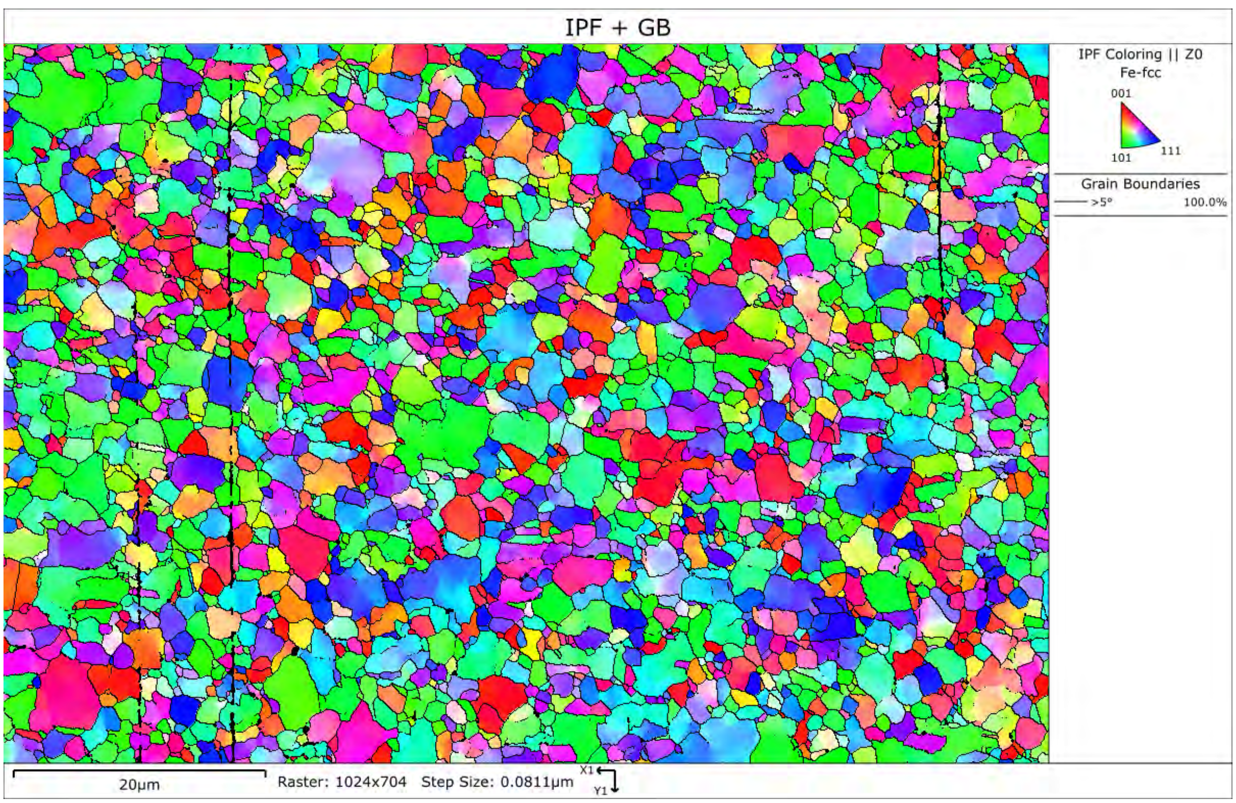

Figure 7.48. Inverse Pole Function - Z Images for L02: Condition C07, Sample SS04

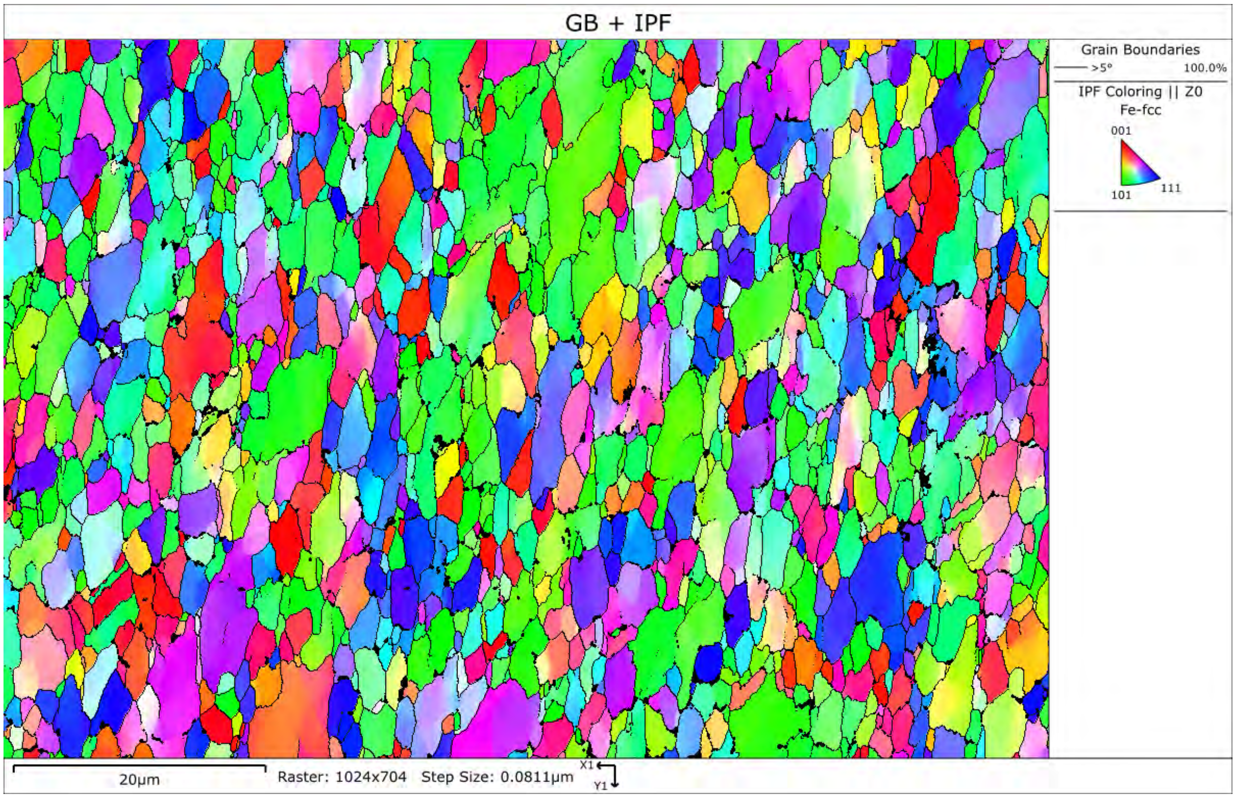

Figure 7.49. Inverse Pole Function - Z Images for L02: Condition C08, Sample SS05

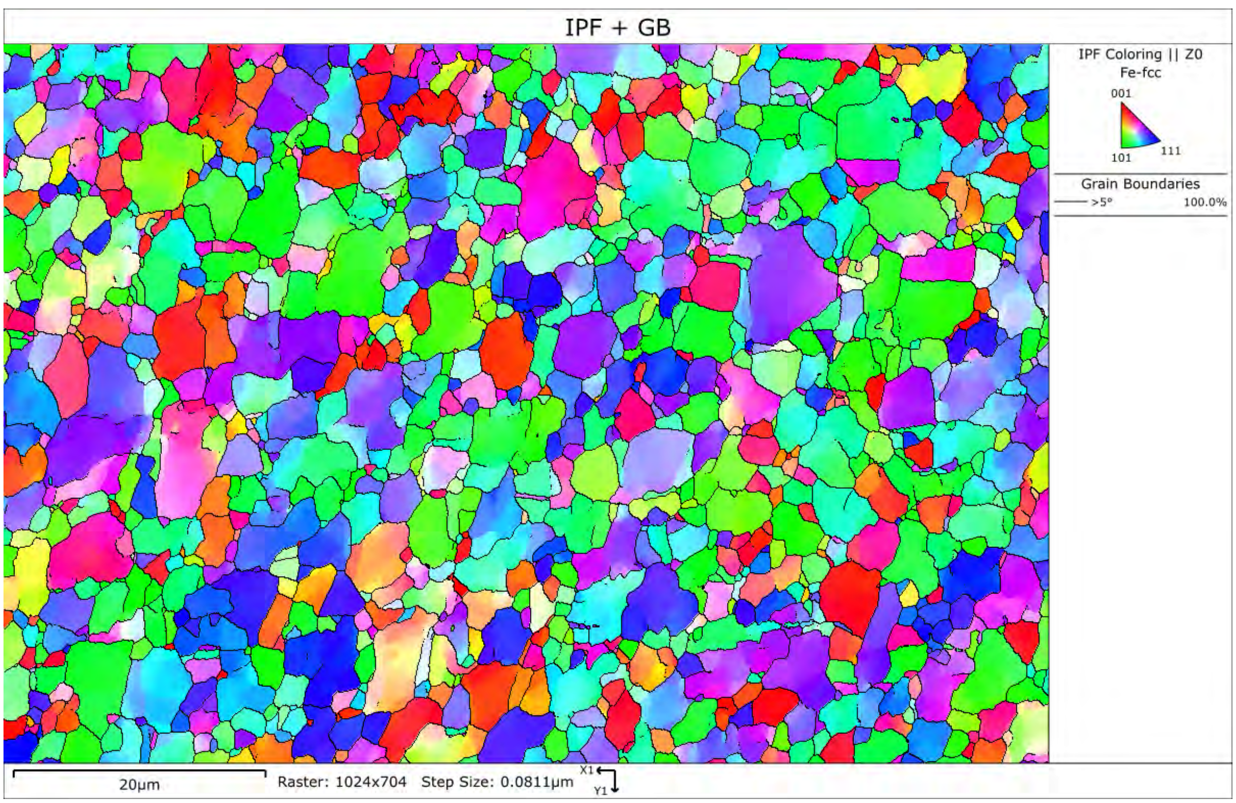

Figure 7.50. Inverse Pole Function - Z Images for L02: Condition C09, Sample SS06

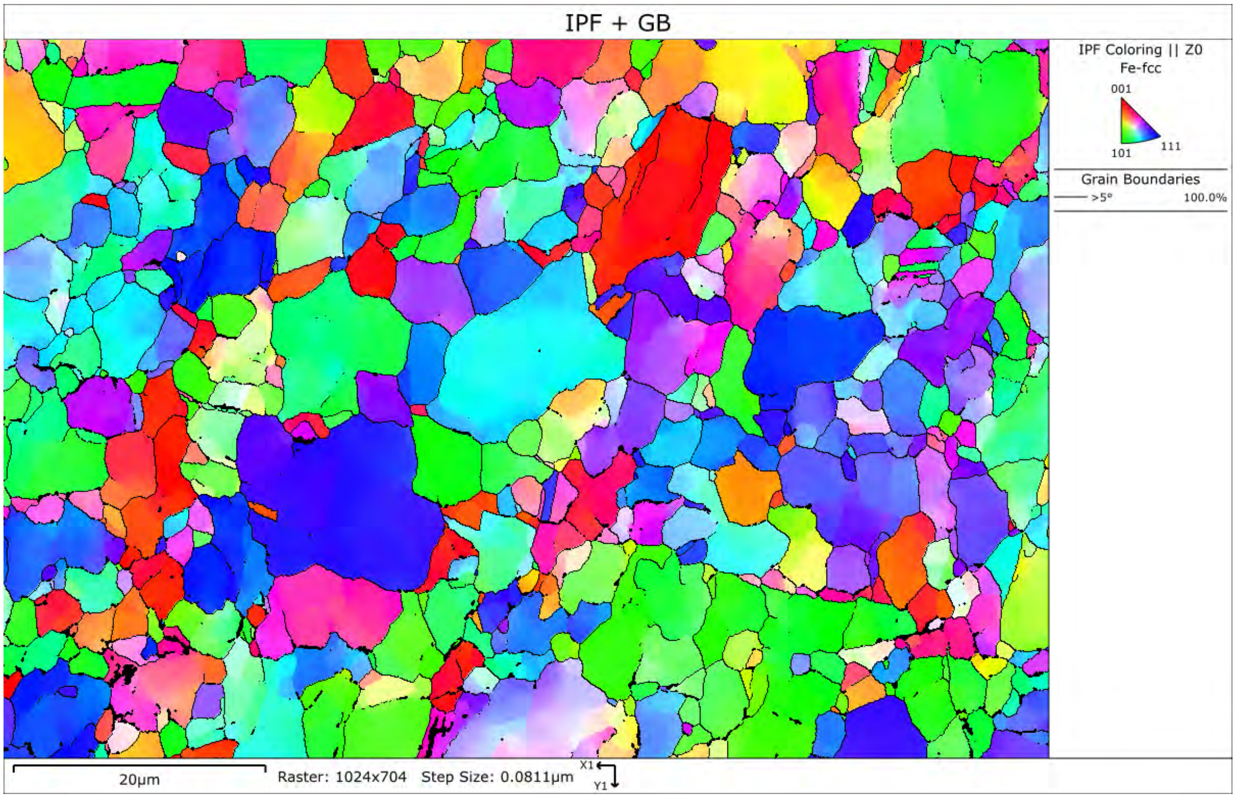

Figure 7.51. Inverse Pole Function - Z Images for L02: Condition C10, Sample SS07

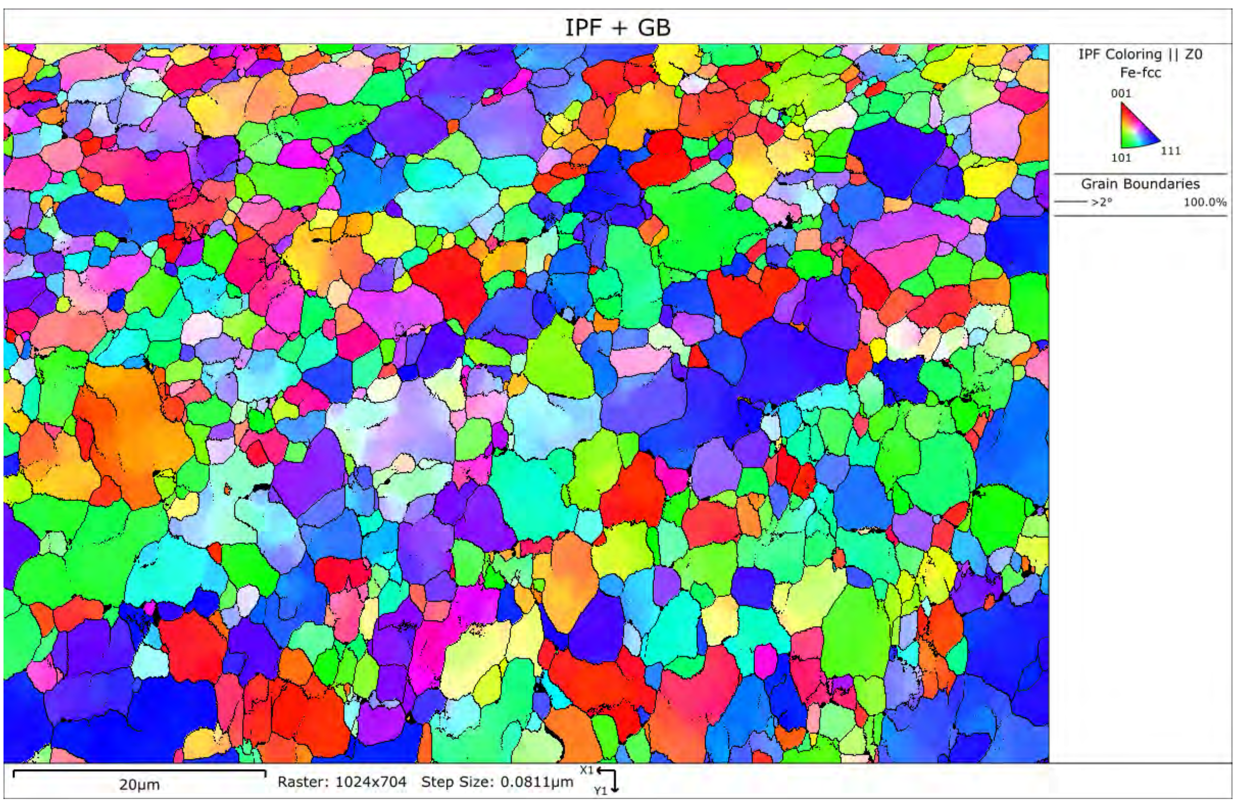

Figure 7.52. Inverse Pole Function - Z Images for L02: Condition C11, Sample SS08

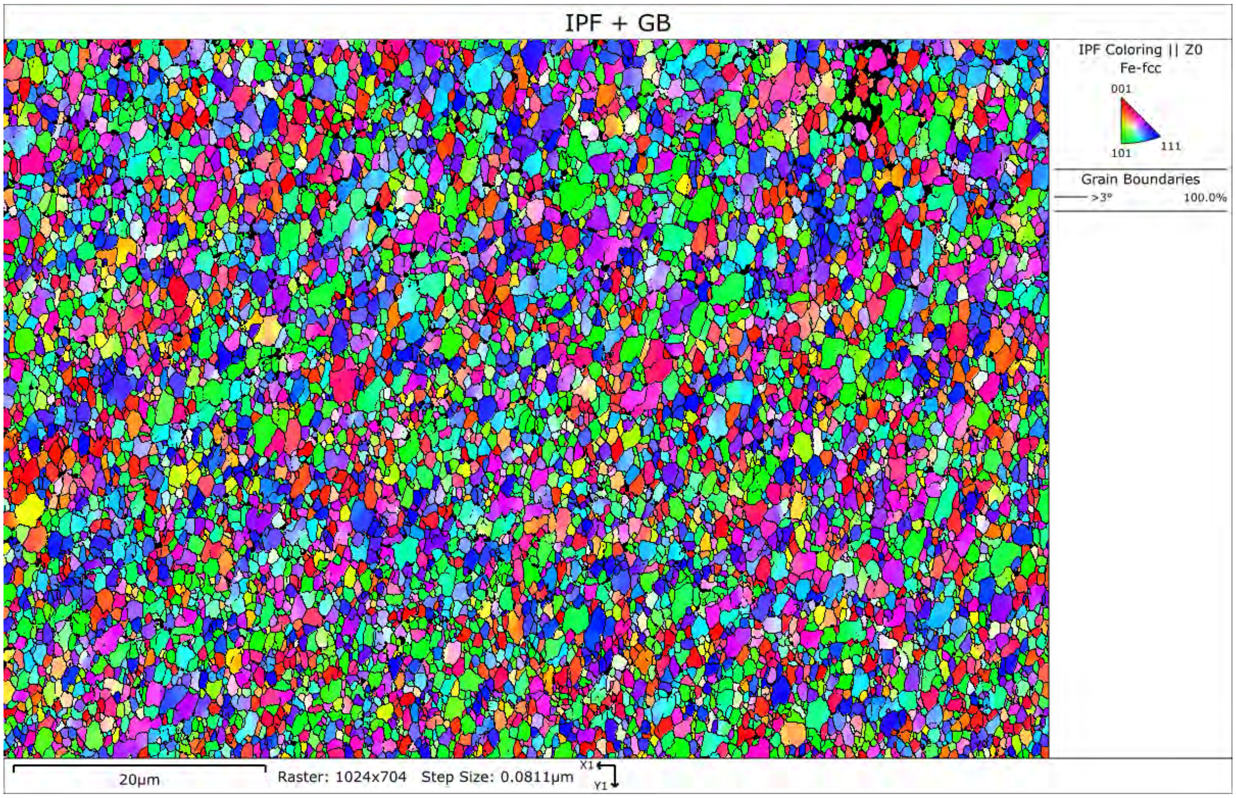

Figure 7.53. Inverse Pole Function - Z Images for L02: Condition C02, Sample SS28

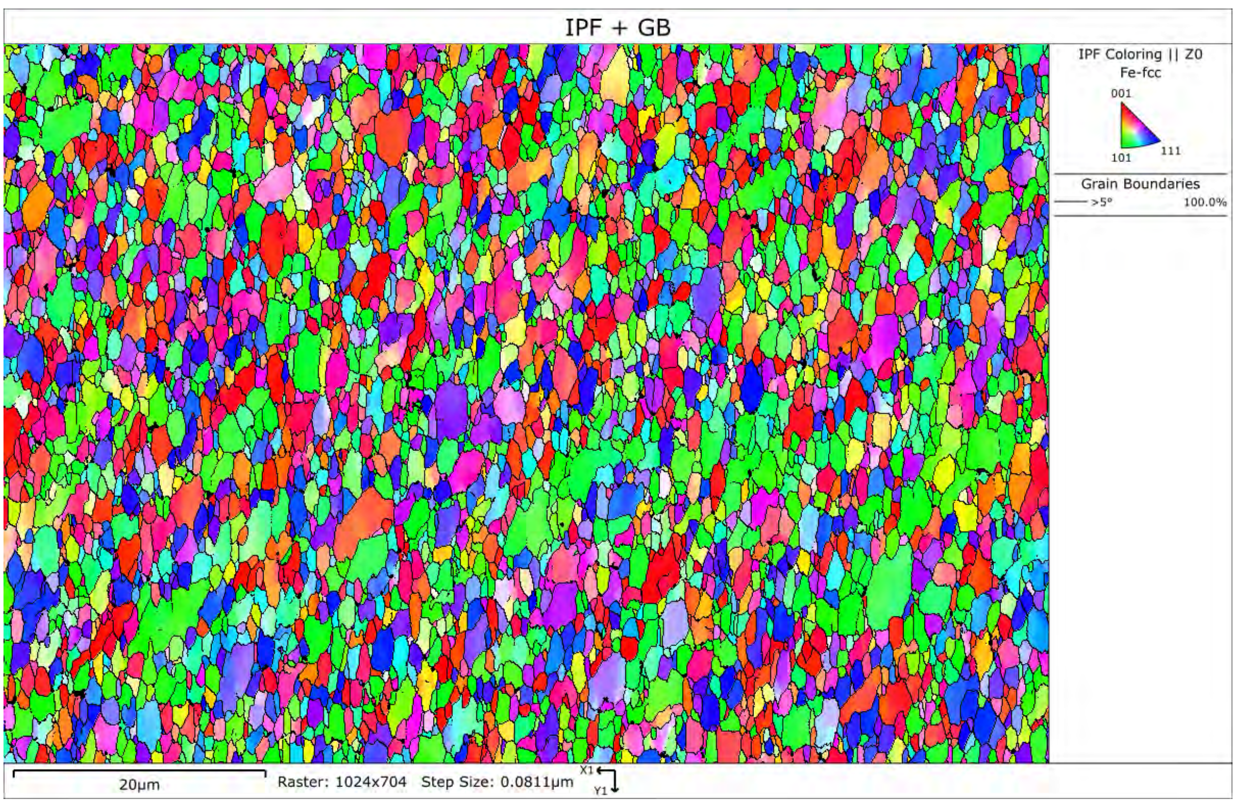

Figure 7.54. Inverse Pole Function - Z Images for L02: Condition C01, Sample SS31

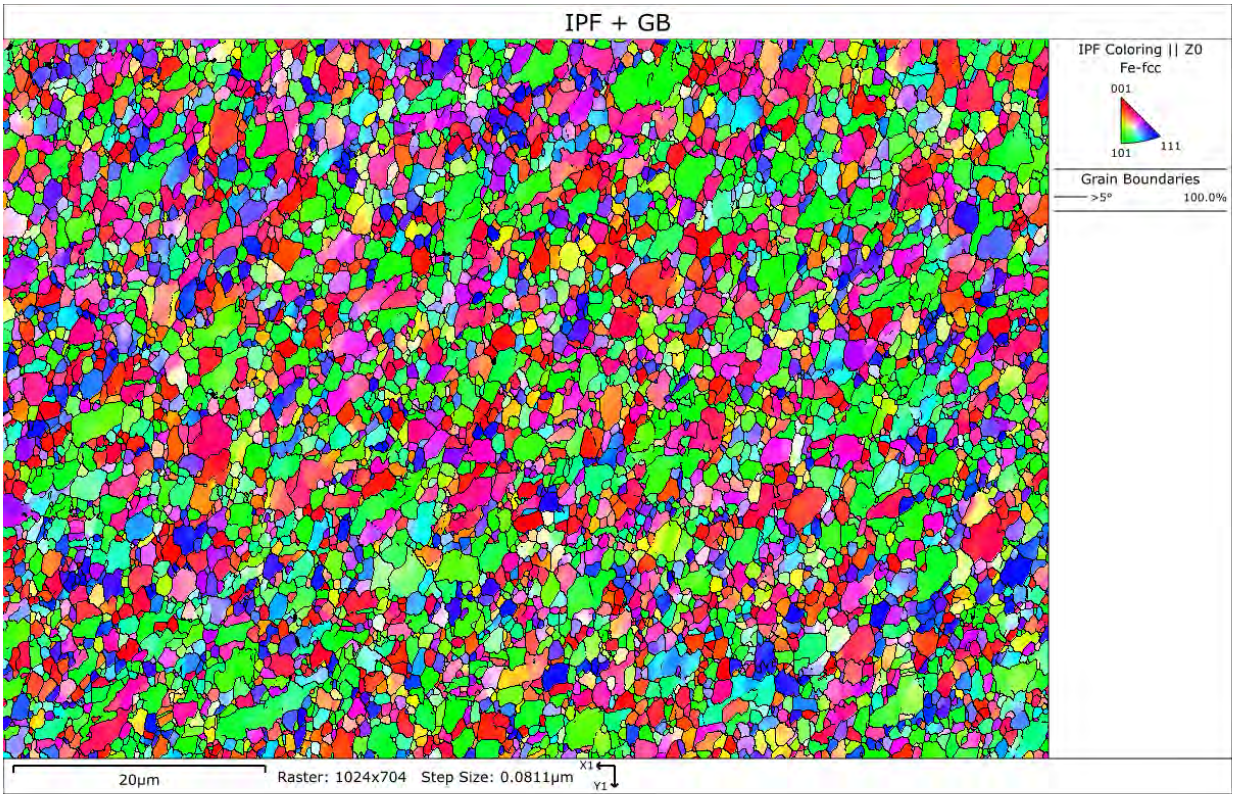

Figure 7.55. Inverse Pole Function - Z Images for L02: Condition C04, Sample SS32

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Attachment B: MCPC Round 1 Material Characterization Results for Modality HARDNESS

A Ortiz
A Guzman
DR Todd
O Linsuain
K Nwe

Attachment B contains 23 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

Two instruments were utilized in testing of specimens described in this attachment. Specifications are provided below. The first listed unit was used to test all specimens, and results listed in the main report are from this unit. The second instrument was purchased after data was collected with the first unit, and a single specimen was polished and retested. Comparison of data from both instruments on the same specimen show equivalent results. The unit used to collect specific data was identified in dataset metadata.

Hardness Tester Model CM-700AT

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

Hardness Tester Model CM-802AT

- Company Name: Sun-Tec Corporation
- Division: Clark
- MOD: CM ARS20 V/K
- SER#: CM222389

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.

- crop-{7, 8, 9, 10, 11}mm (VISUALIZATION Directory) The listed sub-directories display a graph of the stir region cropped to the indicated width in millimeters. These cropped images are suitable for constructing image collages (more on these later).

- {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. This directory contains image collages, tabulation of information, and other resources covering data from multiple datasets. 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.

- crop-{7, 8, 9, 10, 11}mm: These directories contain collages of the hardness data imagery, where the width of the graphics showing the stir zone varies from 7-11 mm. Companion collages are available for optical and SEM imagery. Collages are provided on 2x3 and 4x3 grids. Color scale bars are not provided. The scale is the same across all datasets and can be obtained from individual imagery.

5.0 Notes and Comments

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

1. Hardness data for the control sample (Dataset ID SS36_22782) was not collected. A file stating this is provided in the relevant directory.

2. Dataset SS01_22741a contains measurement results using the micro hardness tester model CM-802AT. The results in this directory shows equivalent results to the earlier CM-700AT tester located in Dataset SS01_22741 for the same specimen. Either set of results can be used, but it is recommended to use Dataset SS01_22741 for consistency with other tests in this Round.

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
SS01	C03	269.27	13.87	267.36	11.93	260.98
SS01*	C03	277.89	16.87	269.46	10.57	271.67
SS02	C05	269.49	16.88	263.69	9.58	265.00
SS03	C06	252.52	15.59	259.24	7.08	260.98
SS04	C07	253.81	17.56	242.69	11.22	249.46
SS05	C08	240.29	16.51	234.03	9.49	233.89
SS06	C09	240.06	15.98	231.38	11.51	233.22
SS07	C10	222.76	17.17	209.96	10.55	202.96
SS08	C11	220.22	19.28	204.78	8.46	206.27
SS28	C02	298.07	10.08	305.02	3.68	307.77
SS31	C01	271.86	10.60	274.94	8.28	281.14
SS32	C04	274.00	9.81	272.63	6.41	275.03

The entry with ”” next to the Sample ID in the above table was obtained with the second tester as described earlier.*

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. Note any figure that displays **MCPC0001_22741a** in the header was obtained with the second tester as described earlier. The ”a” appended at the end was to ensure unique naming of datasets. Finally, as noted in the main report, the spacing between measurement points is smaller for two datasets. This is clearly evident for Sample SS02 and SS05 in the graphics.

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.

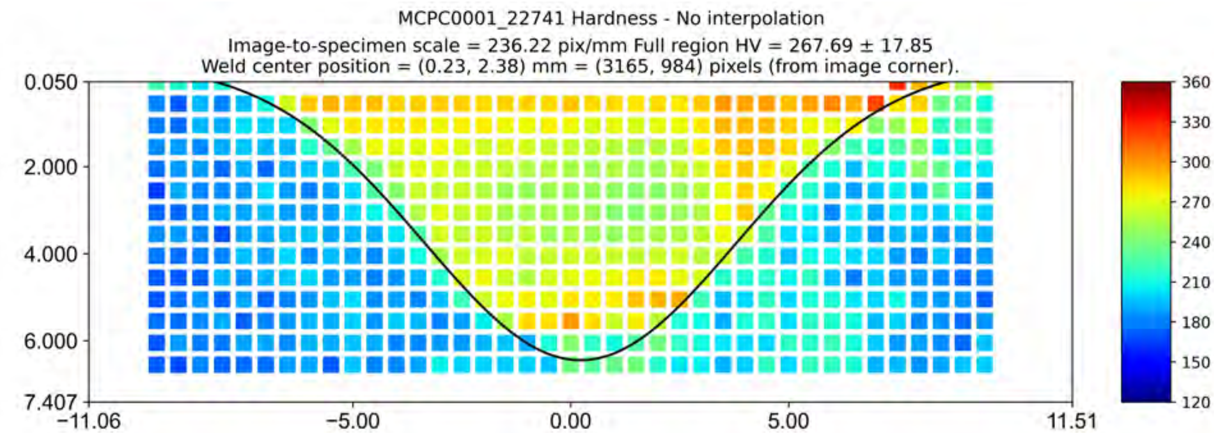

Figure 7.1. Full Stir Region Region with no Interpolation: Condition C03, Sample SS01

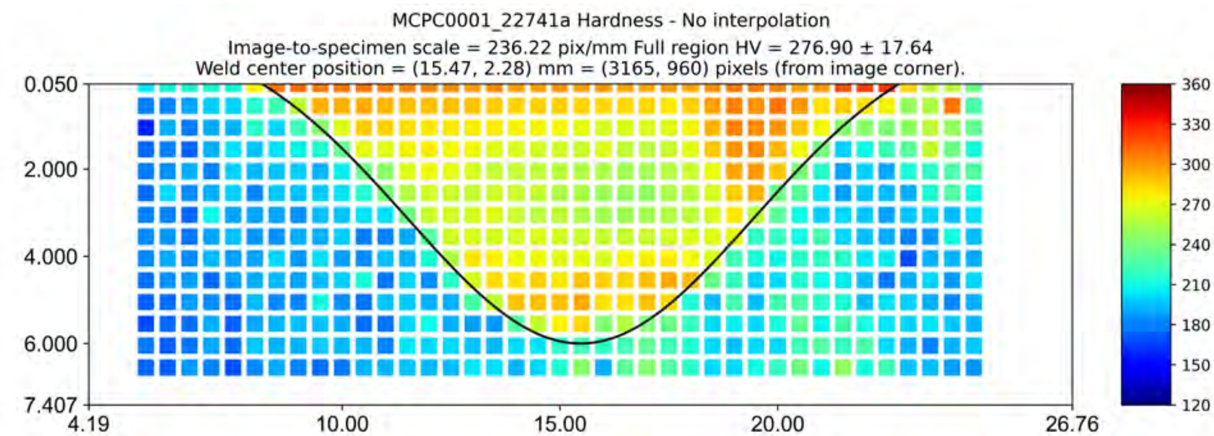

Figure 7.2. Full Stir Region Region with no Interpolation: Condition C03, Sample SS01

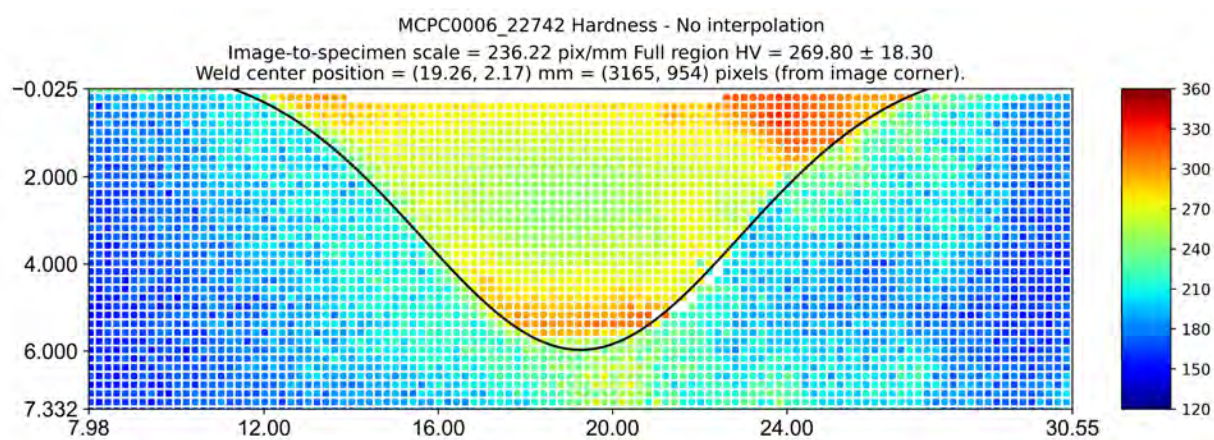

Figure 7.3. Full Stir Region Region with no Interpolation: Condition C05, Sample SS02

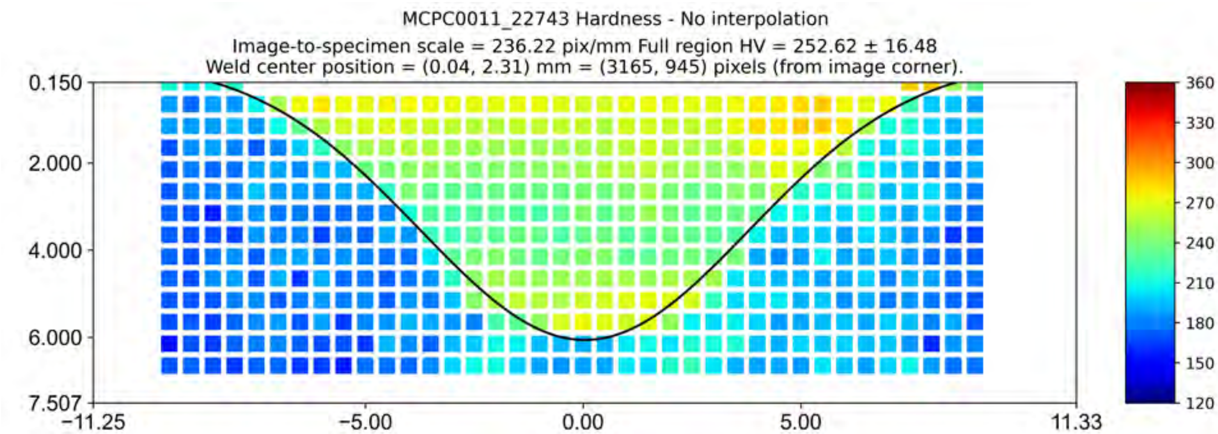

Figure 7.4. Full Stir Region Region with no Interpolation: Condition C06, Sample SS03

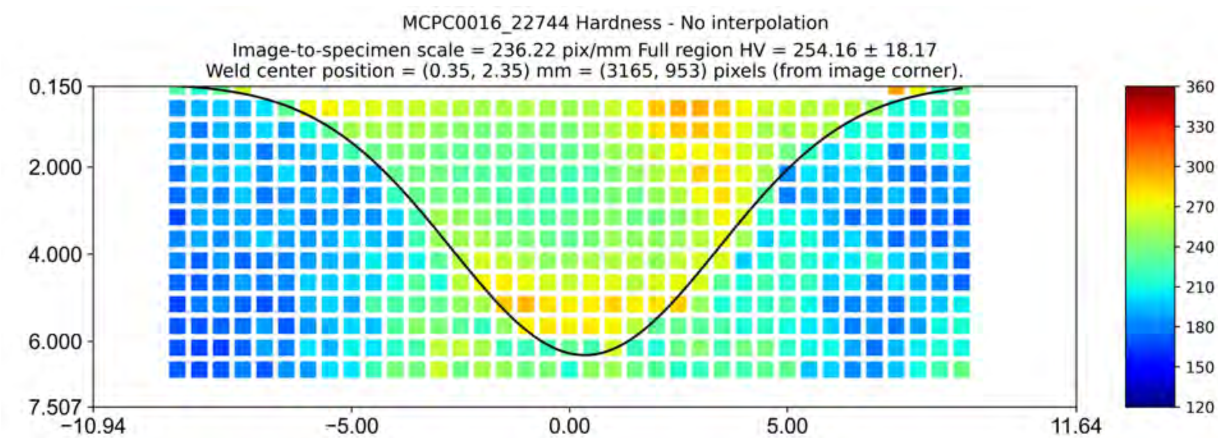

Figure 7.5. Full Stir Region Region with no Interpolation: Condition C07, Sample SS04

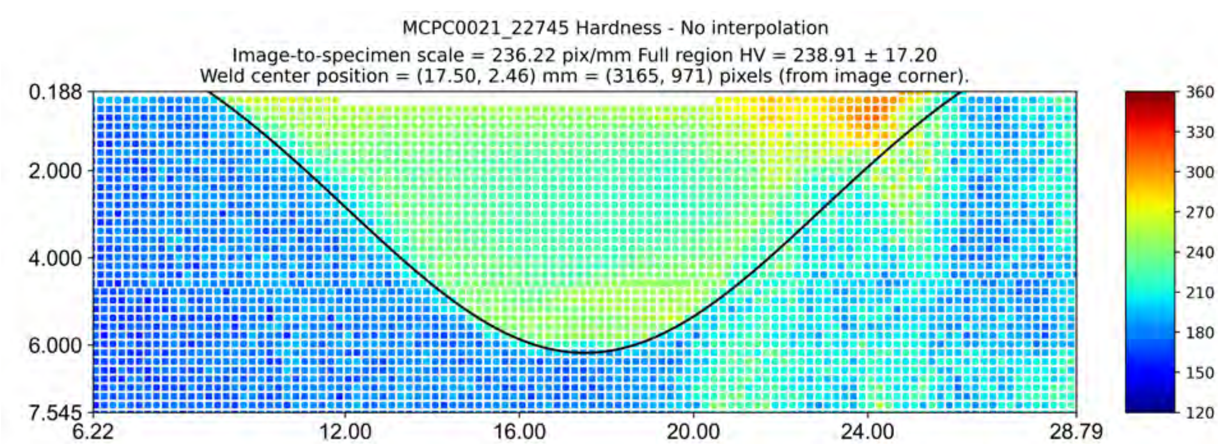

Figure 7.6. Full Stir Region Region with no Interpolation: Condition C08, Sample SS05

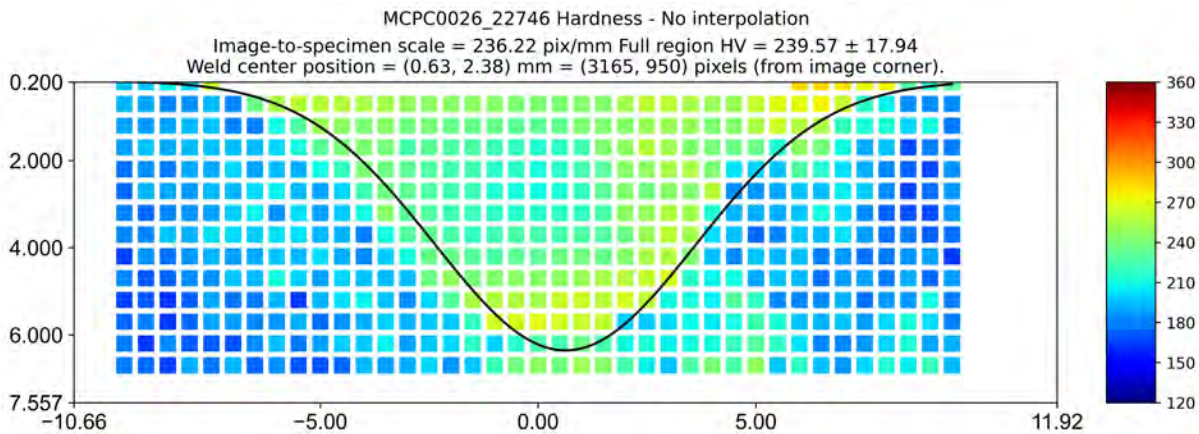

Figure 7.7. Full Stir Region Region with no Interpolation: Condition C09, Sample SS06

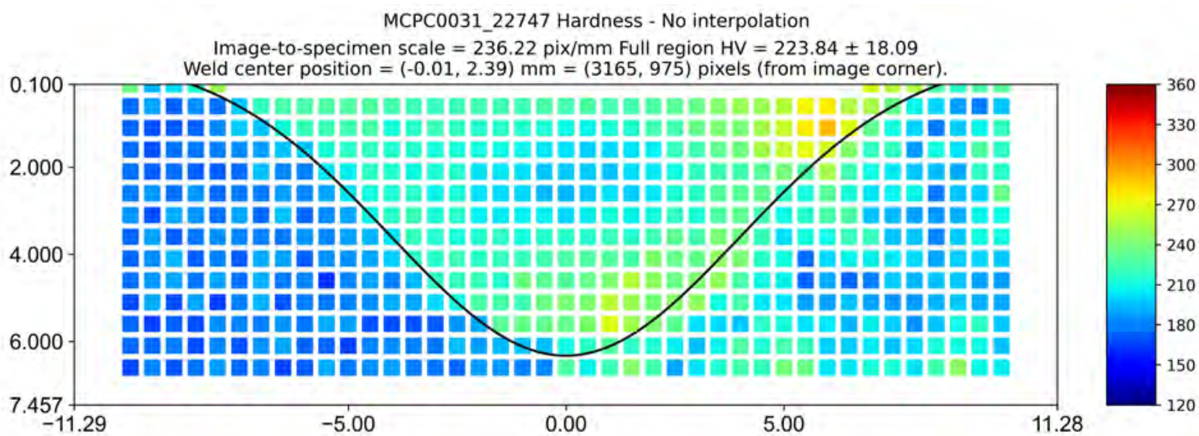

Figure 7.8. Full Stir Region Region with no Interpolation: Condition C10, Sample SS07

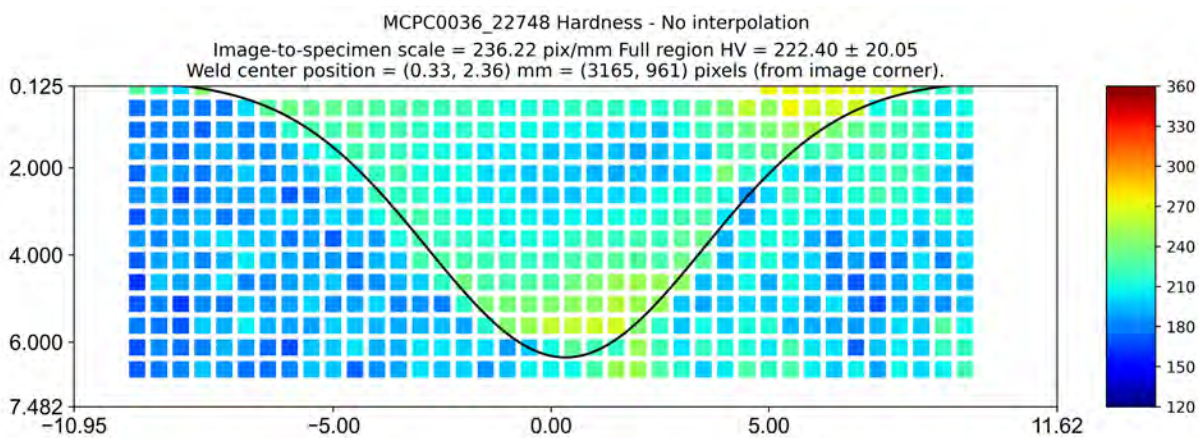

Figure 7.9. Full Stir Region Region with no Interpolation: Condition C11, Sample SS08

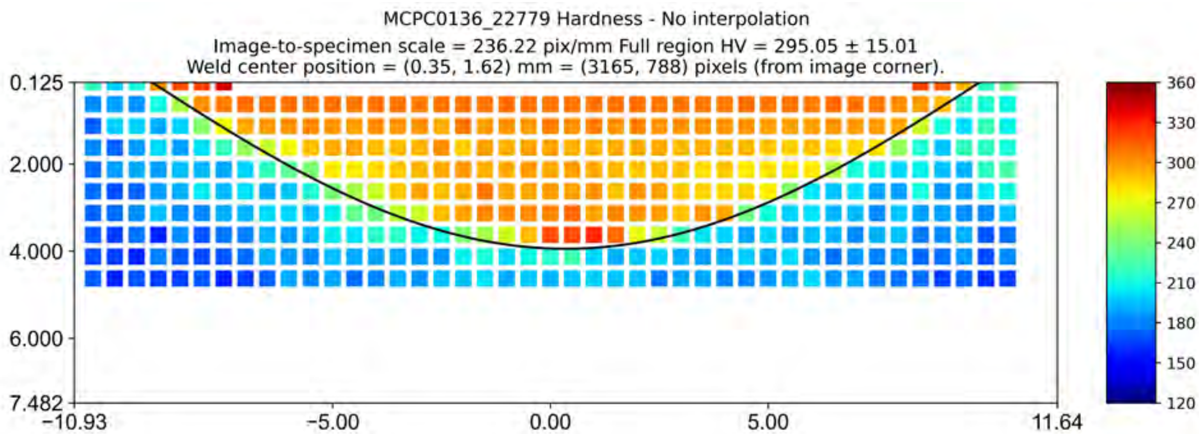

Figure 7.10. Full Stir Region Region with no Interpolation: Condition C02, Sample SS28

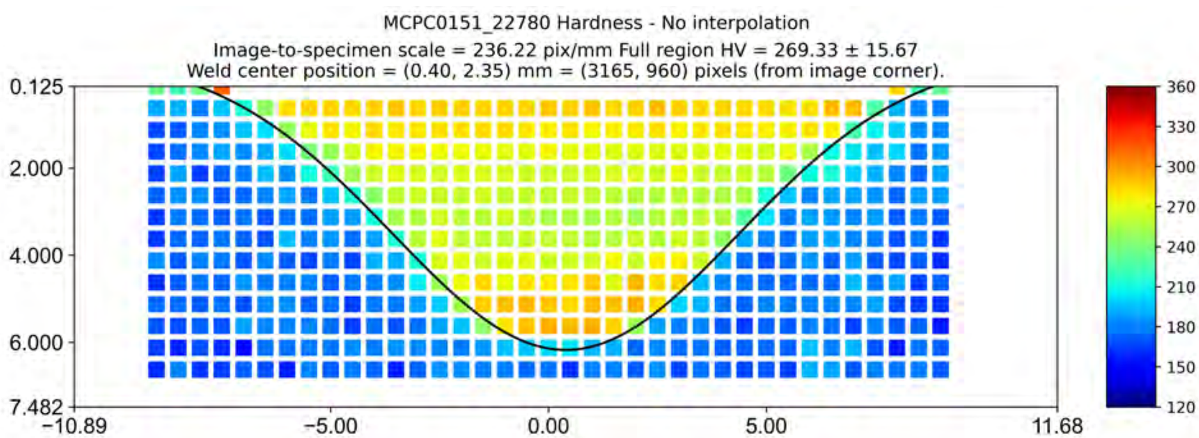

Figure 7.11. Full Stir Region Region with no Interpolation: Condition C01, Sample SS31

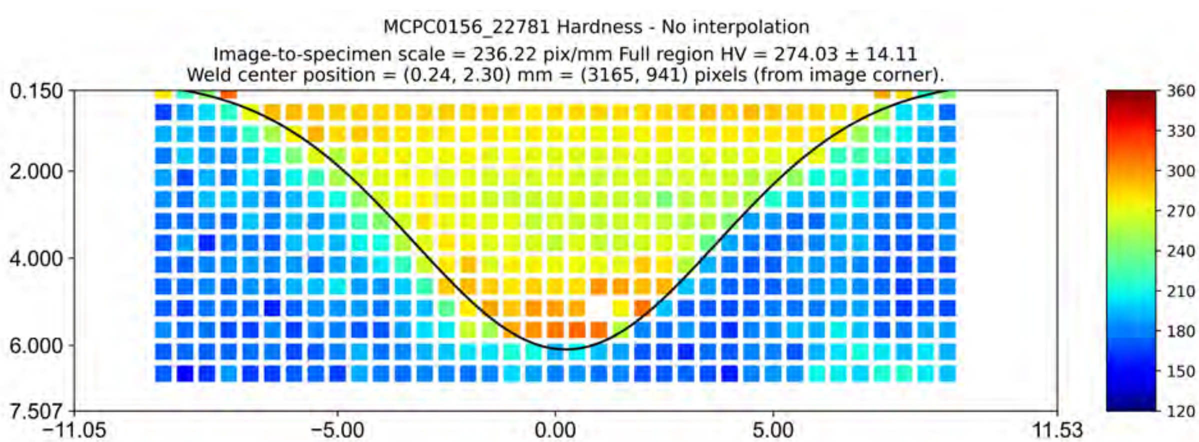

Figure 7.12. Full Stir Region Region with no Interpolation: Condition C04, Sample SS32

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.

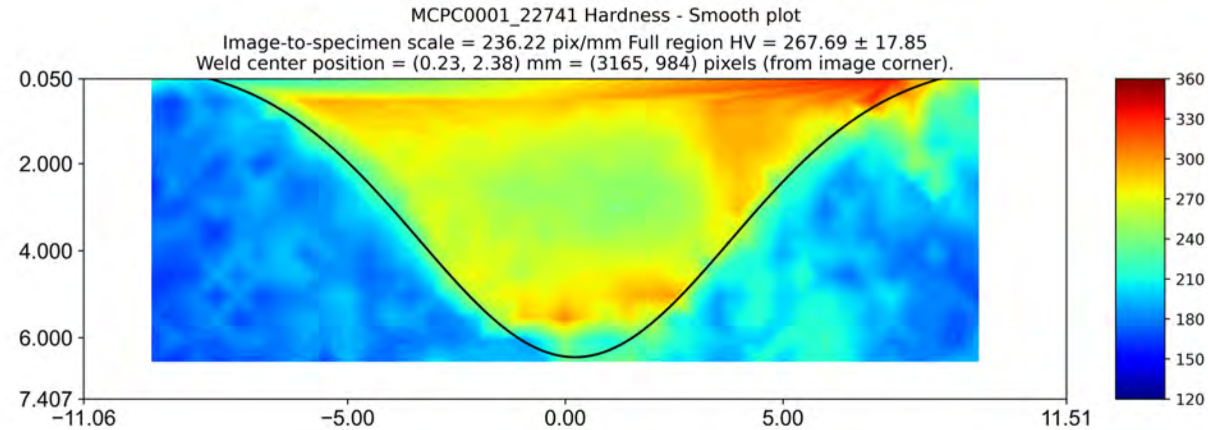

Figure 7.13. Full Stir Region Region with Interpolation: Condition C03, Sample SS01

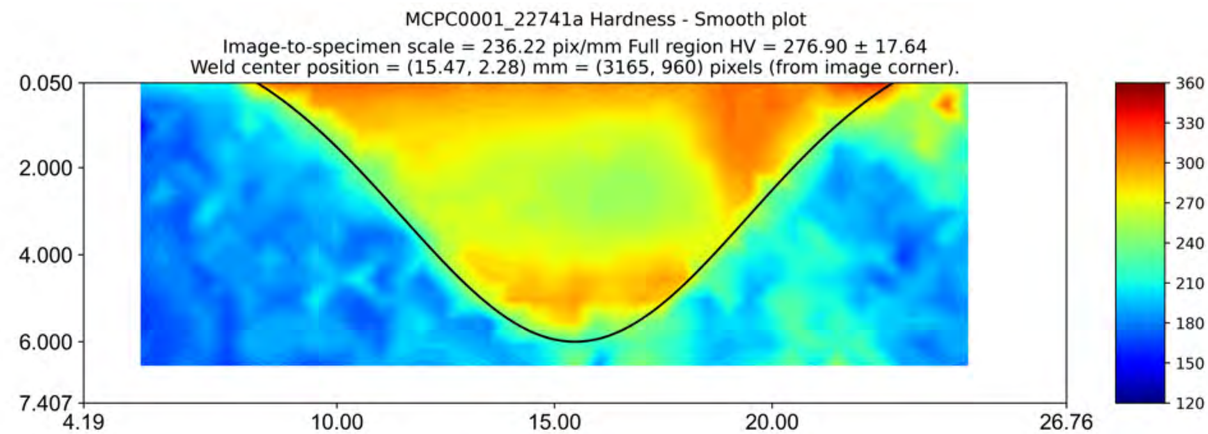

Figure 7.14. Full Stir Region Region with Interpolation: Condition C03, Sample SS01

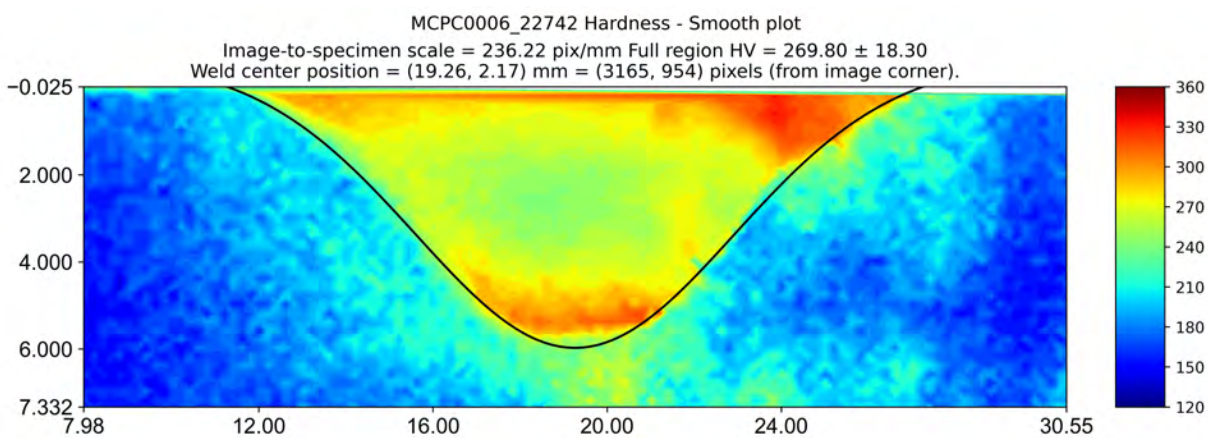

Figure 7.15. Full Stir Region Region with Interpolation: Condition C05, Sample SS02

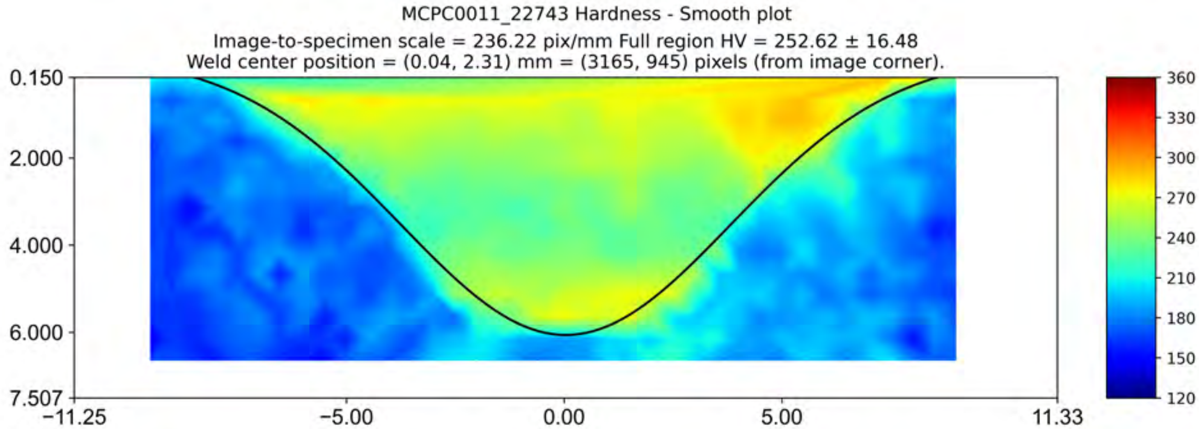

Figure 7.16. Full Stir Region Region with Interpolation: Condition C06, Sample SS03

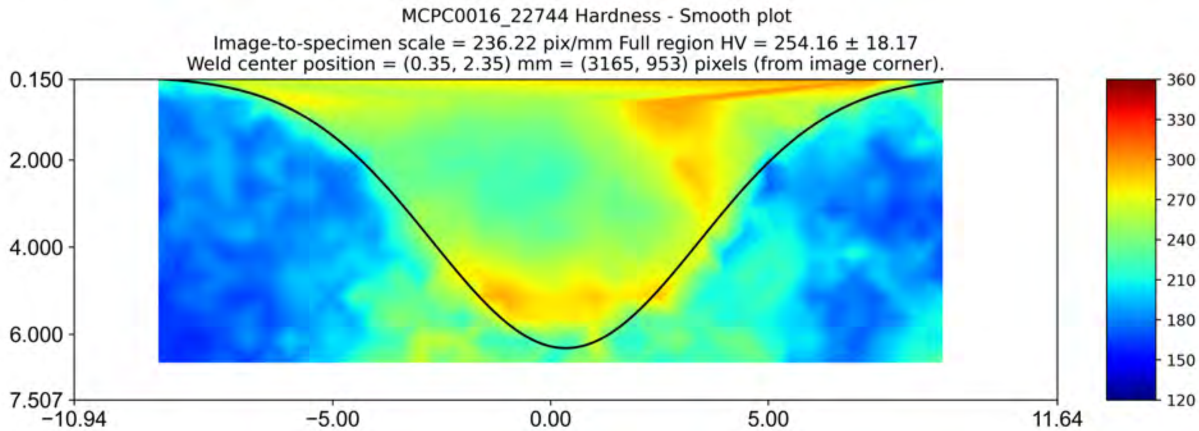

Figure 7.17. Full Stir Region Region with Interpolation: Condition C07, Sample SS04

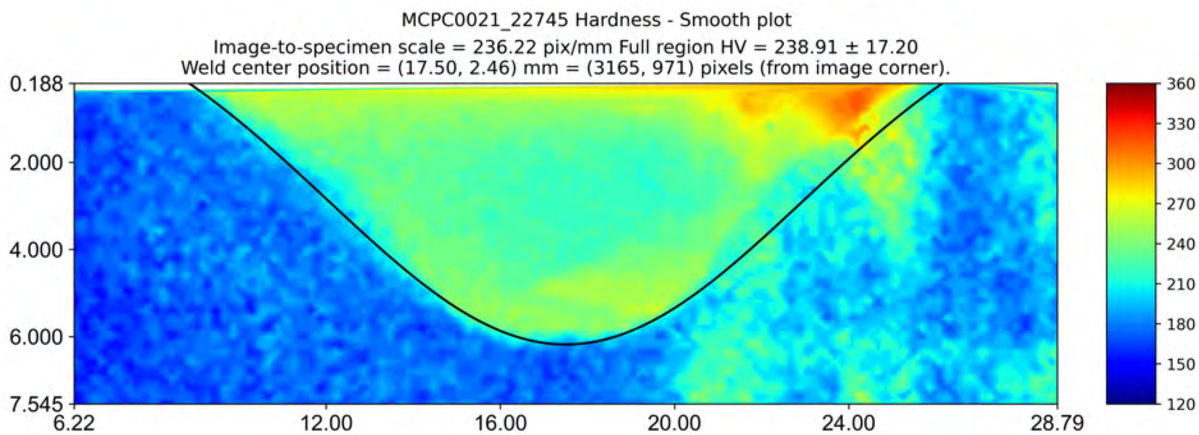

Figure 7.18. Full Stir Region Region with Interpolation: Condition C08, Sample SS05

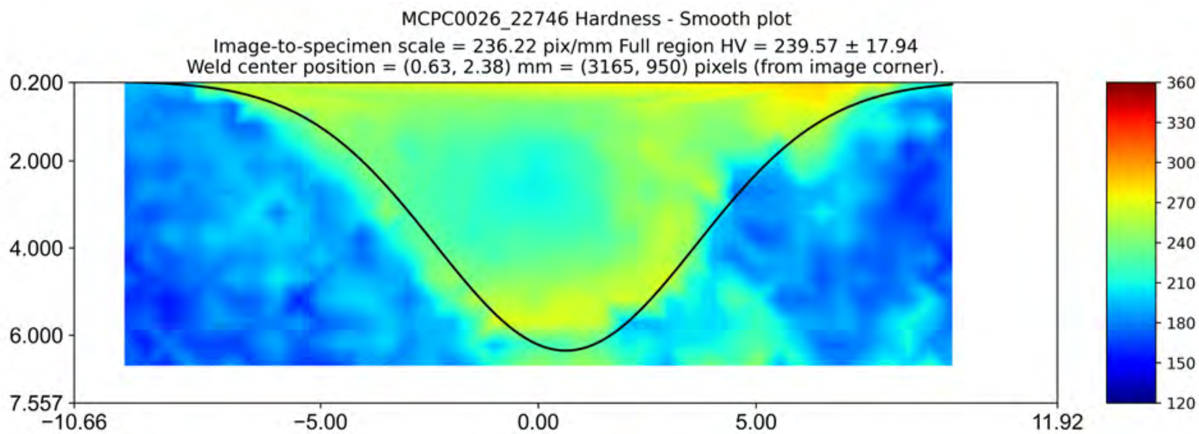

Figure 7.19. Full Stir Region Region with Interpolation: Condition C09, Sample SS06

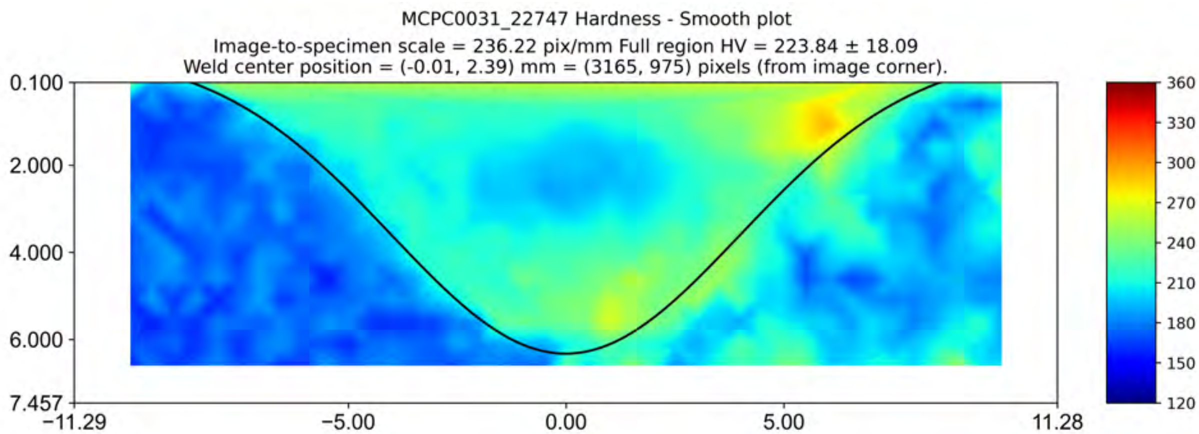

Figure 7.20. Full Stir Region Region with Interpolation: Condition C10, Sample SS07

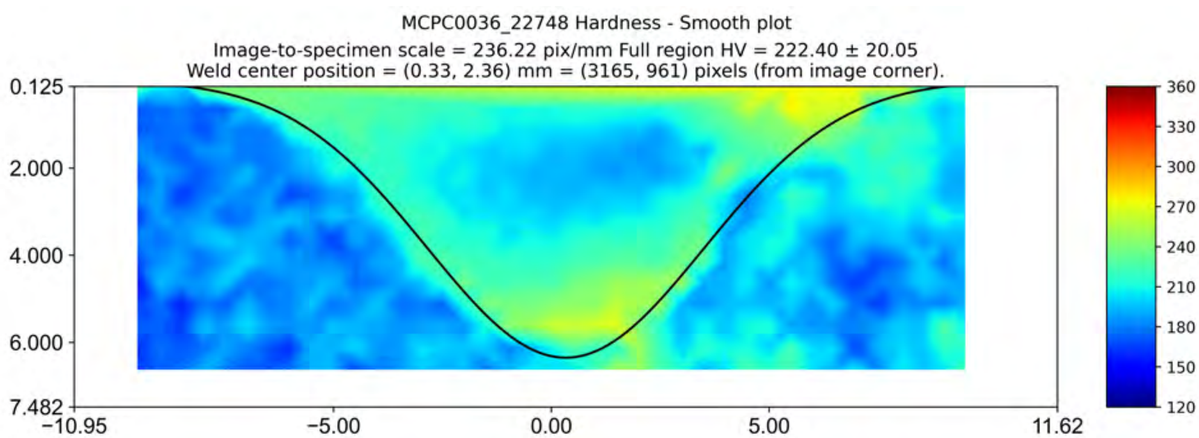

Figure 7.21. Full Stir Region Region with Interpolation: Condition C11, Sample SS08

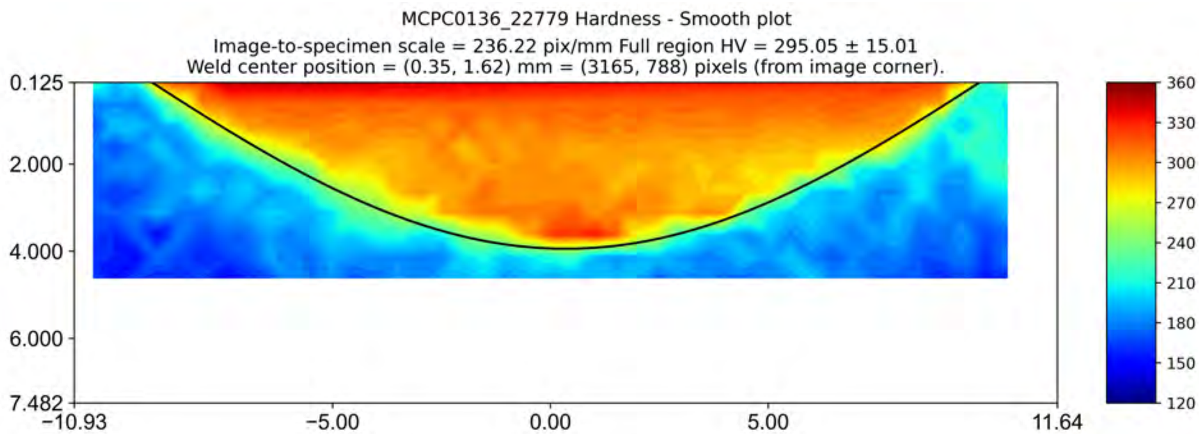

Figure 7.22. Full Stir Region Region with Interpolation: Condition C02, Sample SS28

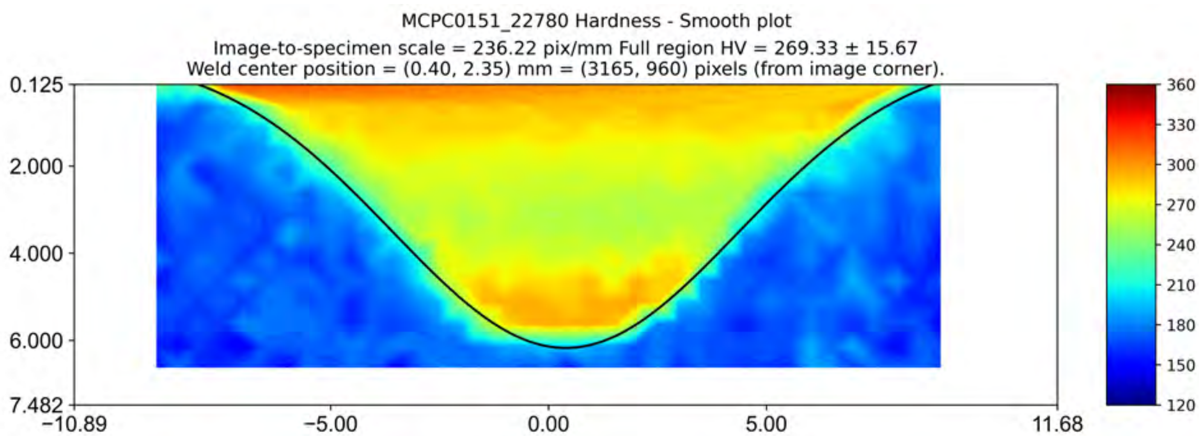

Figure 7.23. Full Stir Region Region with Interpolation: Condition C01, Sample SS31

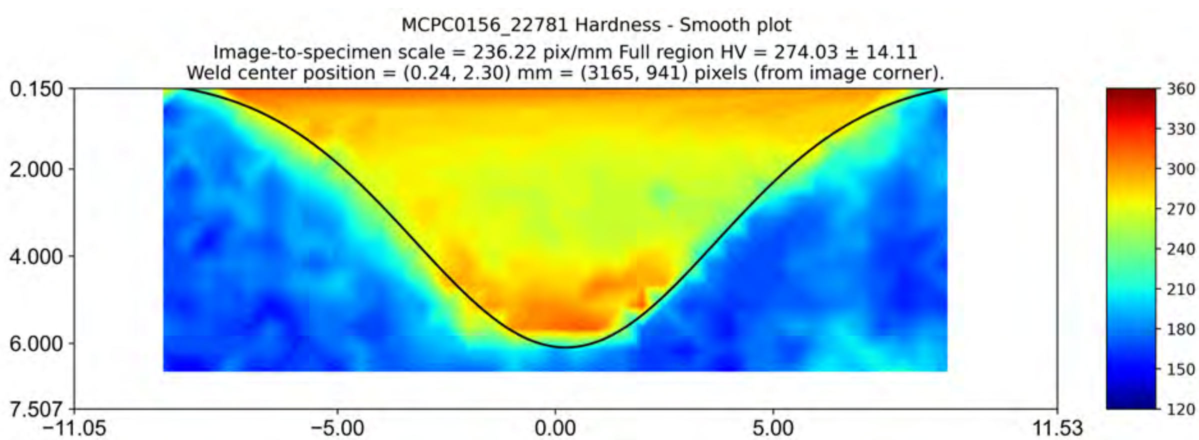

Figure 7.24. Full Stir Region Region with Interpolation: Condition C04, Sample SS32

7.3 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 Table 6.1. instead of the value for the small window near the center that is listed in Table 3 of the main report.

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

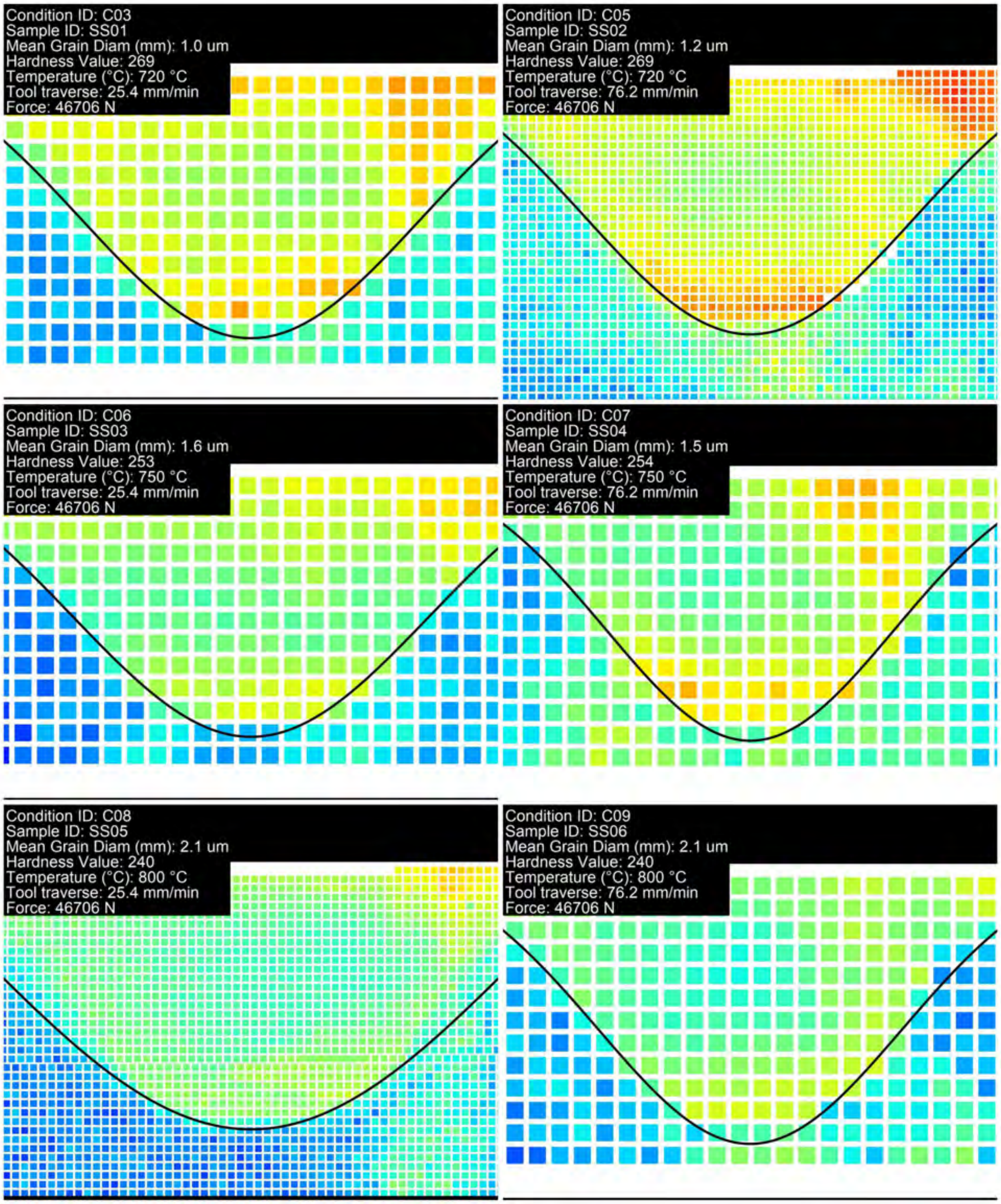

Figure 7.25. Stir Region Collage: Image number 1

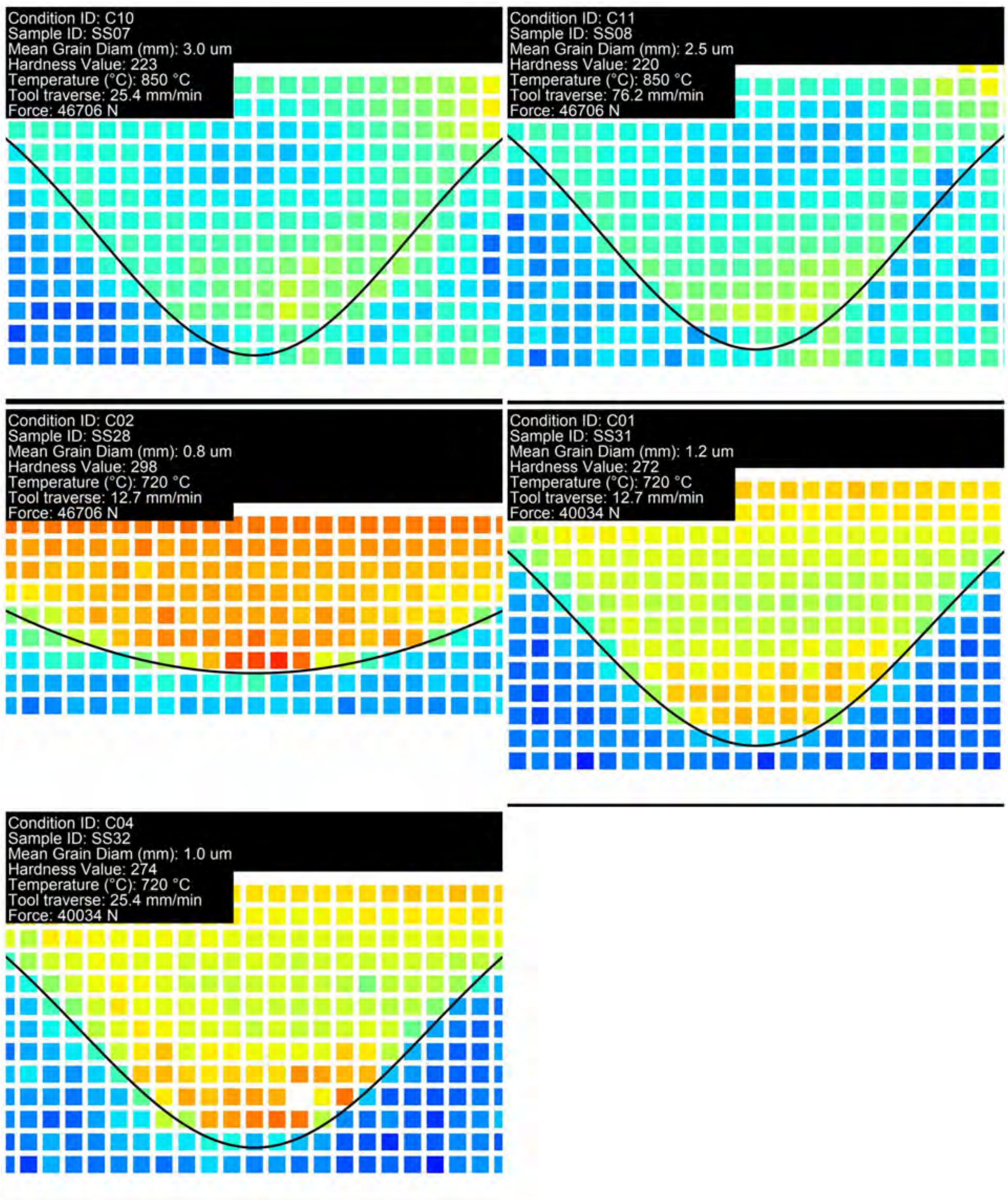

Figure 7.26. Stir Region Collage: Image number 2

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Attachment C: MCPC Round 1 Material Characterization Results for Modality OPTICAL

A Ortiz K Nwe
DR Todd A Guzman

Attachment C contains 26 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. This is the image of primary interest. In several instances a second etched image is available at 50x magnification. And in a few cases 50x images with differential interference contrast (DIC) are provided. The three types of files have the following in their names:

- 10X_Nugget-Region-Etched
- 5X_Montage-Etched
- 5X_Montage-DIC

5.0 Notes and Comments

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

1. Imagery is provided in JPG format. Compression has resulted in loss of granularity impacting its usefulness for a machine learning application.

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 eleven 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.

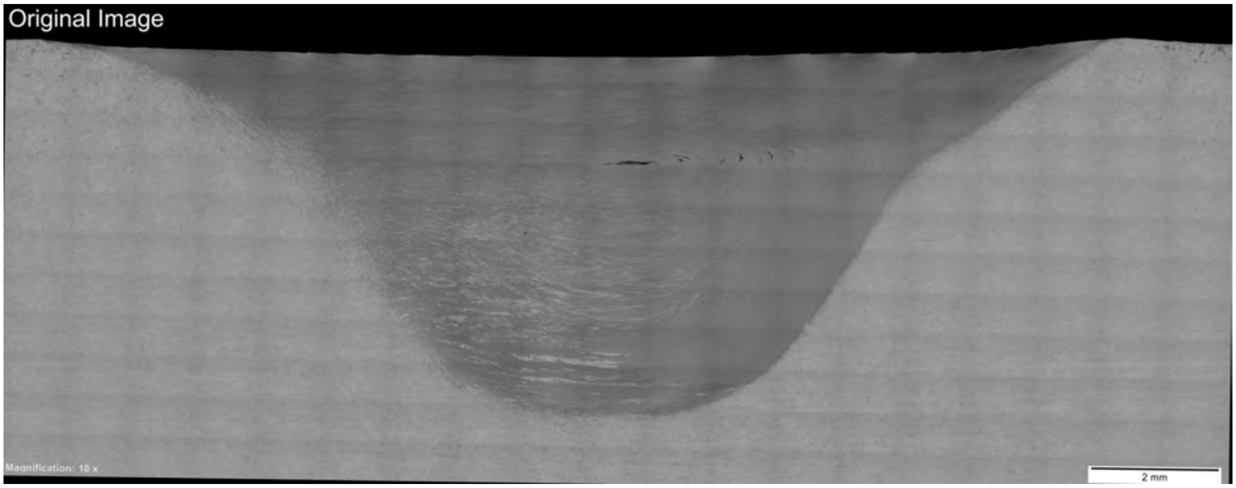

Figure 7.1. Optical Imagery at 100x Magnification: Condition C03, Sample SS01

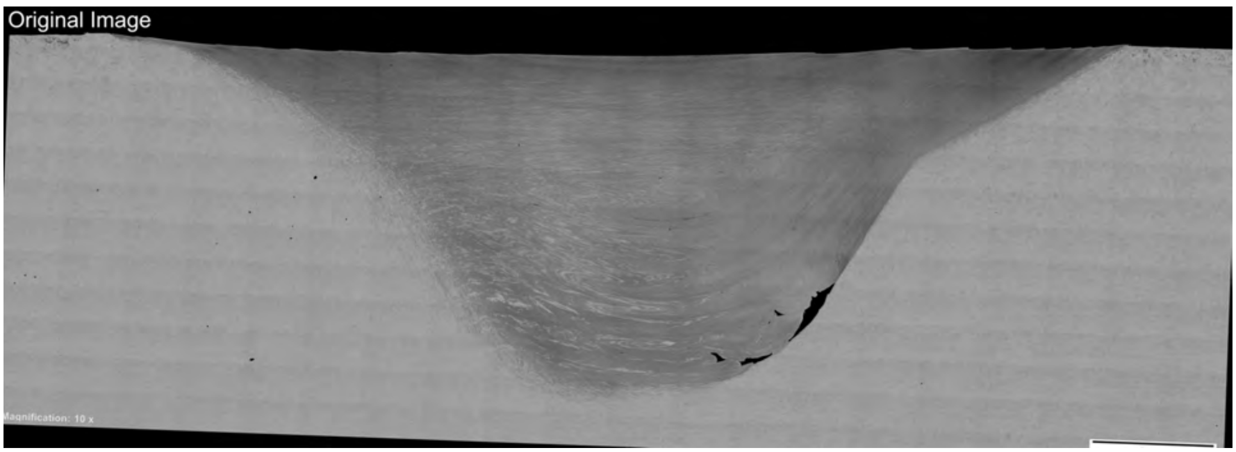

Figure 7.2. Optical Imagery at 100x Magnification: Condition C05, Sample SS02

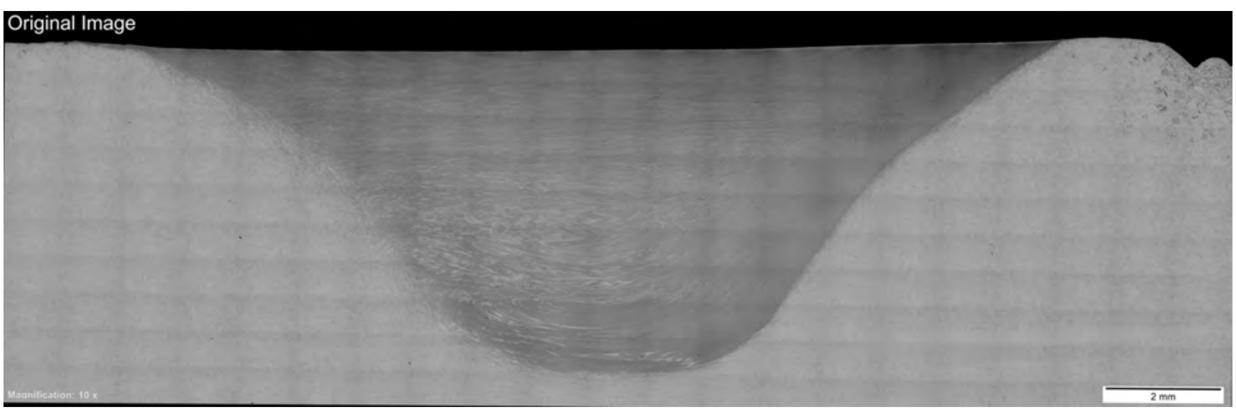

Figure 7.3. Optical Imagery at 100x Magnification: Condition C06, Sample SS03

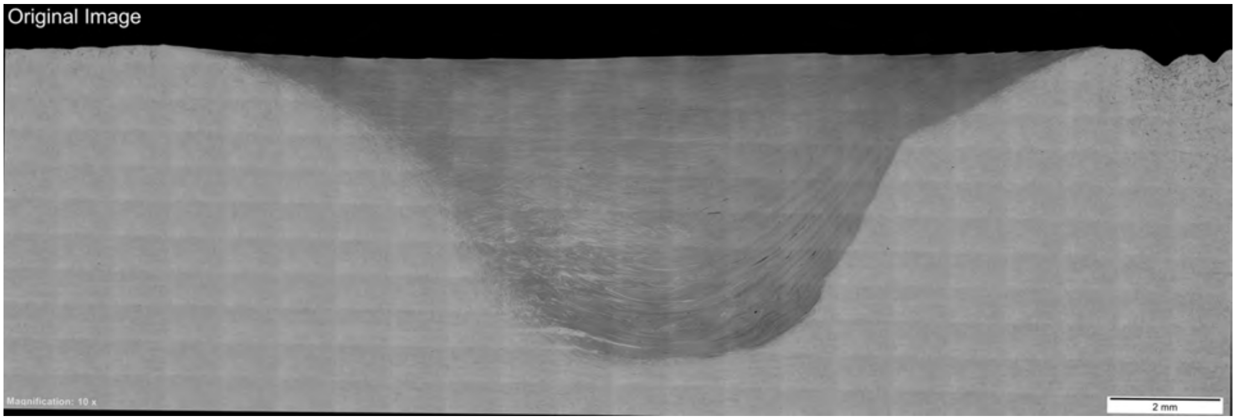

Figure 7.4. Optical Imagery at 100x Magnification: Condition C07, Sample SS04

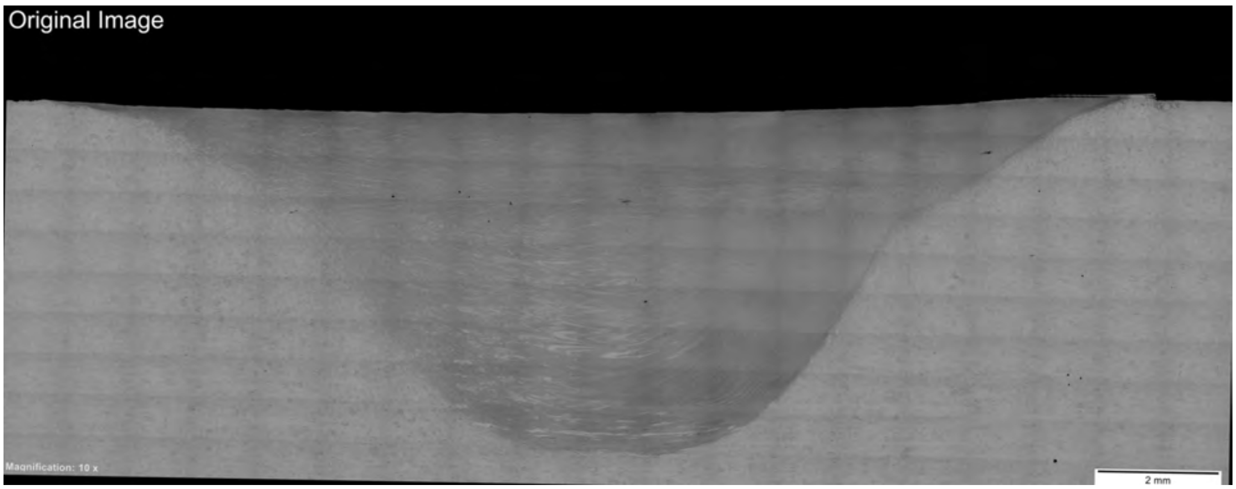

Figure 7.5. Optical Imagery at 100x Magnification: Condition C08, Sample SS05

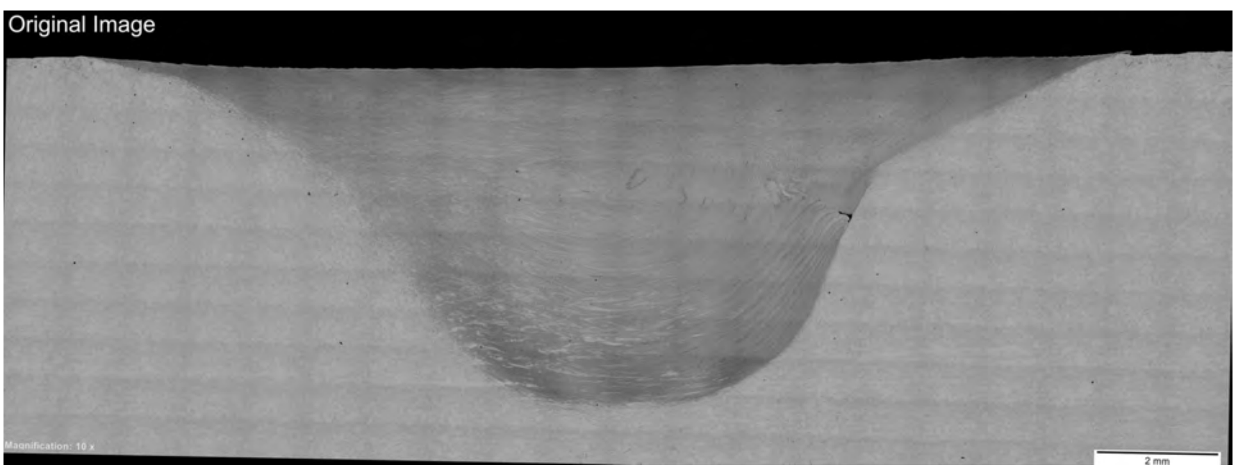

Figure 7.6. Optical Imagery at 100x Magnification: Condition C09, Sample SS06

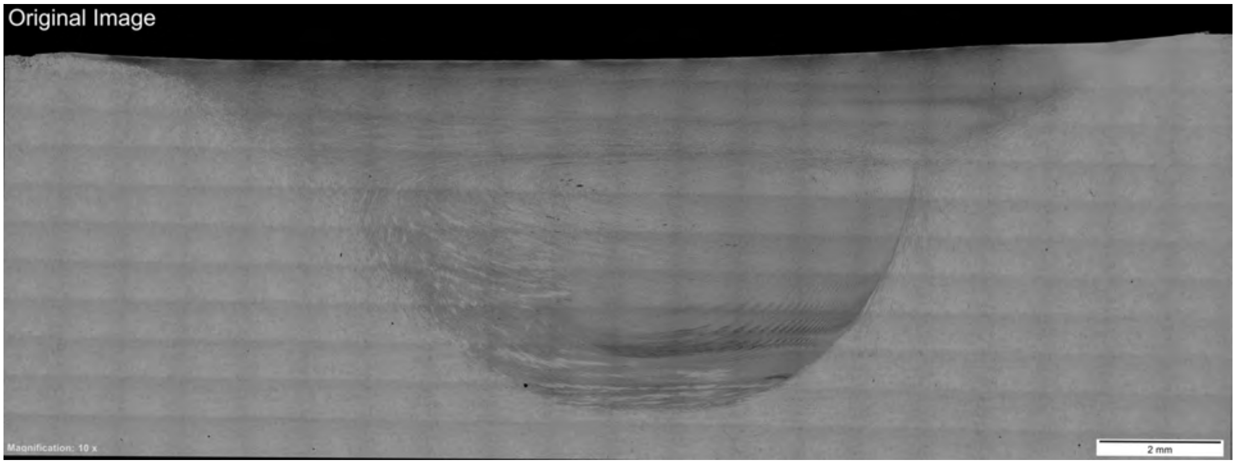

Figure 7.7. Optical Imagery at 100x Magnification: Condition C10, Sample SS07

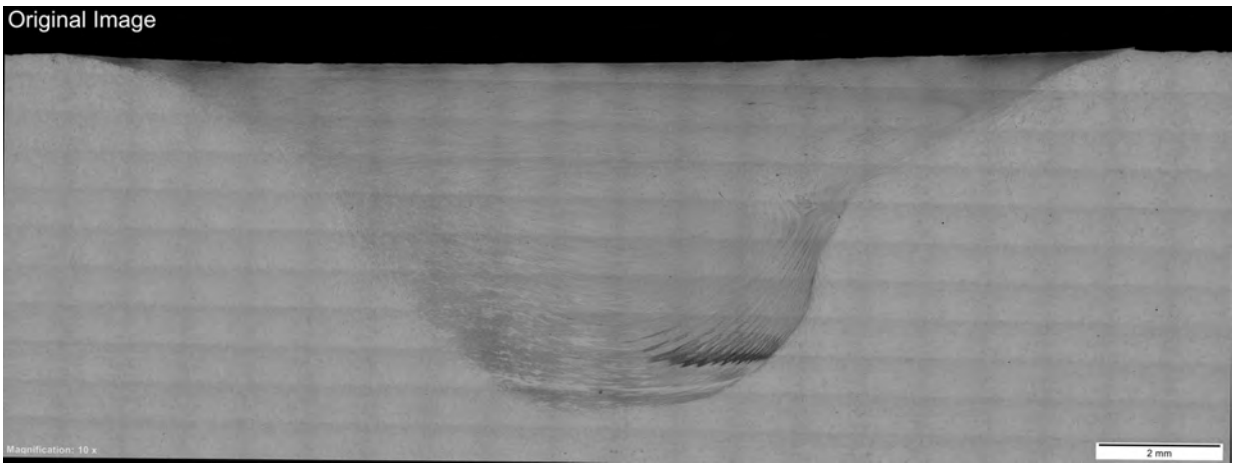

Figure 7.8. Optical Imagery at 100x Magnification: Condition C11, Sample SS08

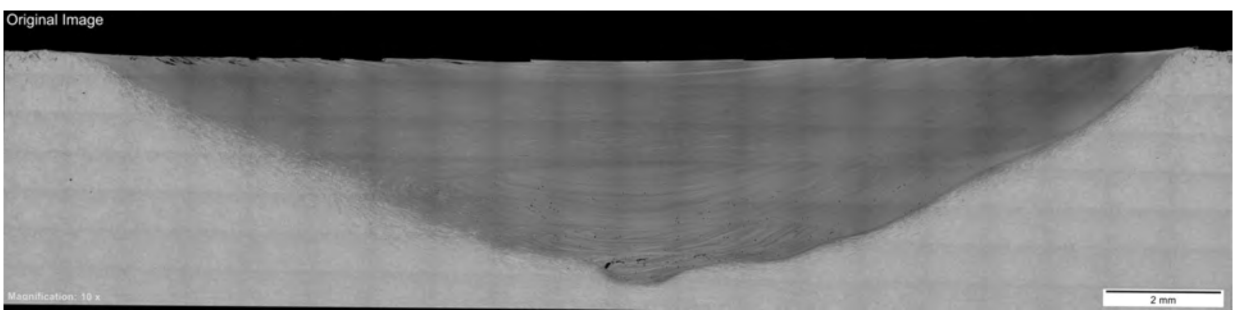

Figure 7.9. Optical Imagery at 100x Magnification: Condition C02, Sample SS28

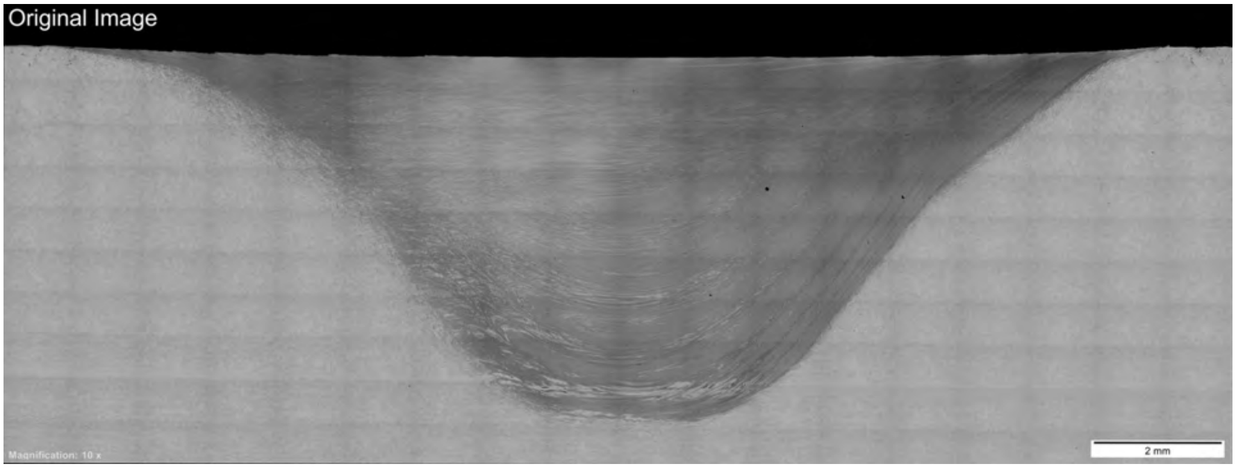

Figure 7.10. Optical Imagery at 100x Magnification: Condition C01, Sample SS31

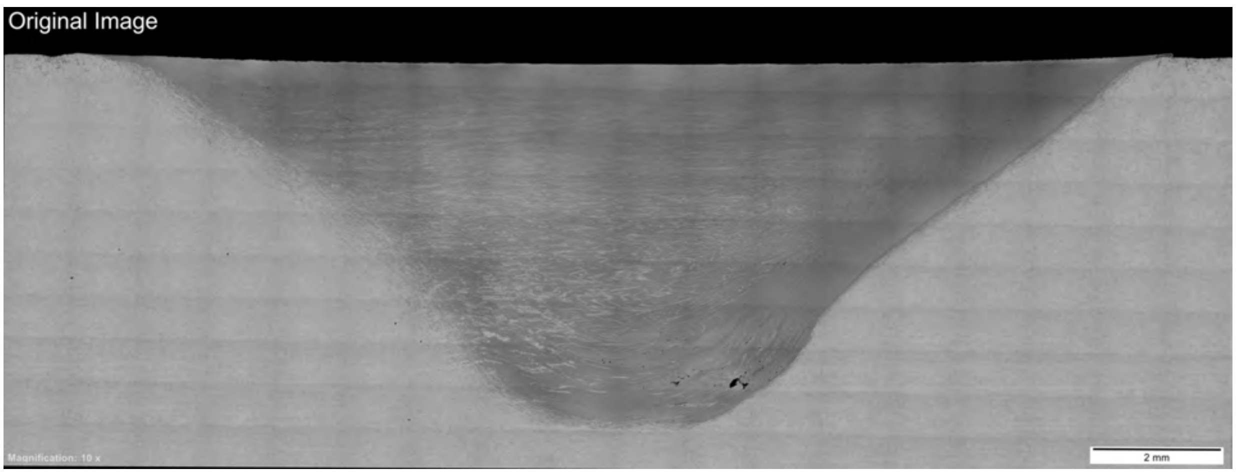

Figure 7.11. Optical Imagery at 100x Magnification: Condition C04, Sample SS32

7.2 EBSD Collection Locations

As described in the main report and in Attachment A, EBSD results were obtained at multiple locations (sites) for each specimen. The distinct locations were labeled L01 through L11. The locations were provided with EBSD documentation for each dataset relative to the stir zone center. An algorithm was used in combination with optical imagery spatial alignment data to produce the imagery provided in this section where locations are overlaid on the optical imagery shown in the last section.

Labels are listed with values that reflect (horizontal, vertical) +/- estimated uncertainty (uncertainty is only provided for L01-L05) of each location in millimeters relative to the stir zone center, with more negative values associated with the top left corner of the image. Note that the label font size is relatively small for the images embedded in this report. Refer to the full size files as needed.

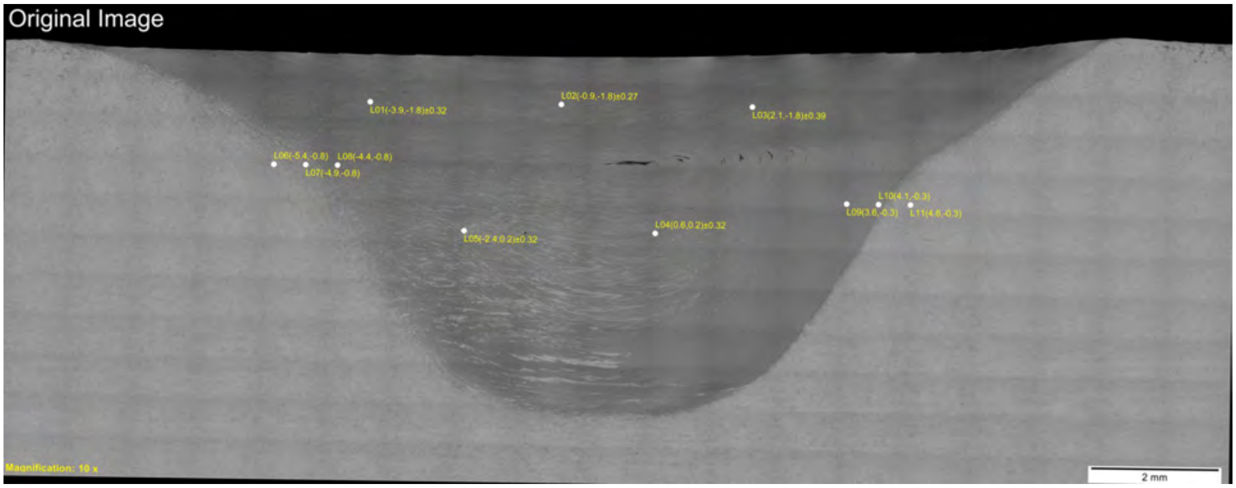

Figure 7.12. Collection Locations: Condition C03, Sample SS01

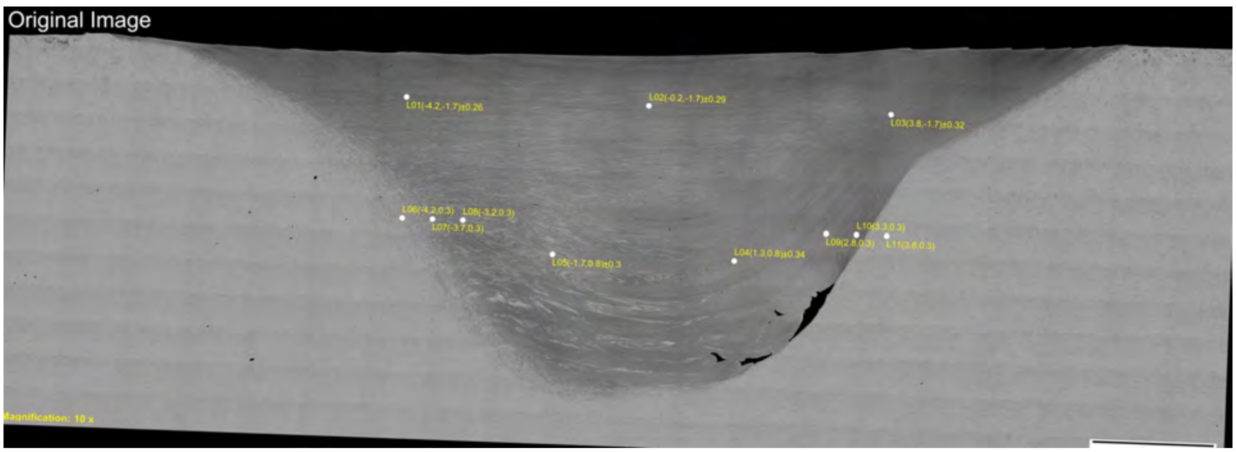

Figure 7.13. Collection Locations: Condition C05, Sample SS02

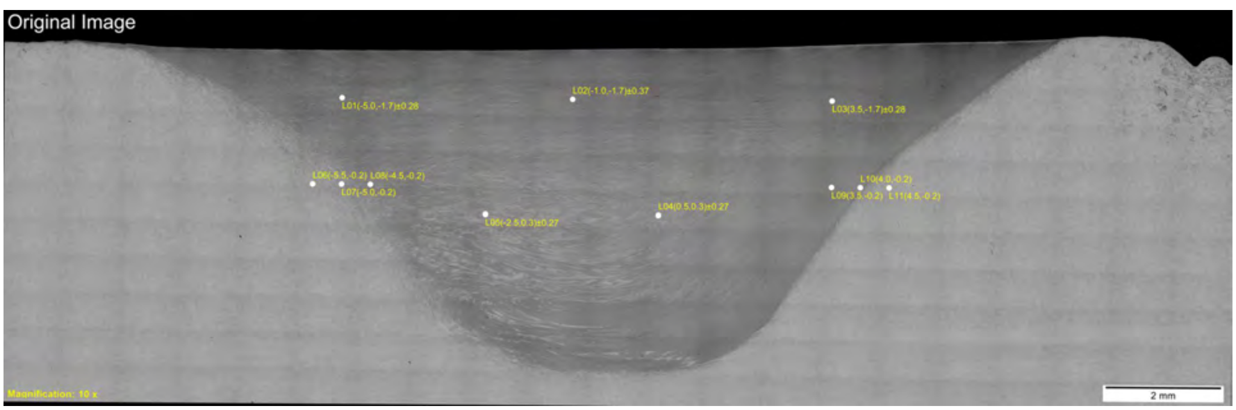

Figure 7.14. Collection Locations: Condition C06, Sample SS03

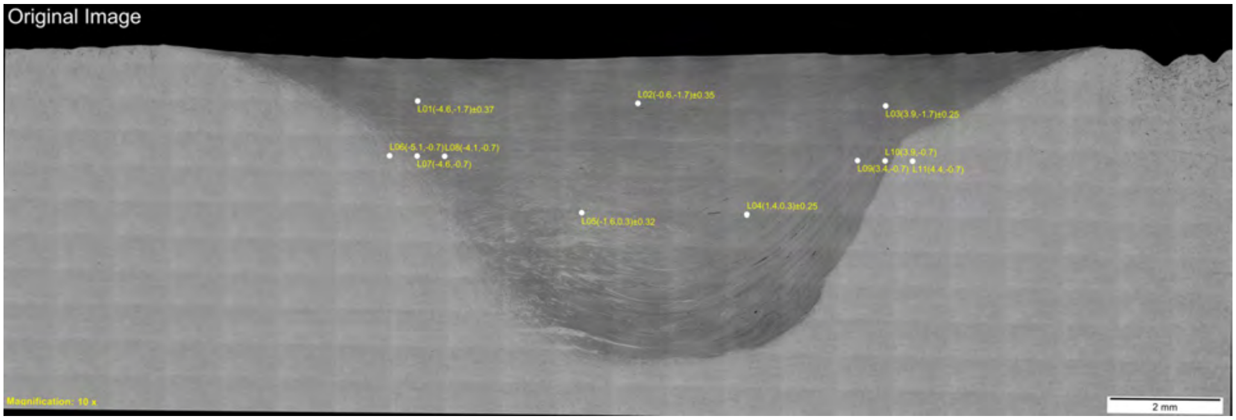

Figure 7.15. Collection Locations: Condition C07, Sample SS04

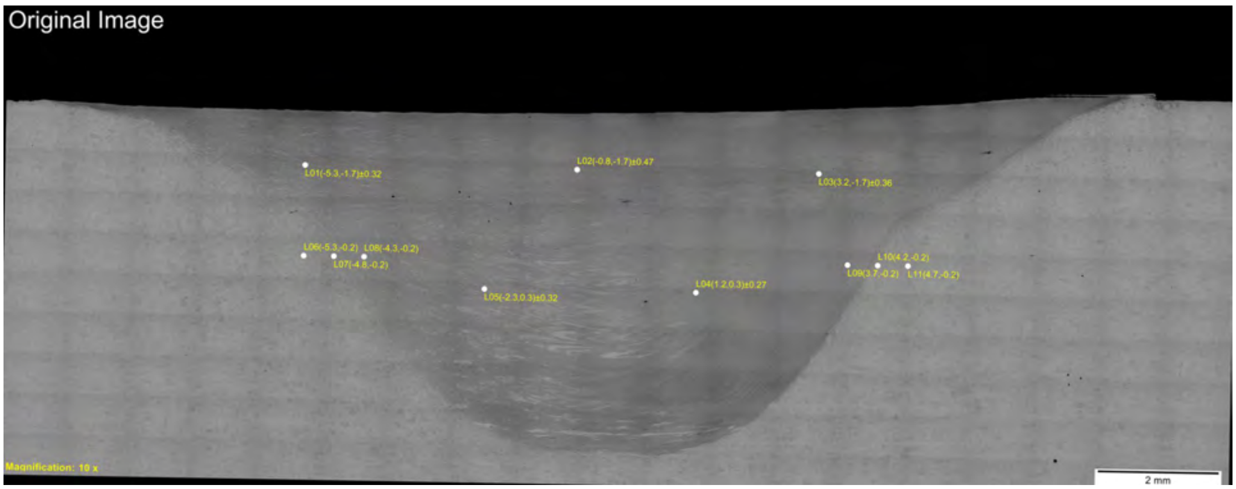

Figure 7.16. Collection Locations: Condition C08, Sample SS05

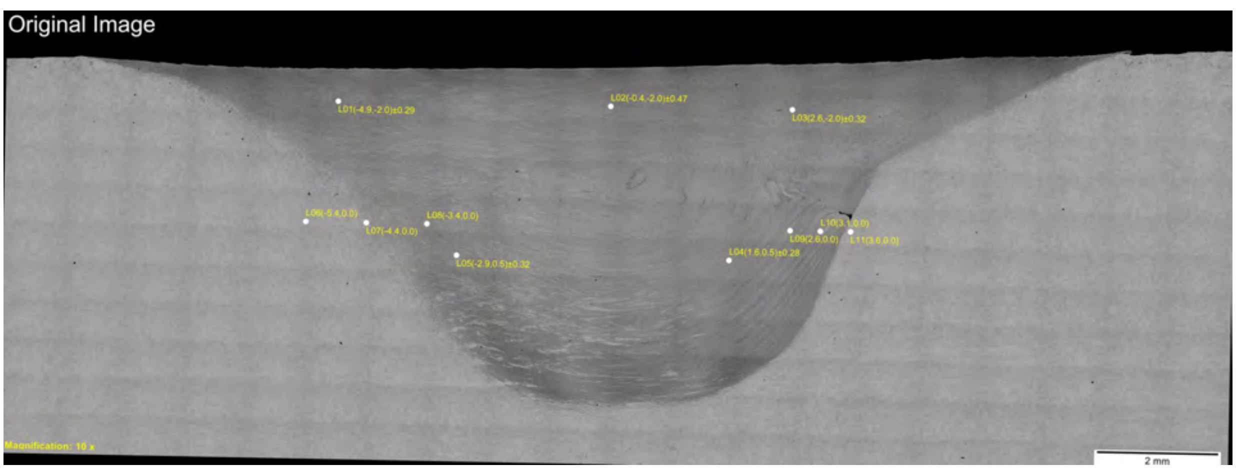

Figure 7.17. Collection Locations: Condition C09, Sample SS06

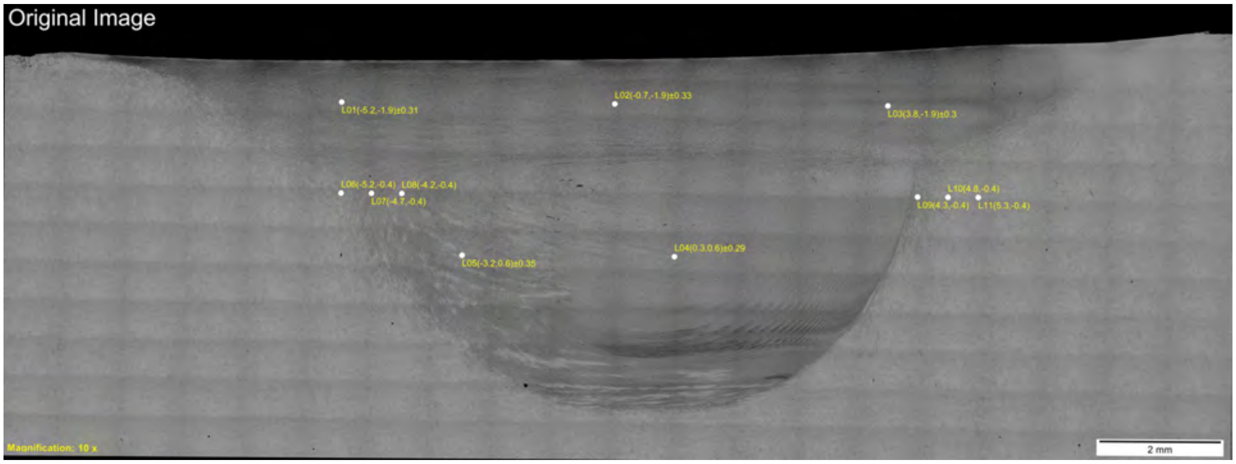

Figure 7.18. Collection Locations: Condition C10, Sample SS07

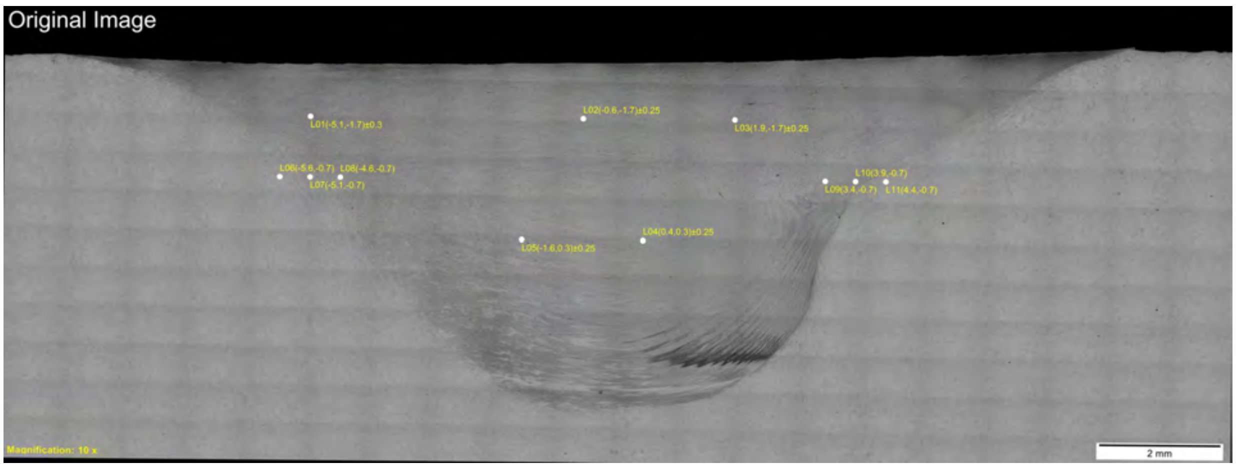

Figure 7.19. Collection Locations: Condition C11, Sample SS08

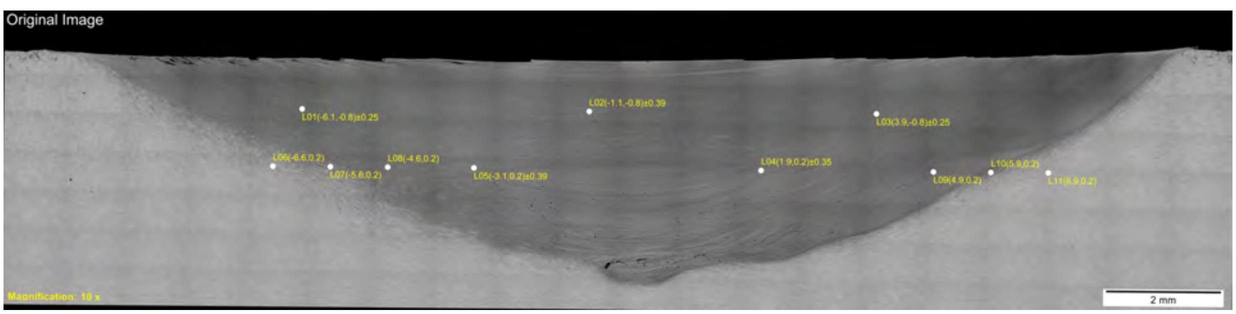

Figure 7.20. Collection Locations: Condition C02, Sample SS28

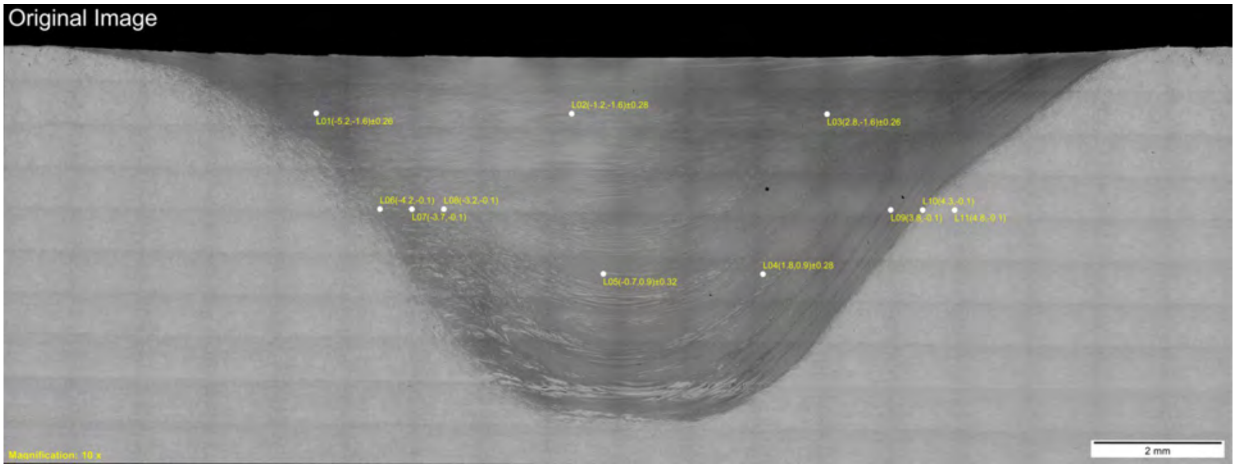

Figure 7.21. Collection Locations: Condition C01, Sample SS31

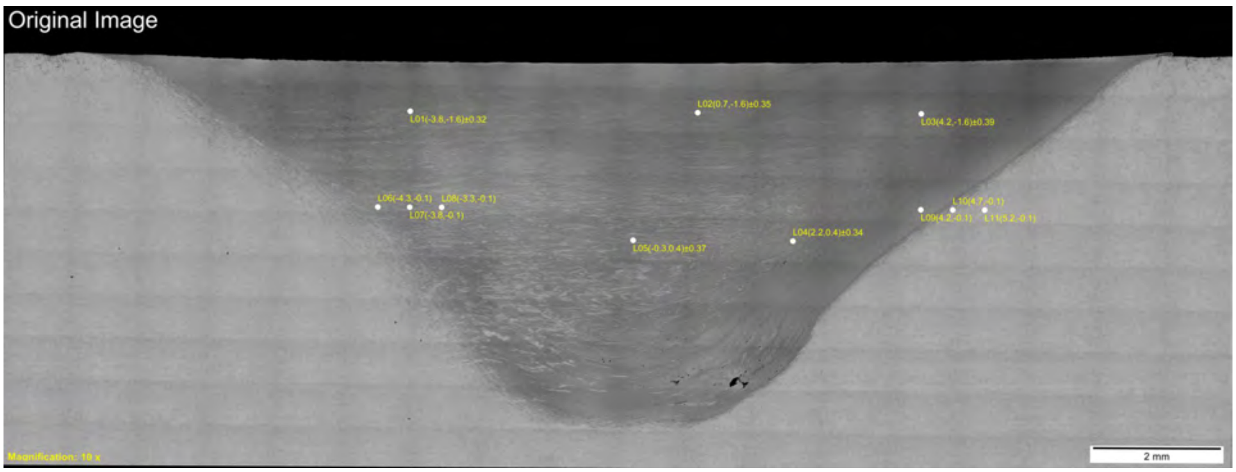

Figure 7.22. Collection Locations: Condition C04, Sample SS32

7.3 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.

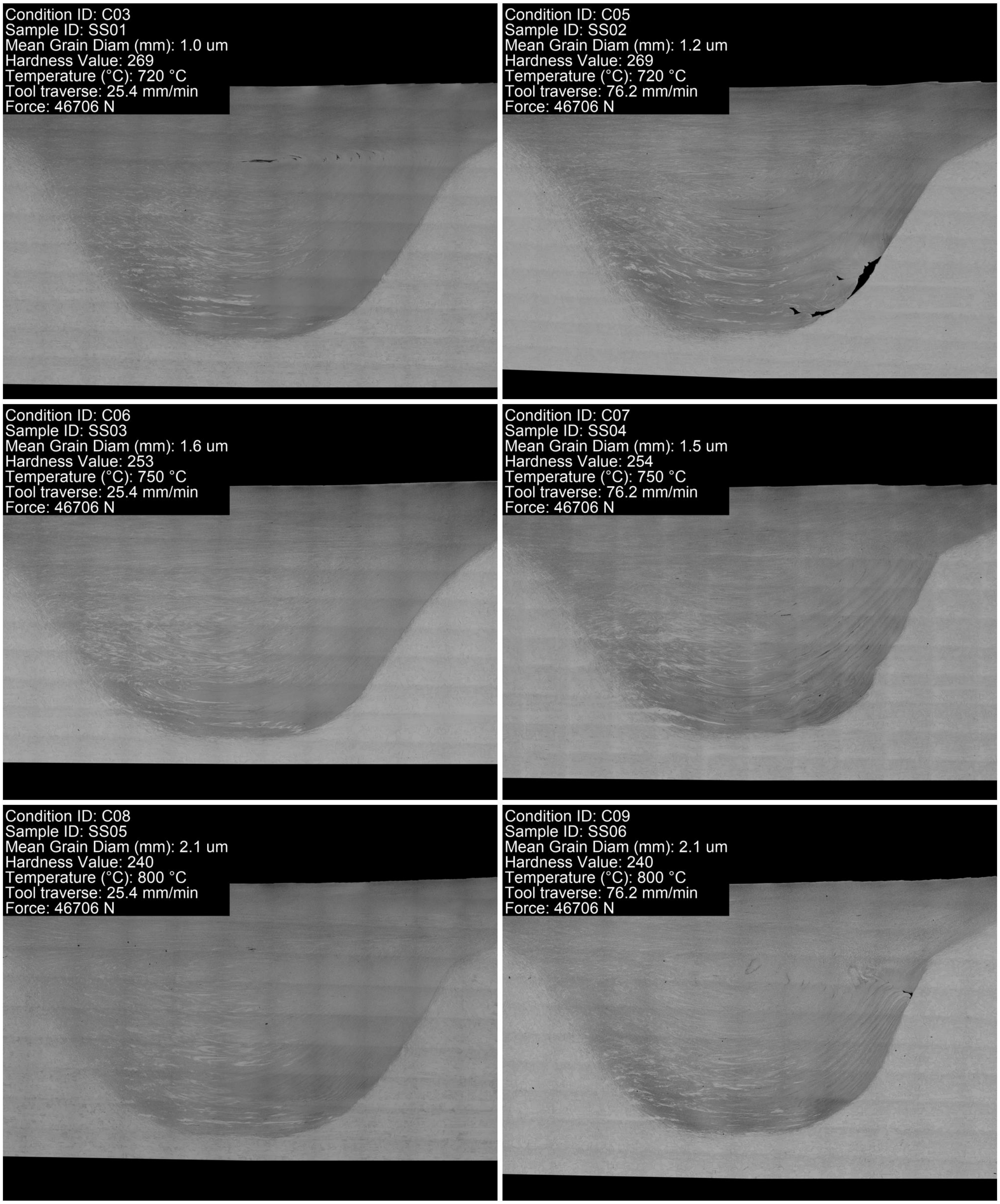

Figure 7.23. Stir Region Collage: Image number 1

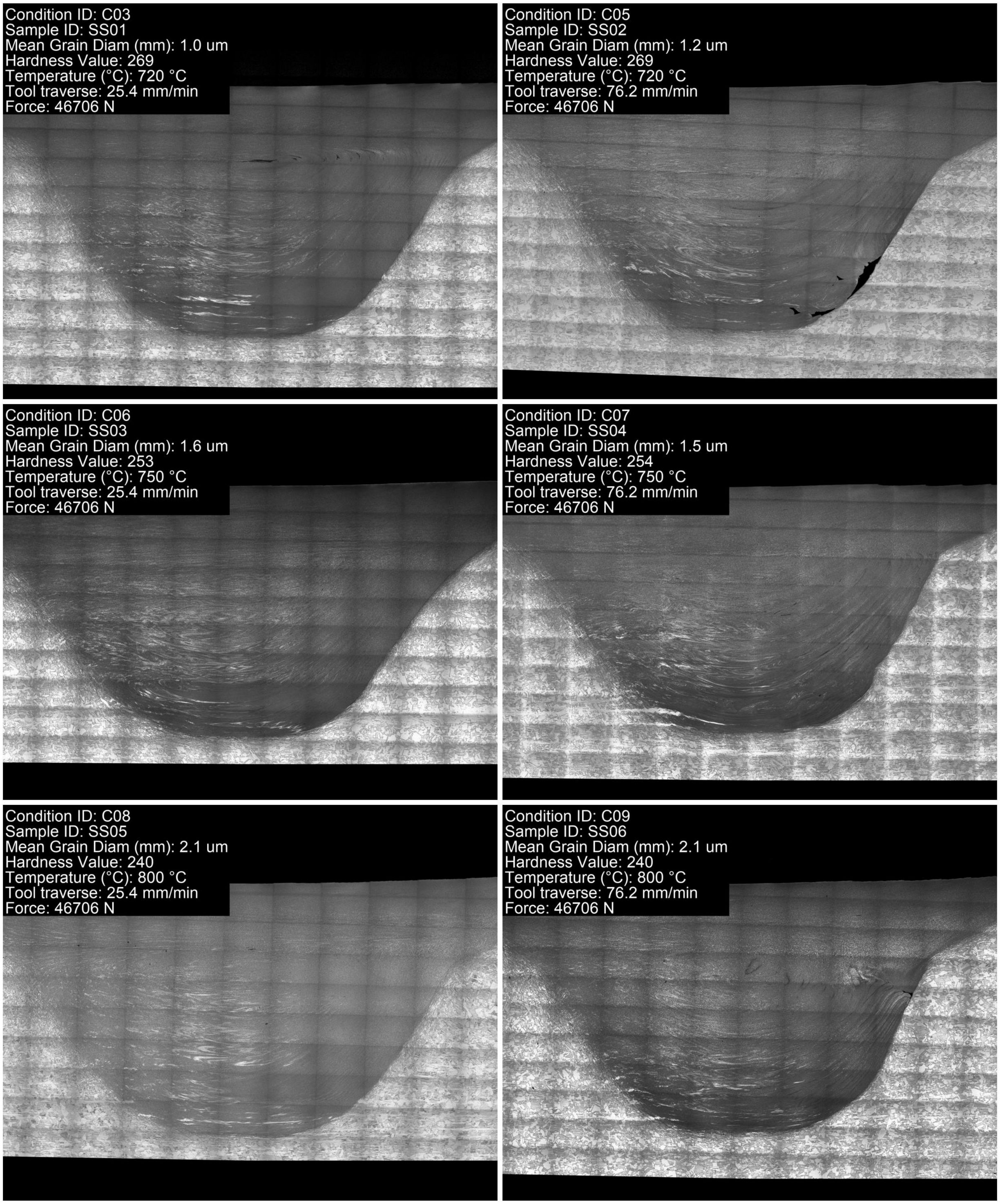

Figure 7.24. Stir Region Collage: Image number 2

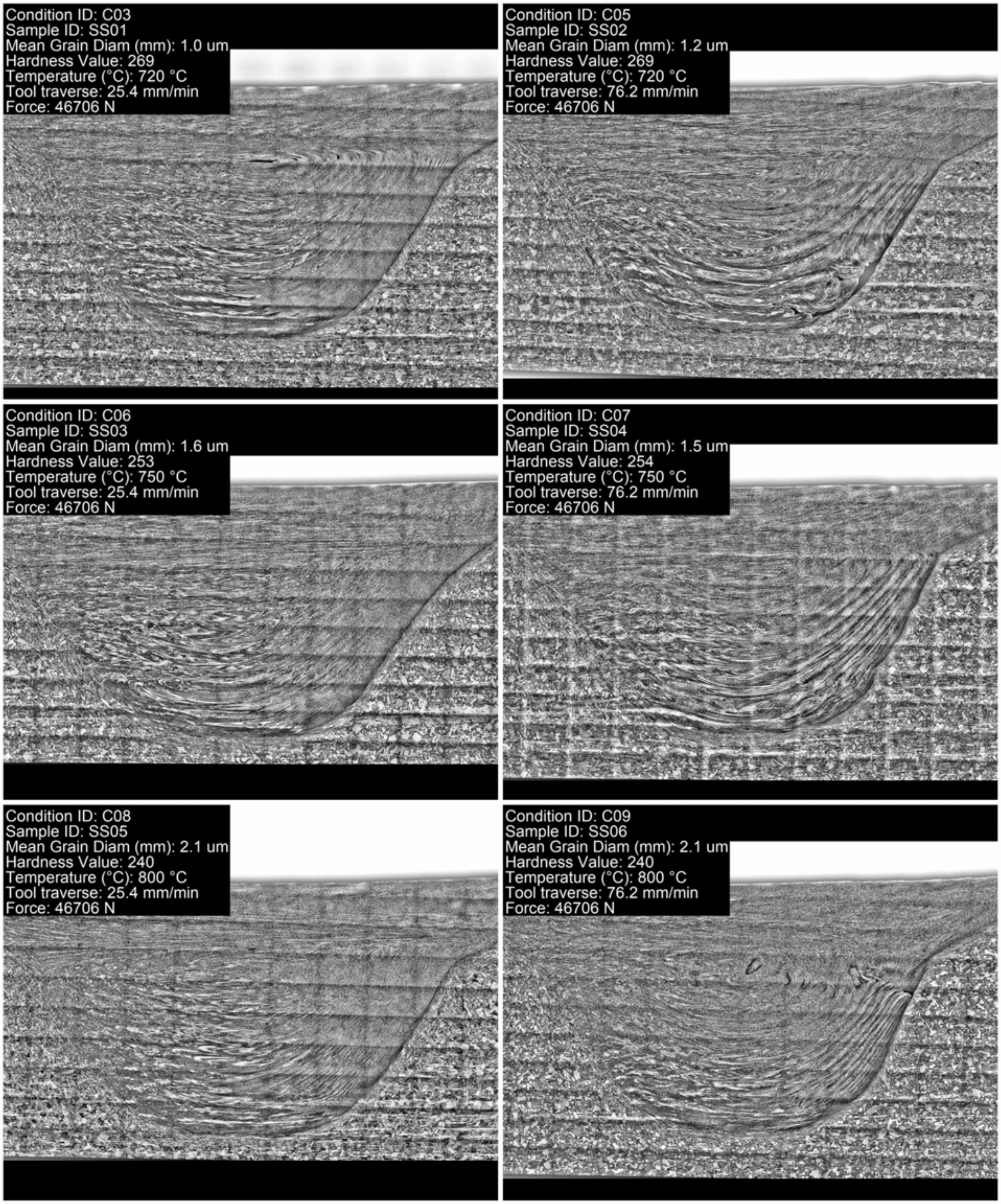

Figure 7.25. Stir Region Collage: Image number 3

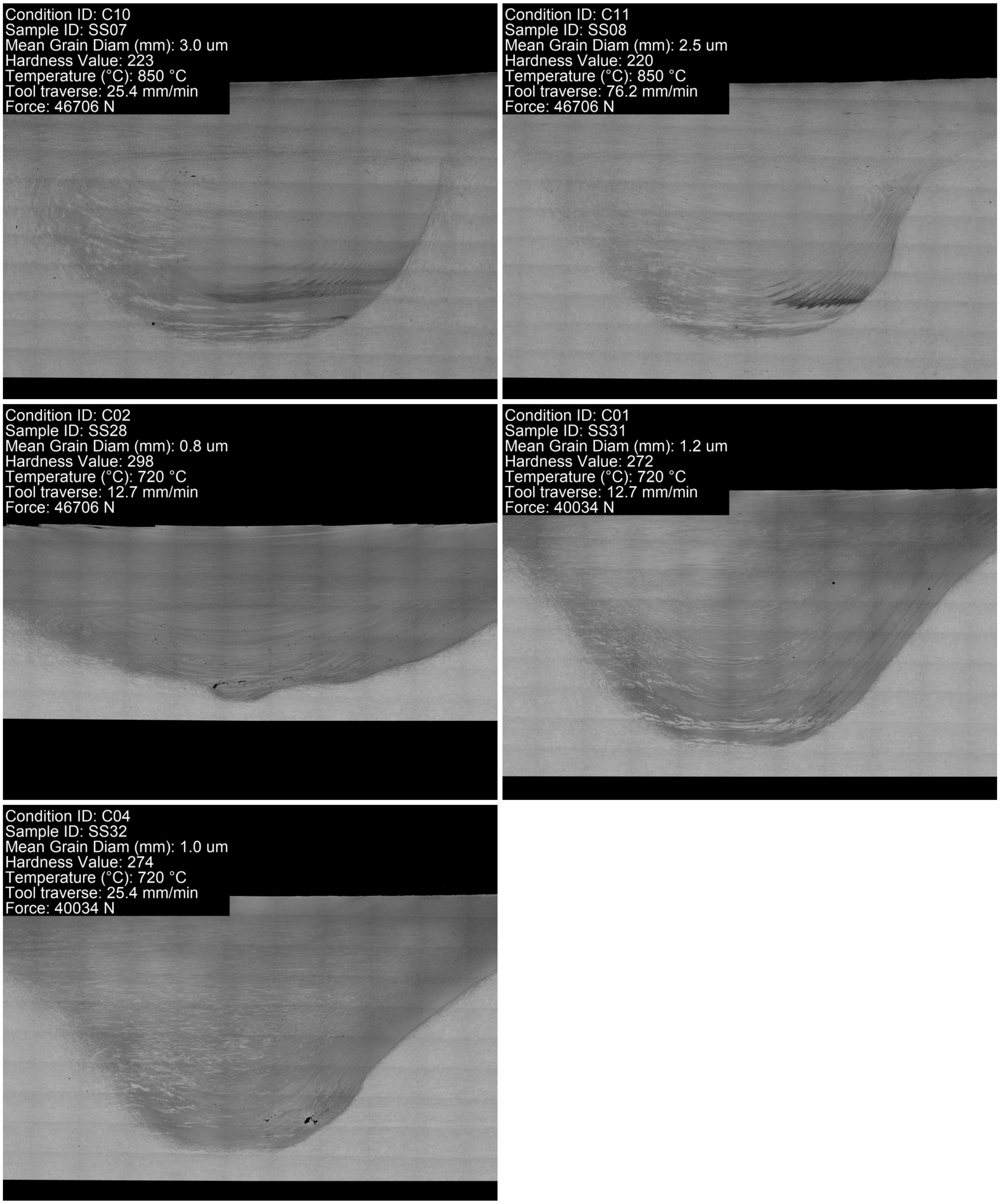

Figure 7.26. Stir Region Collage: Image number 4

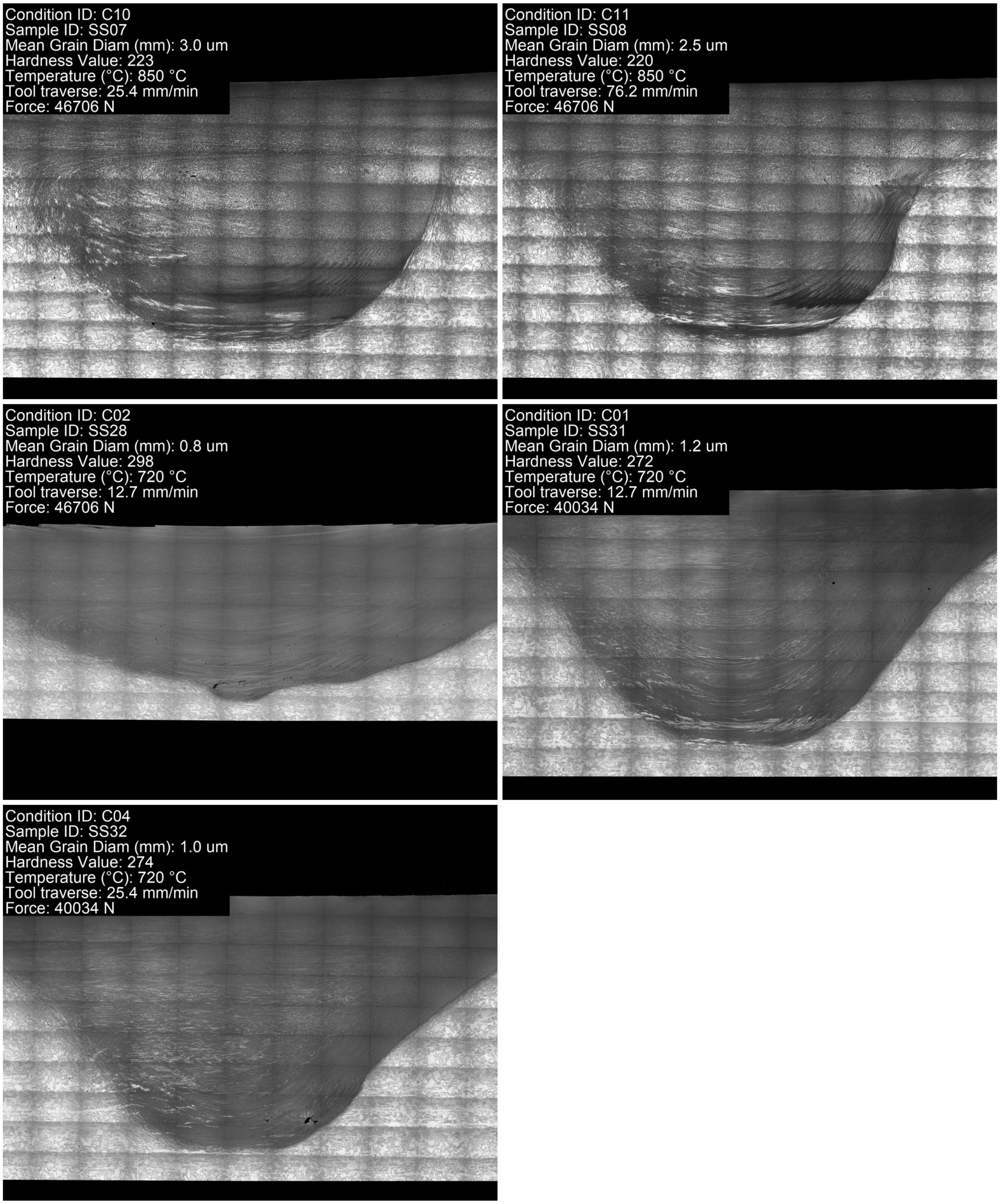

Figure 7.27. Stir Region Collage: Image number 5

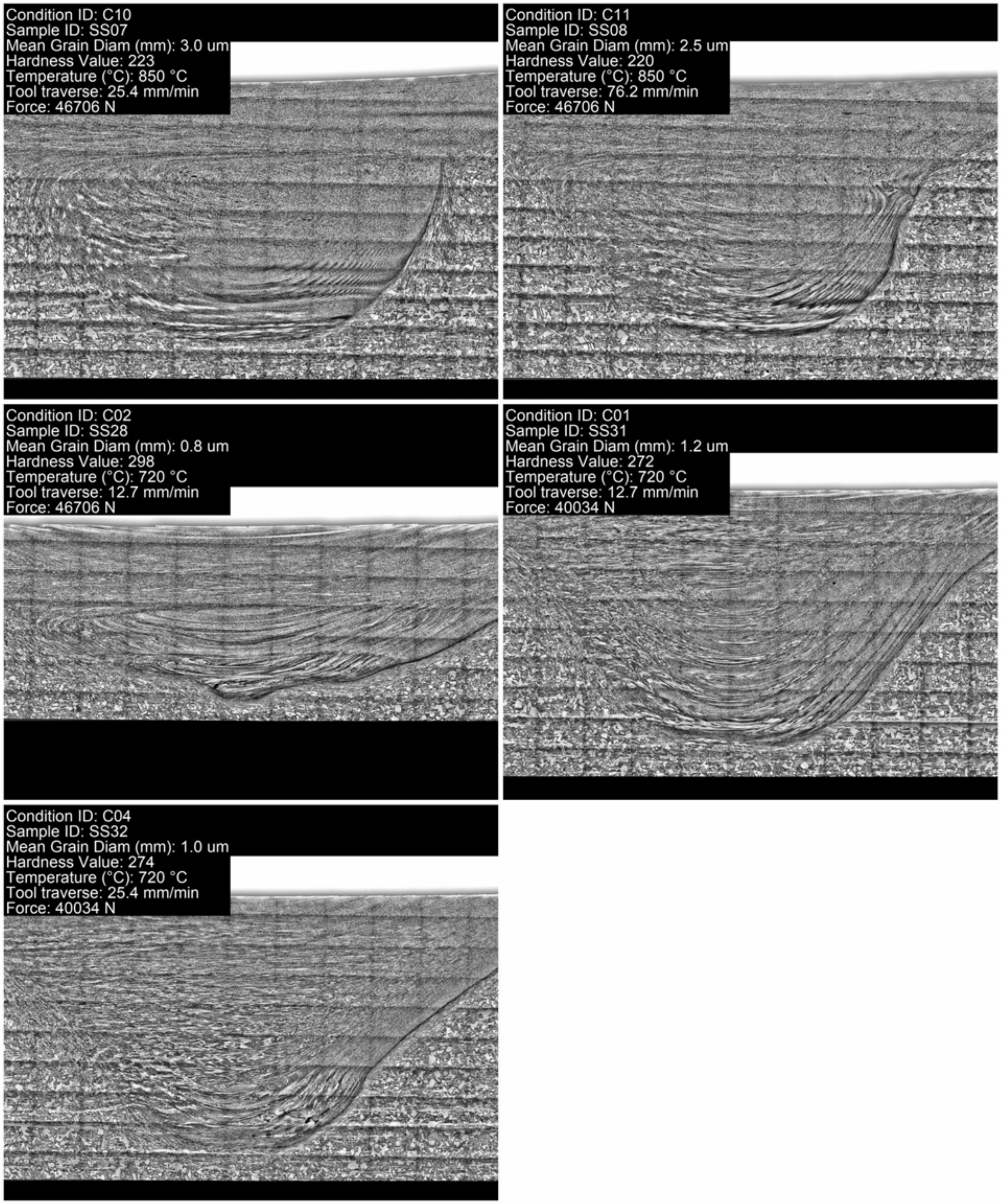

Figure 7.28. Stir Region Collage: Image number 6

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Attachment D: MCPC Round 1 Material Characterization Results for Modality SEM

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DR Todd A Guzman

Attachment D contains 27 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 instrument used to collect data is identified as follows:

- Company Name: JEOL
- MOD: Jsm-lt800
- Serial #: SM1040000060006

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. For SEM, the key data file is a montage image provided in PNG file format that highlights all information obtained for the dataset. Additionally, the individual tiles obtained by the instrument and used to construct the montage are provided for use in machine learning analysis.

- **(name).png** (PNG Image type): The montage SEM image. Tiles are stitched and rotated by proprietary software that may choose to exclude some tiles, which appear as black regions.

- **(name)_Tile-nnnn.tif** (TIFF Image type, nnnn is a unique four digit tile number): The individual tiles obtained by the instrument and used to construct the montage. These tiles are added to the montage by moving from the top right corner of the montage to the lower left (except as noted in Comments, below). Enumeration of the tiles may not start at 1.

4.2 VISUALIZATION Directory Contents

The following visualizations are provided:

- **(name)_Tile-map.jpg** (JPG Image type): A reconstructed map of the tile locations in the montage. Tile boundaries and tile numbers are indicated to assist in finding specific SEM tiles in the DATA directory. This image is intended as an aide and not for computational analysis, so it is reduced size and in JPG format.

5.0 Notes and Comments

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

1. Due to a timing issue with availability of the specimens, SEM for Sample SS02 was performed on Specimen MCPC0006 (the same as Optical and Hardness) while EBSD was performed on Specimen MCPC0007. These specimens are from the same sample.

2. SS28_22775 was declared invalid based on visual inspection. The data was recollected and named dataset SS28_22775a

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

4. Ordering of tiles for dataset SS06, SS07, and SS28 is not ordered as described above. The ordering is random based on a setting chosen during data collection that was used to overcome charging effects. A map for these datasets is not provided.

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 Full Stir Region

Measurements with SEM were performed on a relatively low magnification that revealed the gross view of the FSP-affected region. The magnification level resolved grain structure in unprocessed material but was not sufficient to resolve individual grains within the FSP-affected region. Results are provided below in a montaged image constructed of individual image tiles.

The displayed images have been slightly rotated to improve alignment and image size reduced to 2000 pixels in width for handling convenience. The raw files are provided with electronic data.

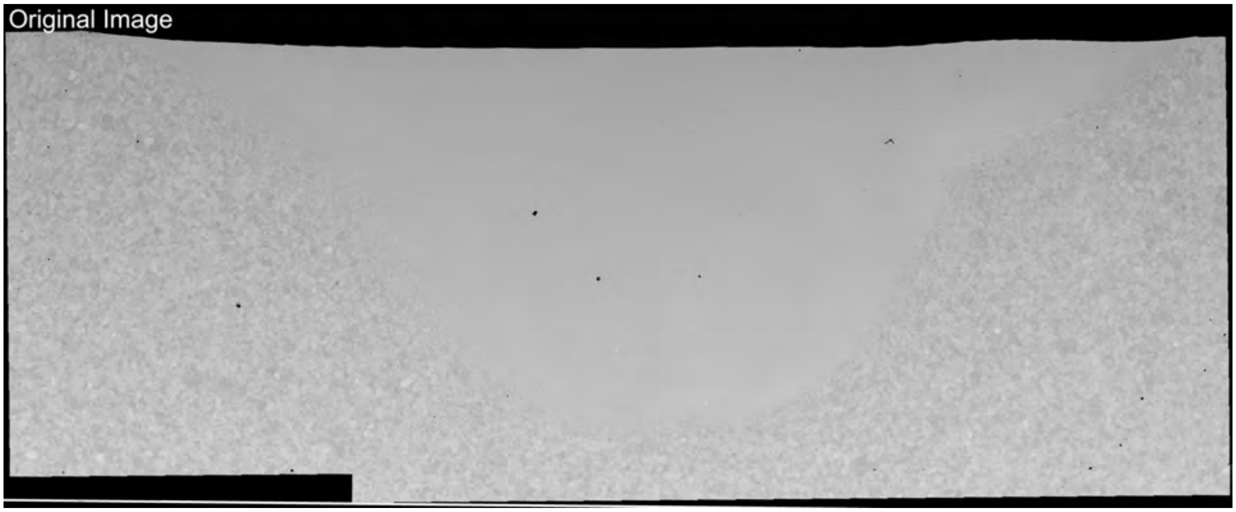

Figure 7.1. Full Stir Region SEM Montage: Condition C03, Sample SS01

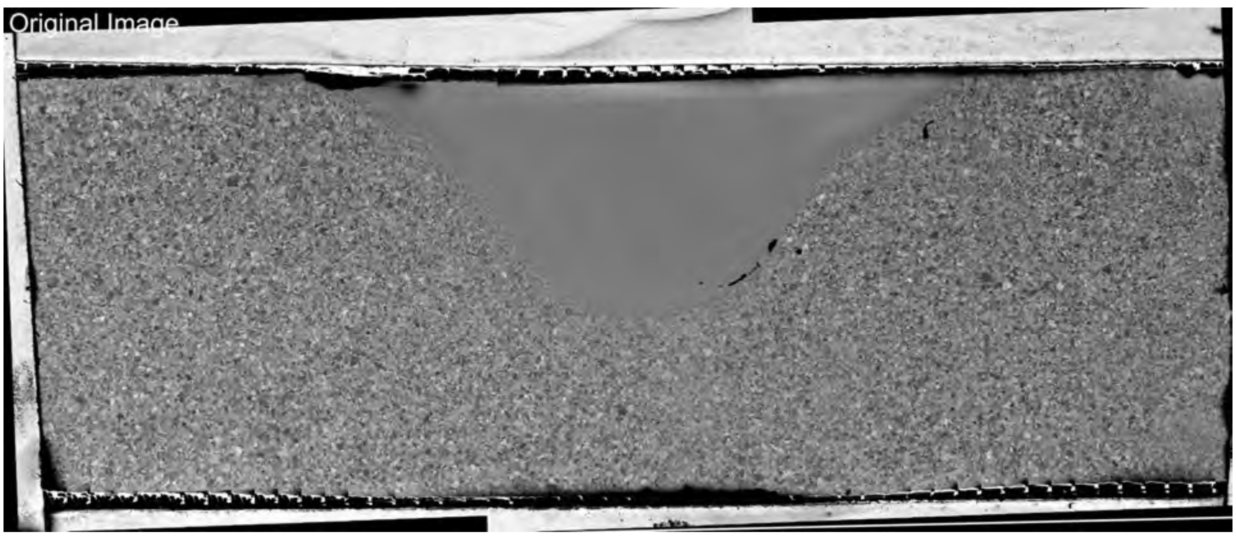

Figure 7.2. Full Stir Region SEM Montage: Condition C05, Sample SS02

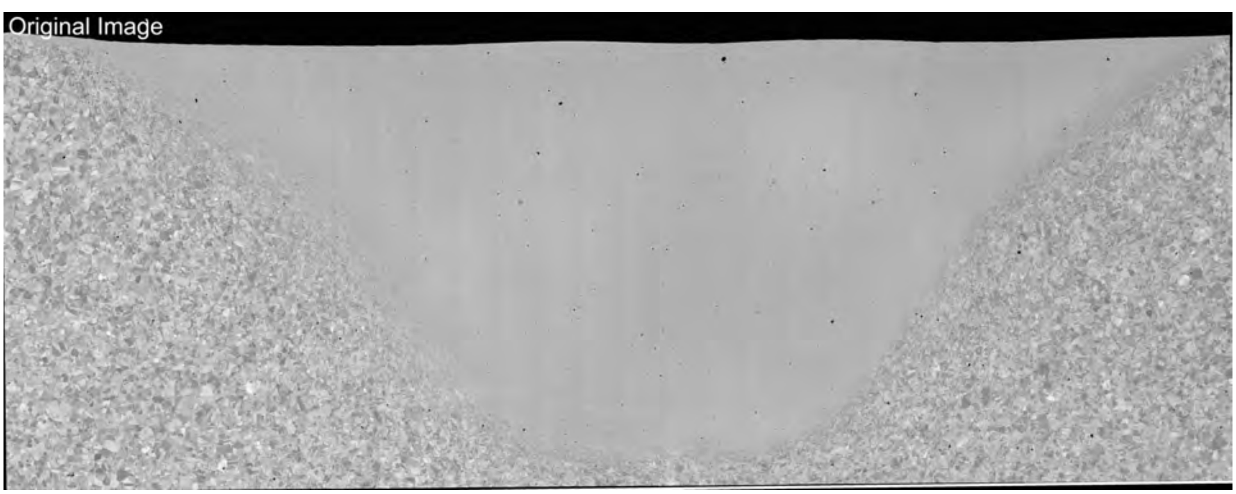

Figure 7.3. Full Stir Region SEM Montage: Condition C06, Sample SS03

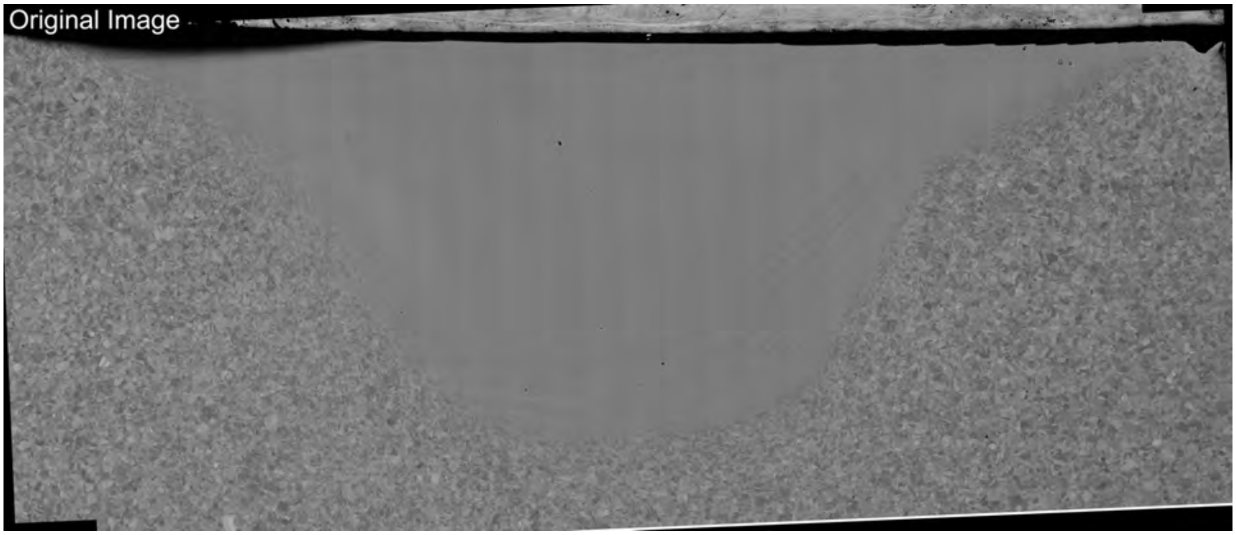

Figure 7.4. Full Stir Region SEM Montage: Condition C07, Sample SS04

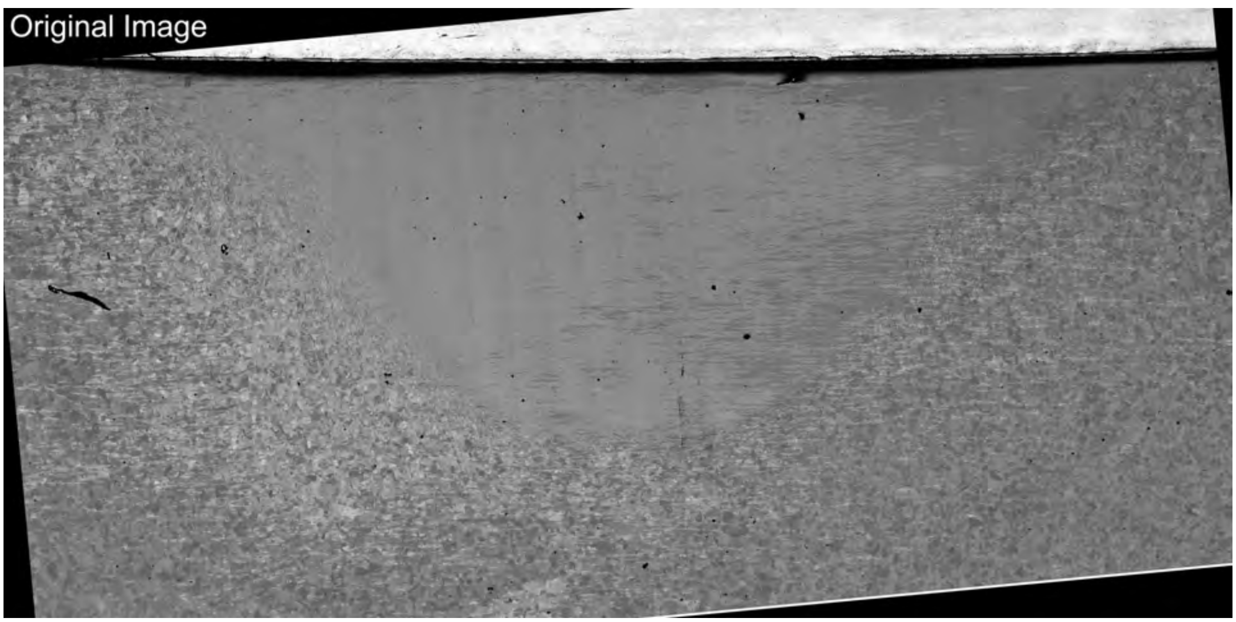

Figure 7.5. Full Stir Region SEM Montage: Condition C08, Sample SS05

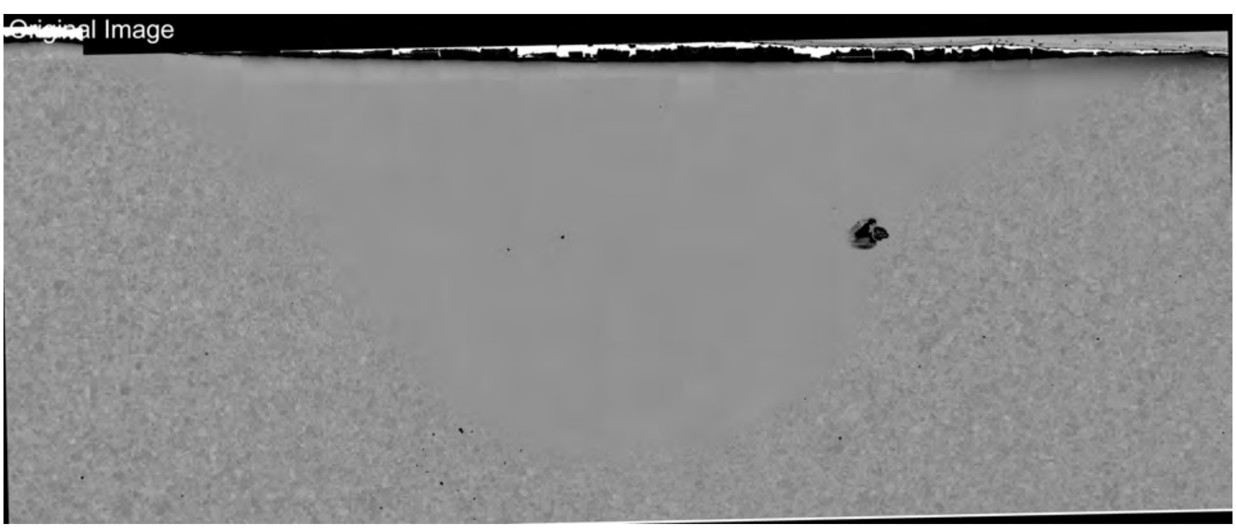

Figure 7.6. Full Stir Region SEM Montage: Condition C09, Sample SS06

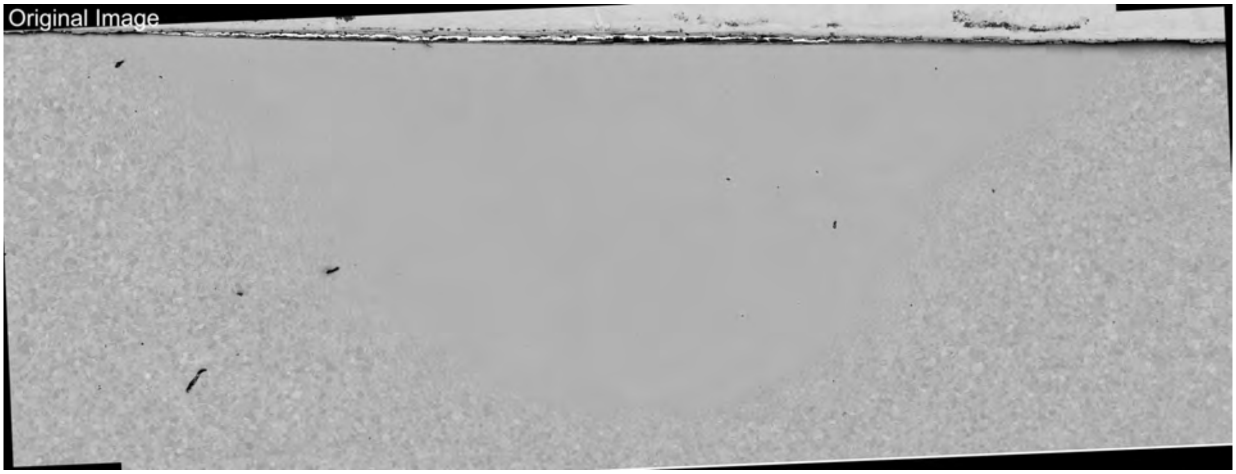

Figure 7.7. Full Stir Region SEM Montage: Condition C10, Sample SS07

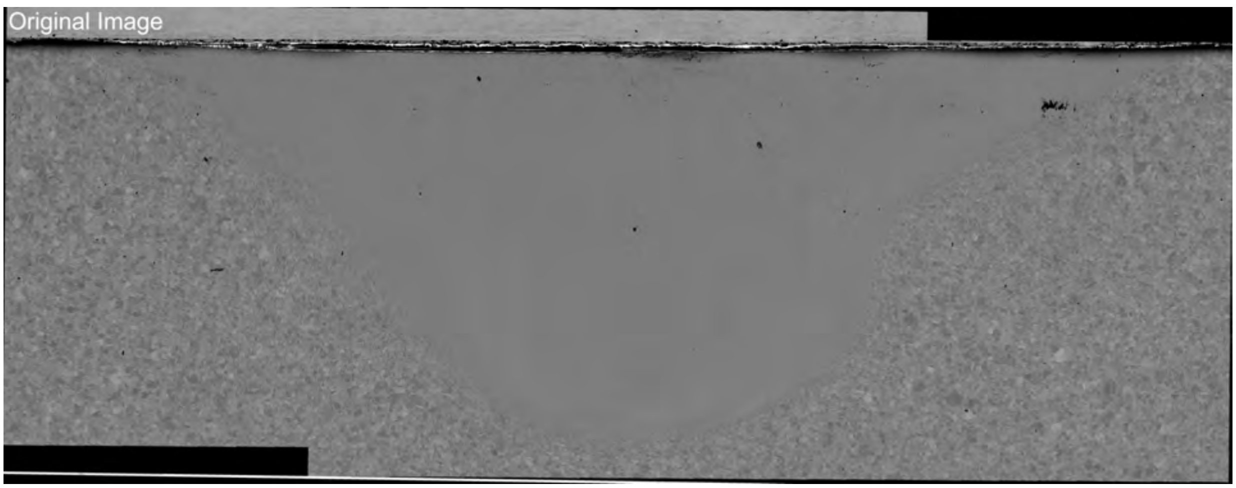

Figure 7.8. Full Stir Region SEM Montage: Condition C11, Sample SS08

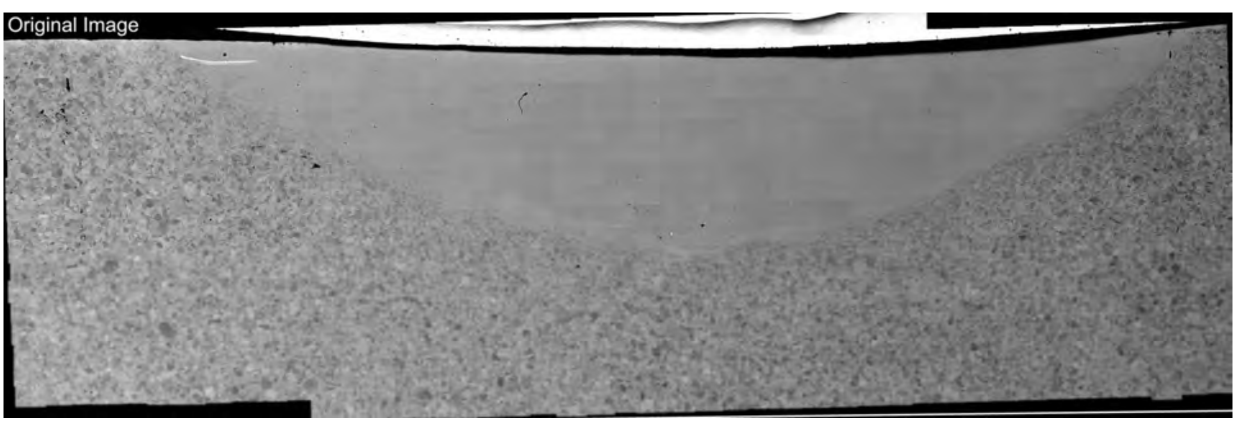

Figure 7.9. Full Stir Region SEM Montage: Condition C02, Sample SS28

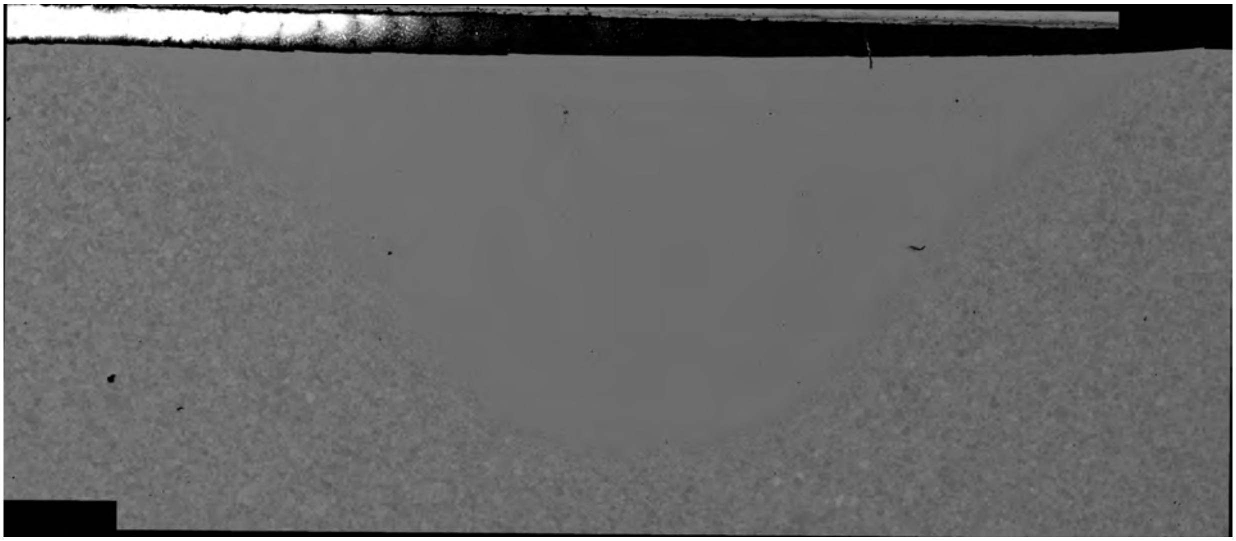

Figure 7.10. Full Stir Region SEM Montage: Condition C01, Sample SS31

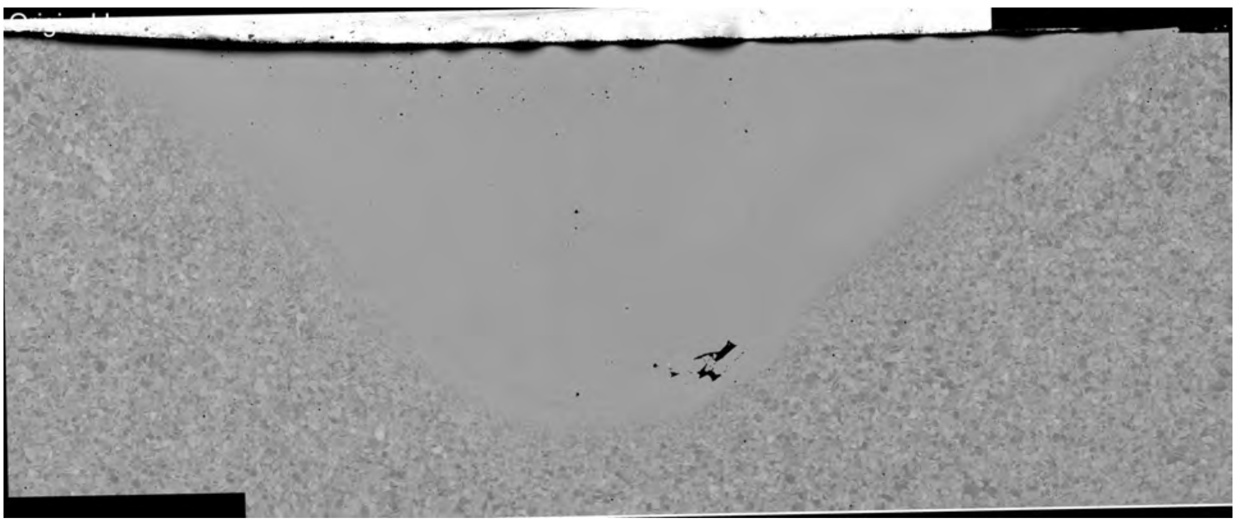

Figure 7.11. Full Stir Region SEM Montage: Condition C04, Sample SS32

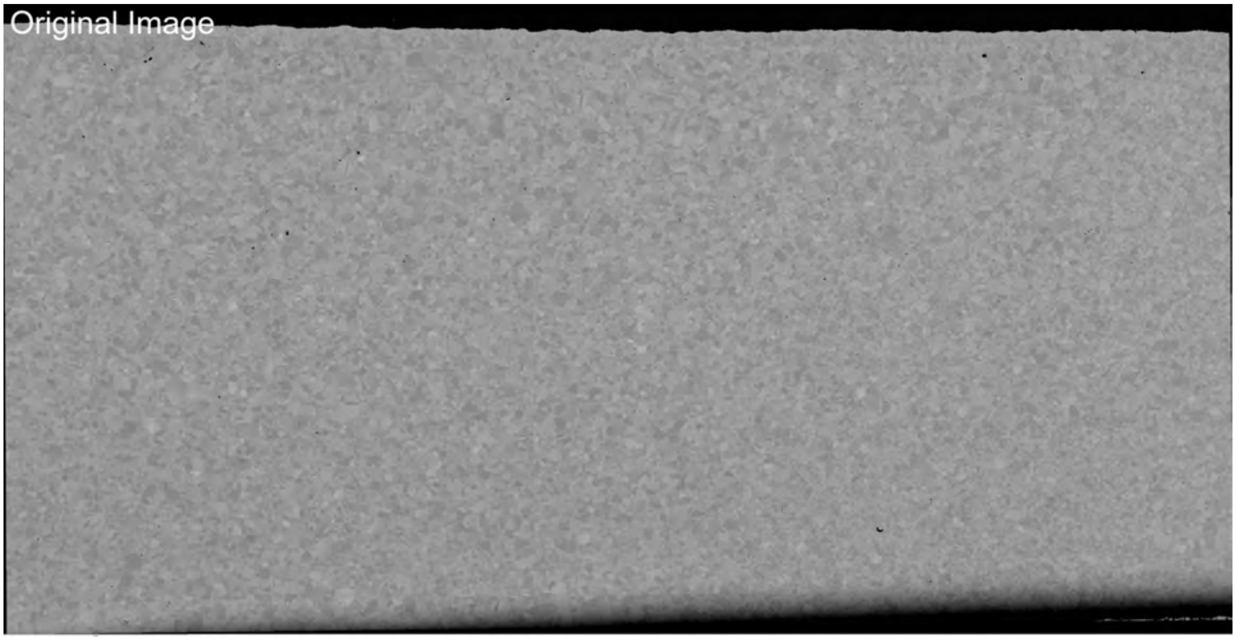

Figure 7.12. Full Stir Region SEM Montage: Condition C00, Sample SS36

7.2 Full Stir Region with Tile Maps

The image montages in the last section were constructed from individual SEM collections. In general, ordering of the SEM tiles proceeded from the upper right hand corner of the montage. But not necessarily from zero. Results are provided below that show the reconstructed tile locations in each montage with blue numbers corresponding to the tile numbers. The numerical labels in these images are small, refer to the archived numbers associated with nnnn in archived files. Ordering of tiles for dataset SS06, SS07, and SS28 was done pseudo-randomly as described earlier and tile maps are not provided. Note that these images were not slightly rotated to improve alignment as was done in the last section.

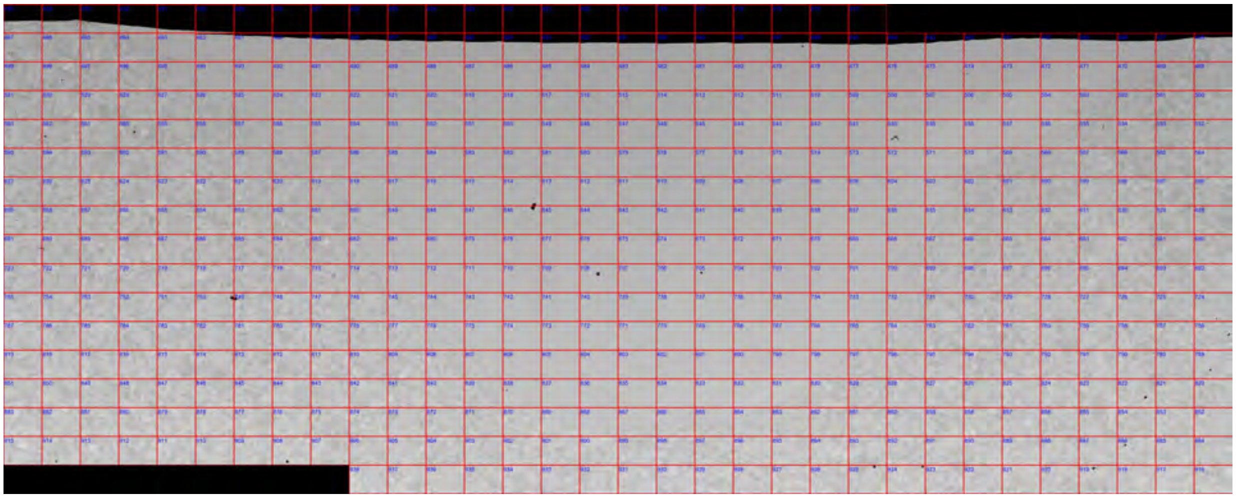

Figure 7.13. Full Stir Region SEM Montage with Tile Map: Condition C03, Sample SS01

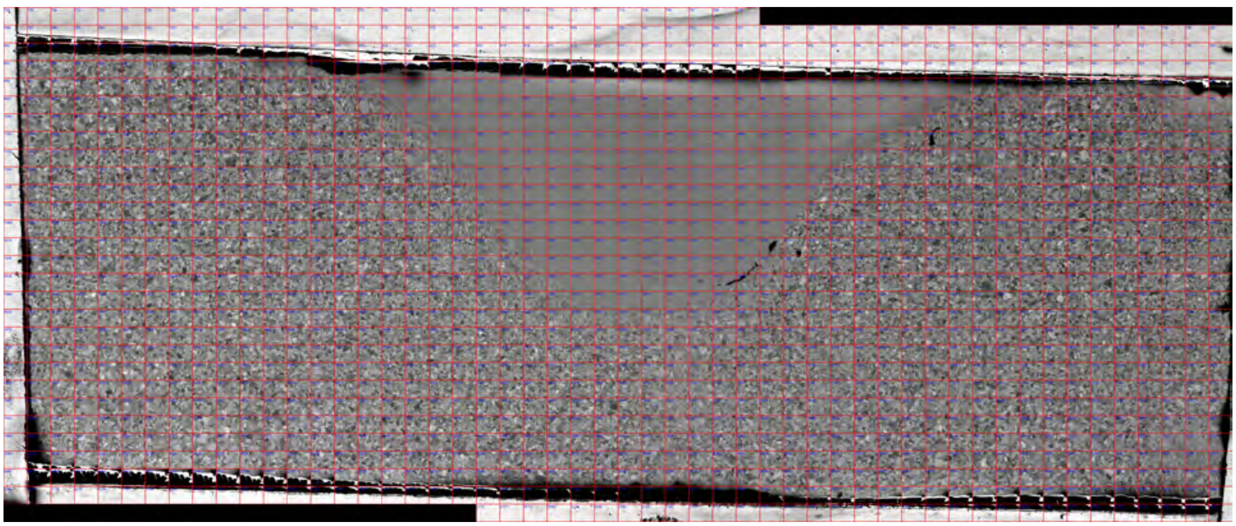

Figure 7.14. Full Stir Region SEM Montage with Tile Map: Condition C05, Sample SS02

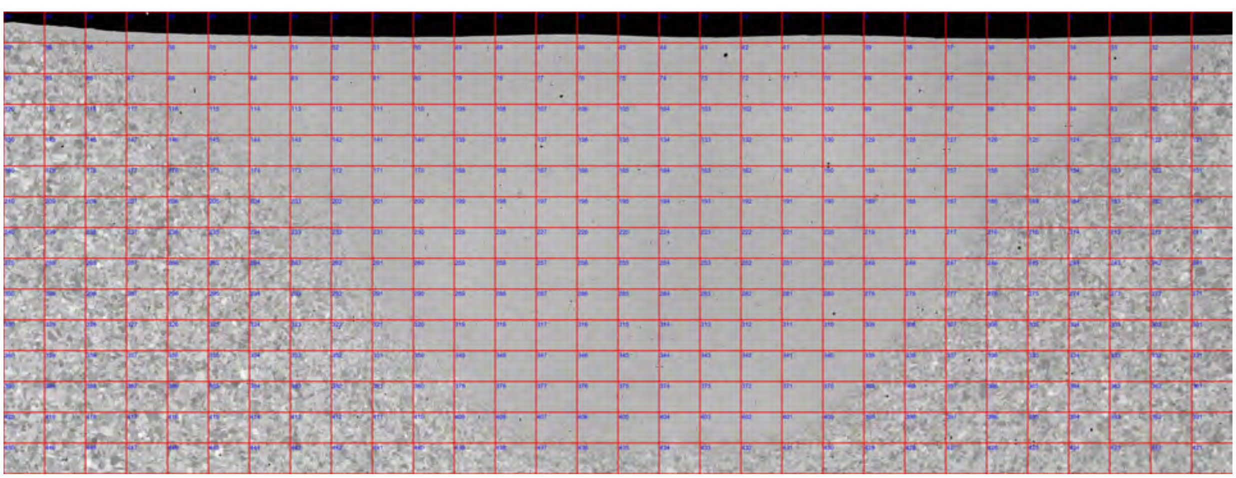

Figure 7.15. Full Stir Region SEM Montage with Tile Map: Condition C06, Sample SS03

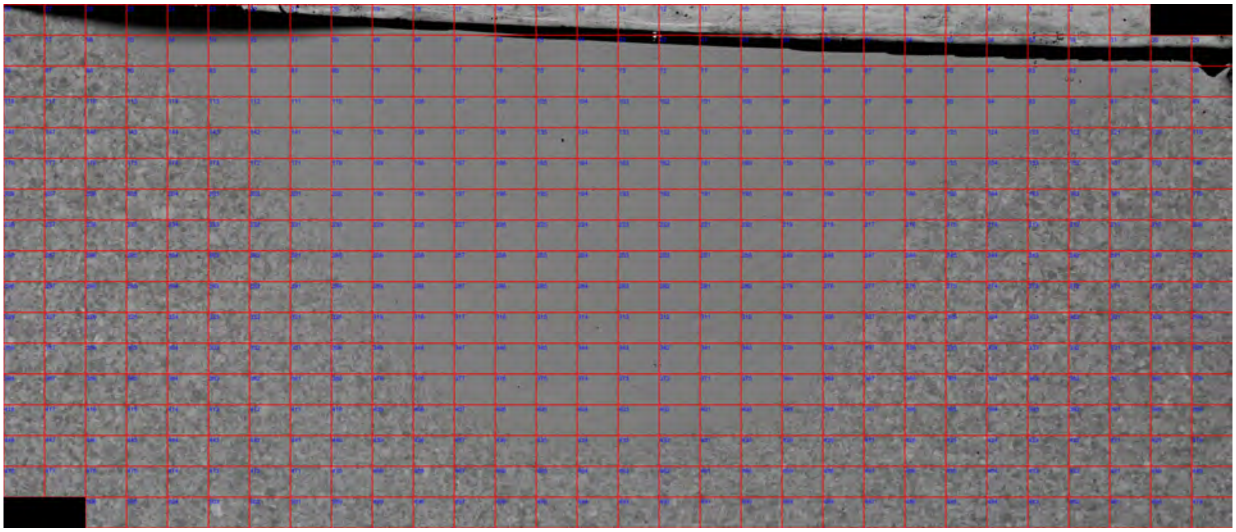

Figure 7.16. Full Stir Region SEM Montage with Tile Map: Condition C07, Sample SS04

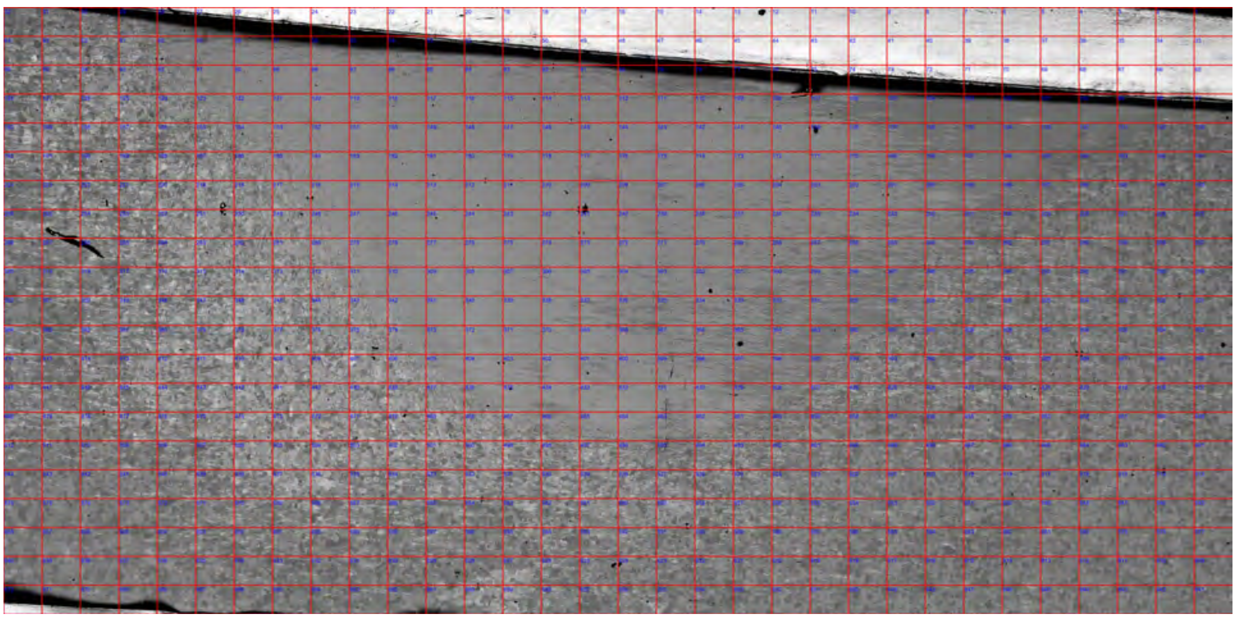

Figure 7.17. Full Stir Region SEM Montage with Tile Map: Condition C08, Sample SS05

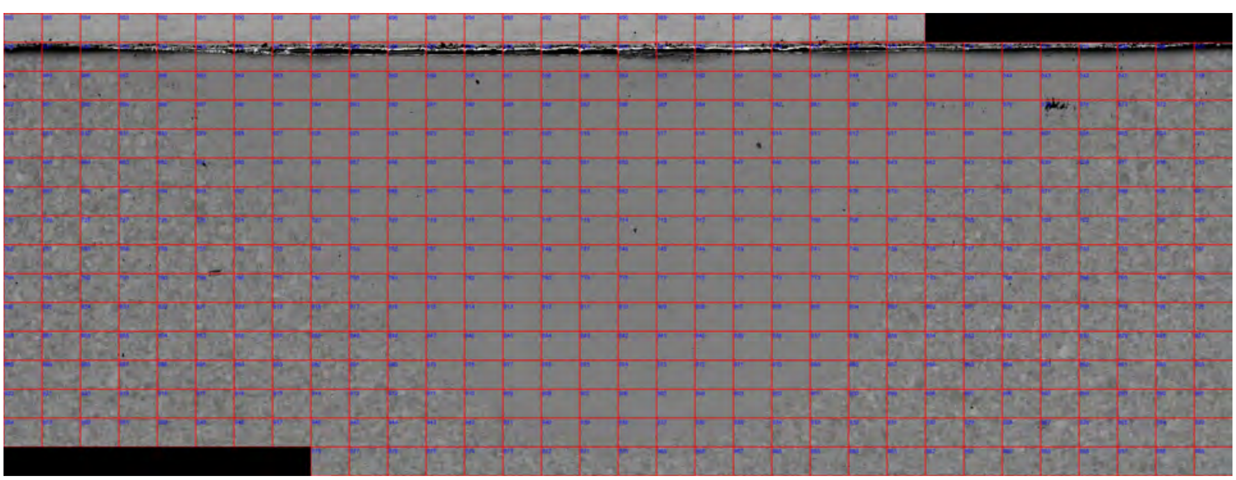

Figure 7.18. Full Stir Region SEM Montage with Tile Map: Condition C11, Sample SS08

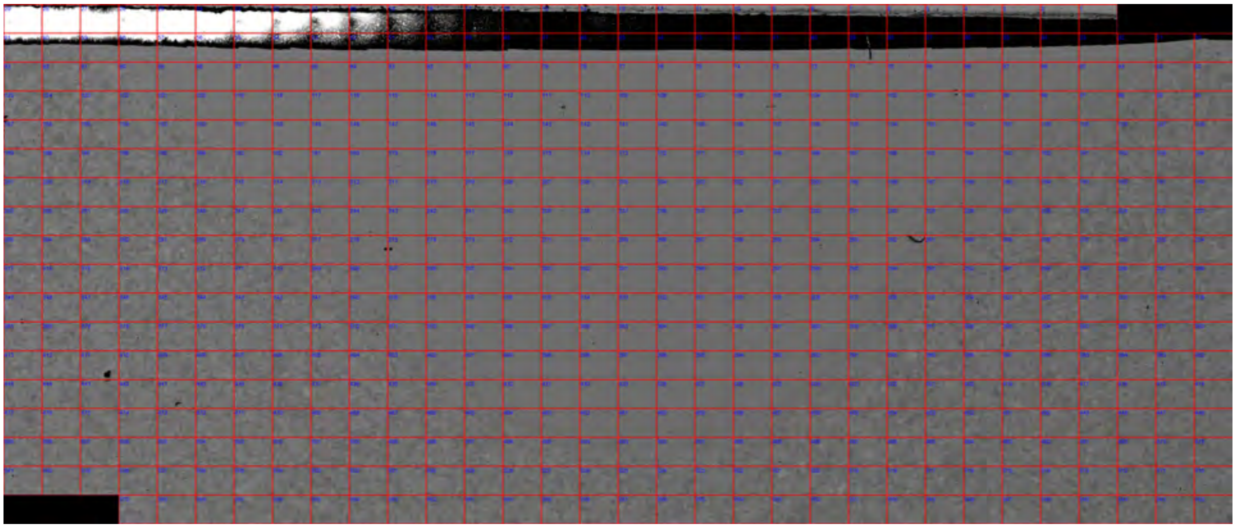

Figure 7.19. Full Stir Region SEM Montage with Tile Map: Condition C01, Sample SS31

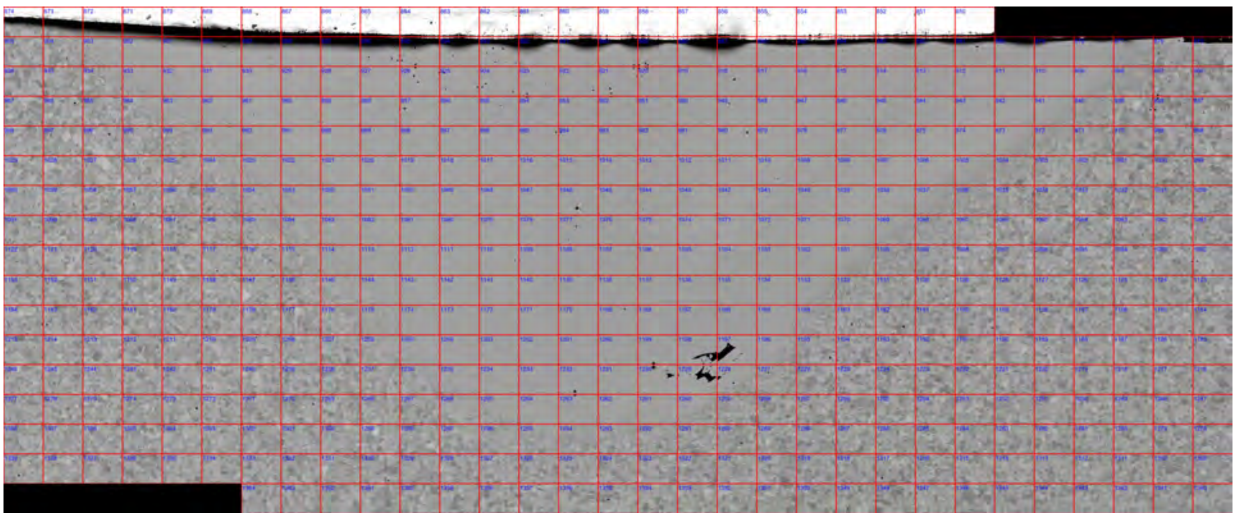

Figure 7.20. Full Stir Region SEM Montage with Tile Map: Condition C04, Sample SS32

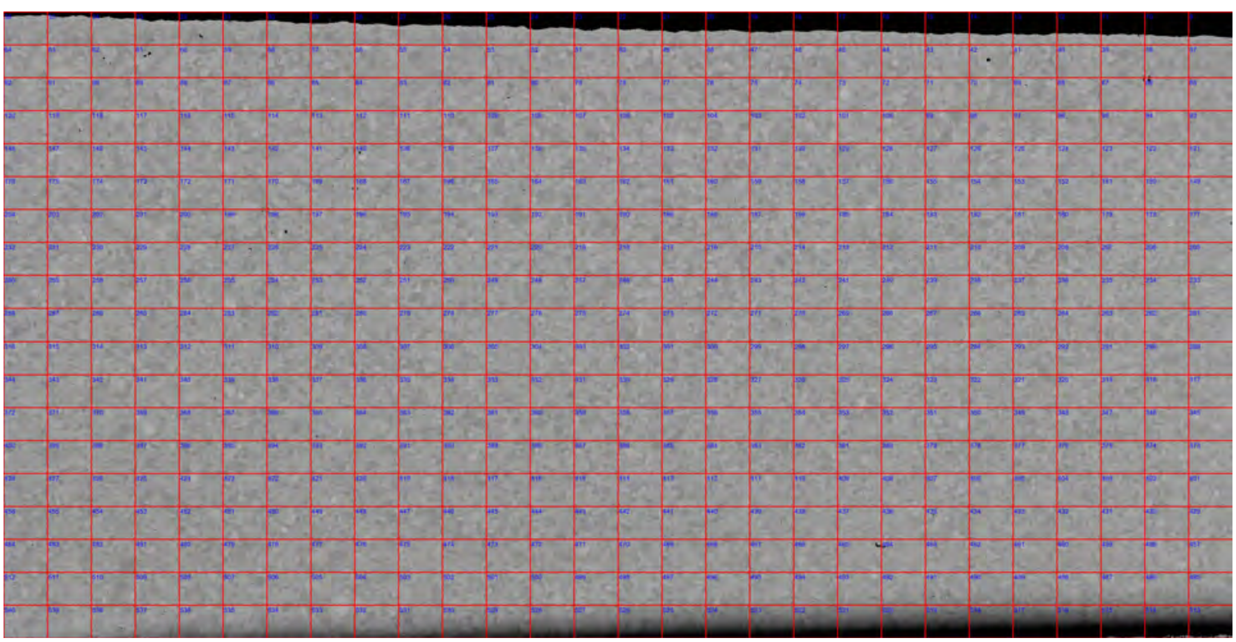

Figure 7.21. Full Stir Region SEM Montage with Tile Map: Condition C00, Sample SS36

7.3 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.

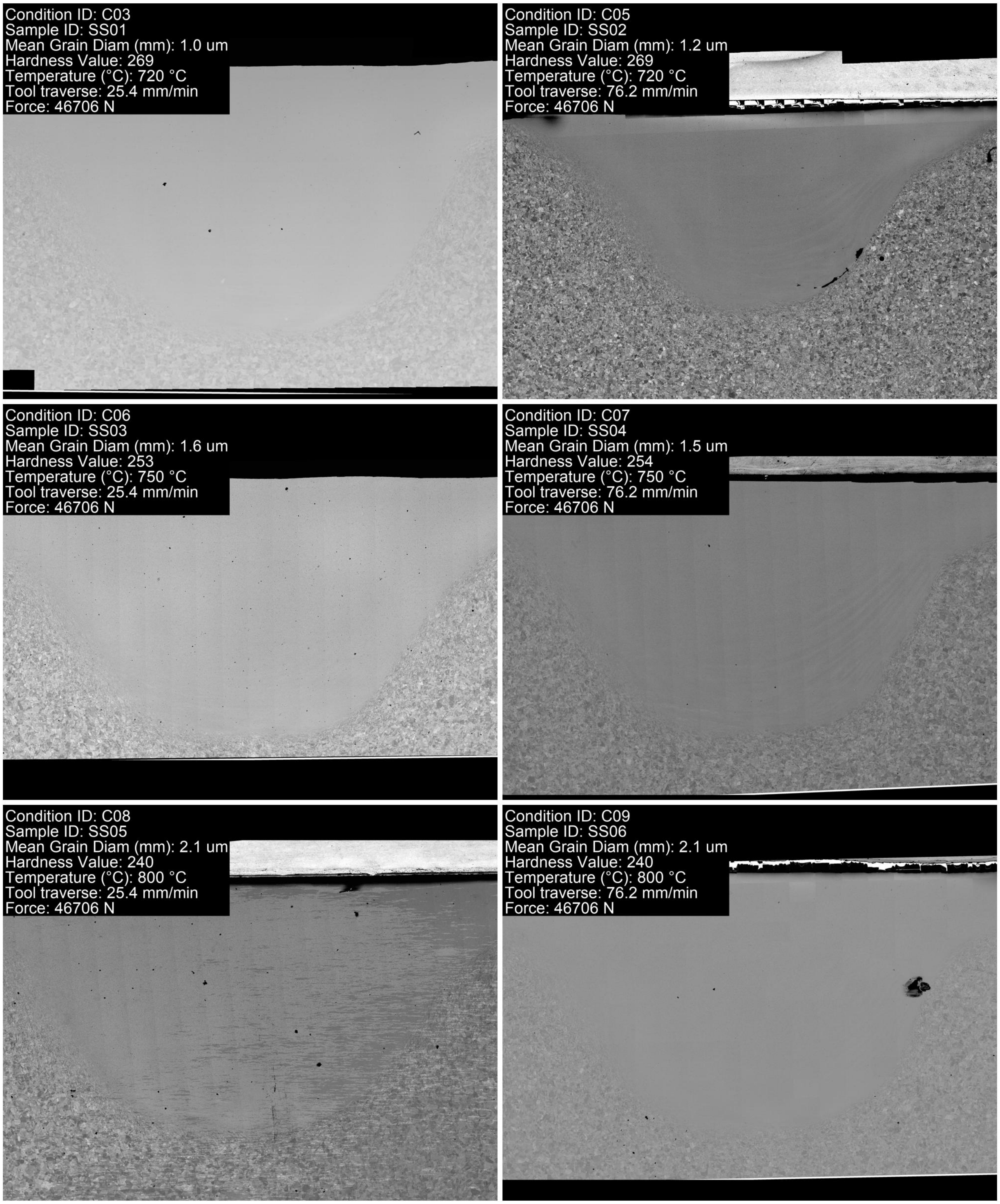

Figure 7.22. Stir Region Collage: Image number 1

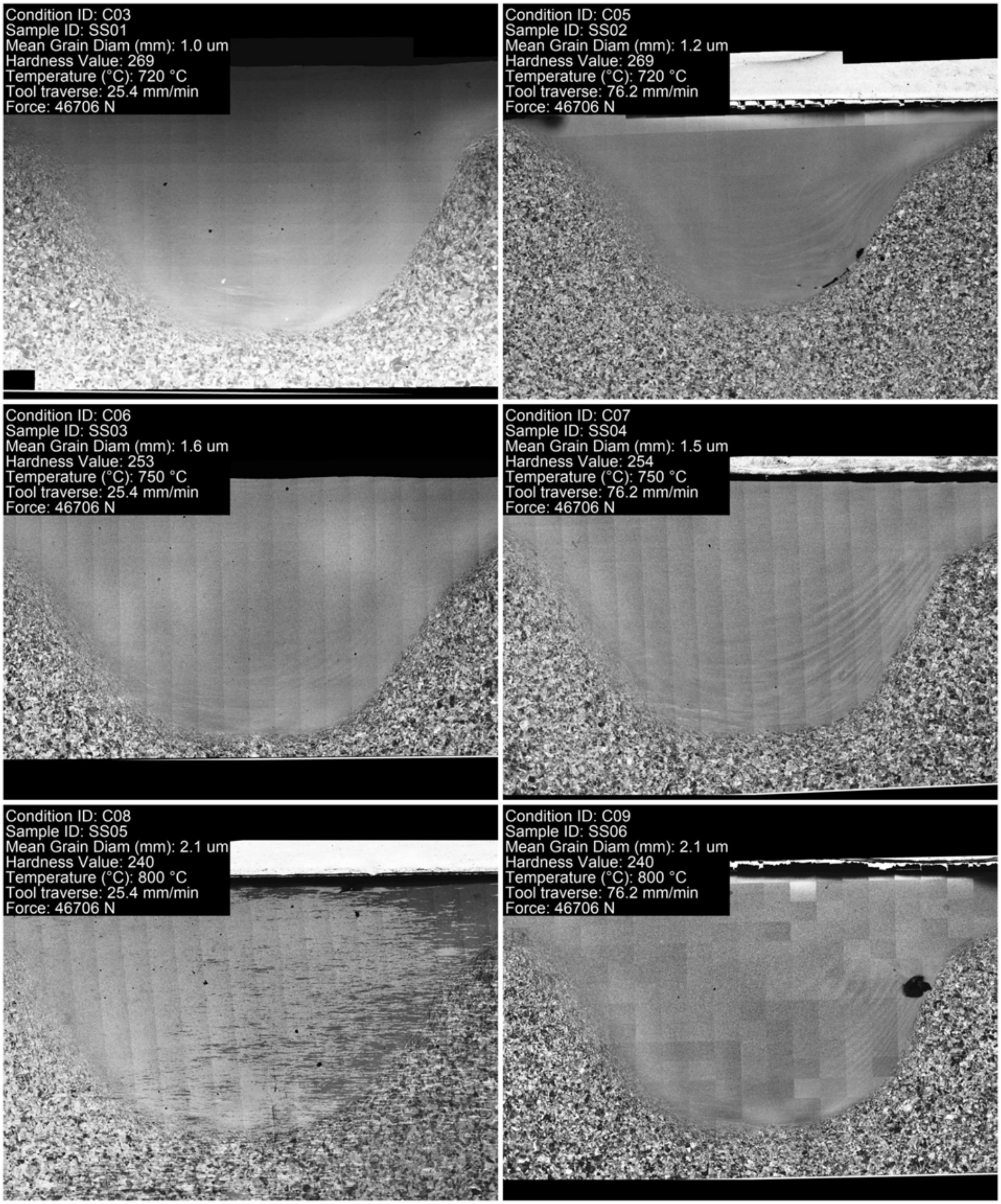

Figure 7.23. Stir Region Collage: Image number 2

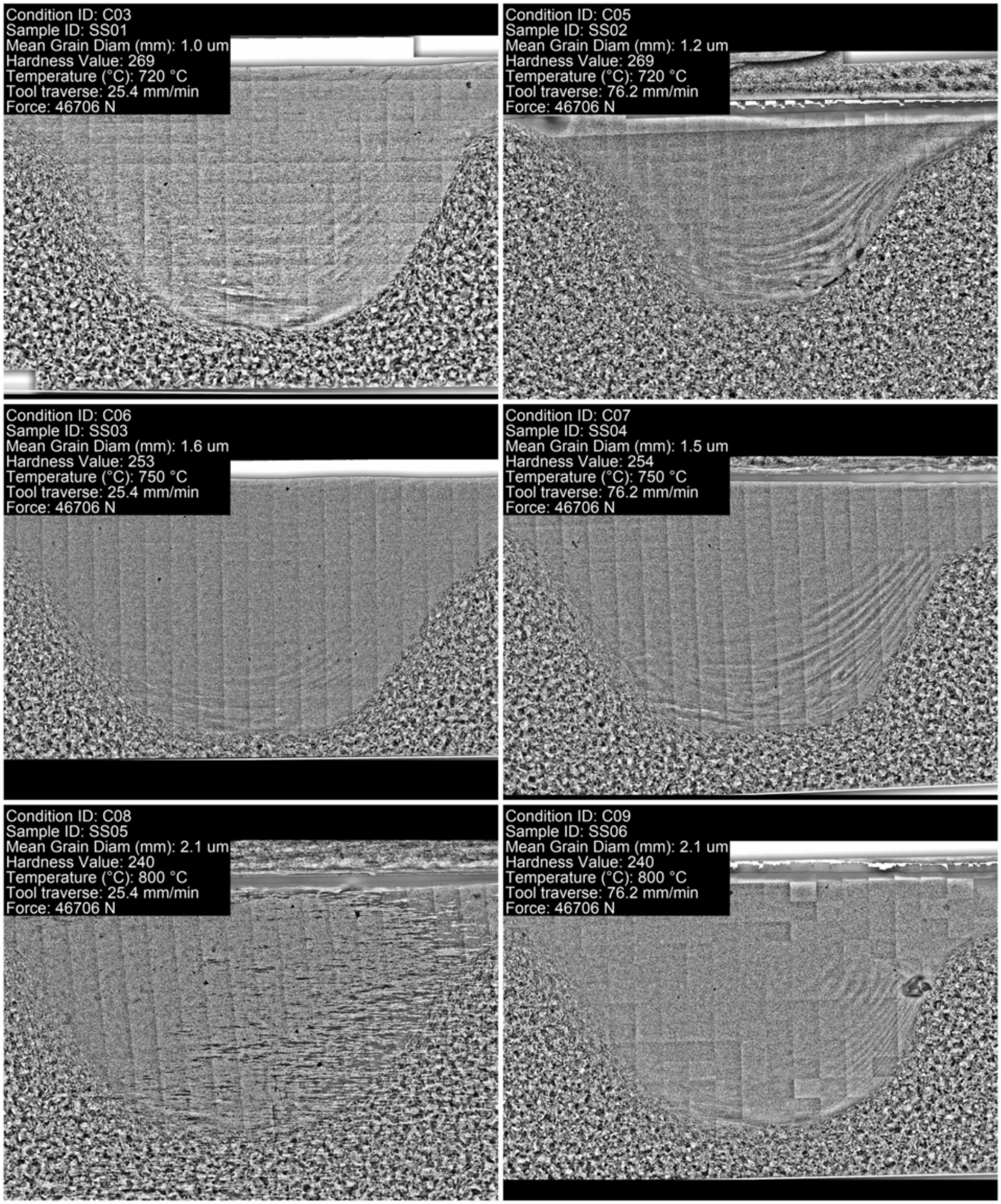

Figure 7.24. Stir Region Collage: Image number 3

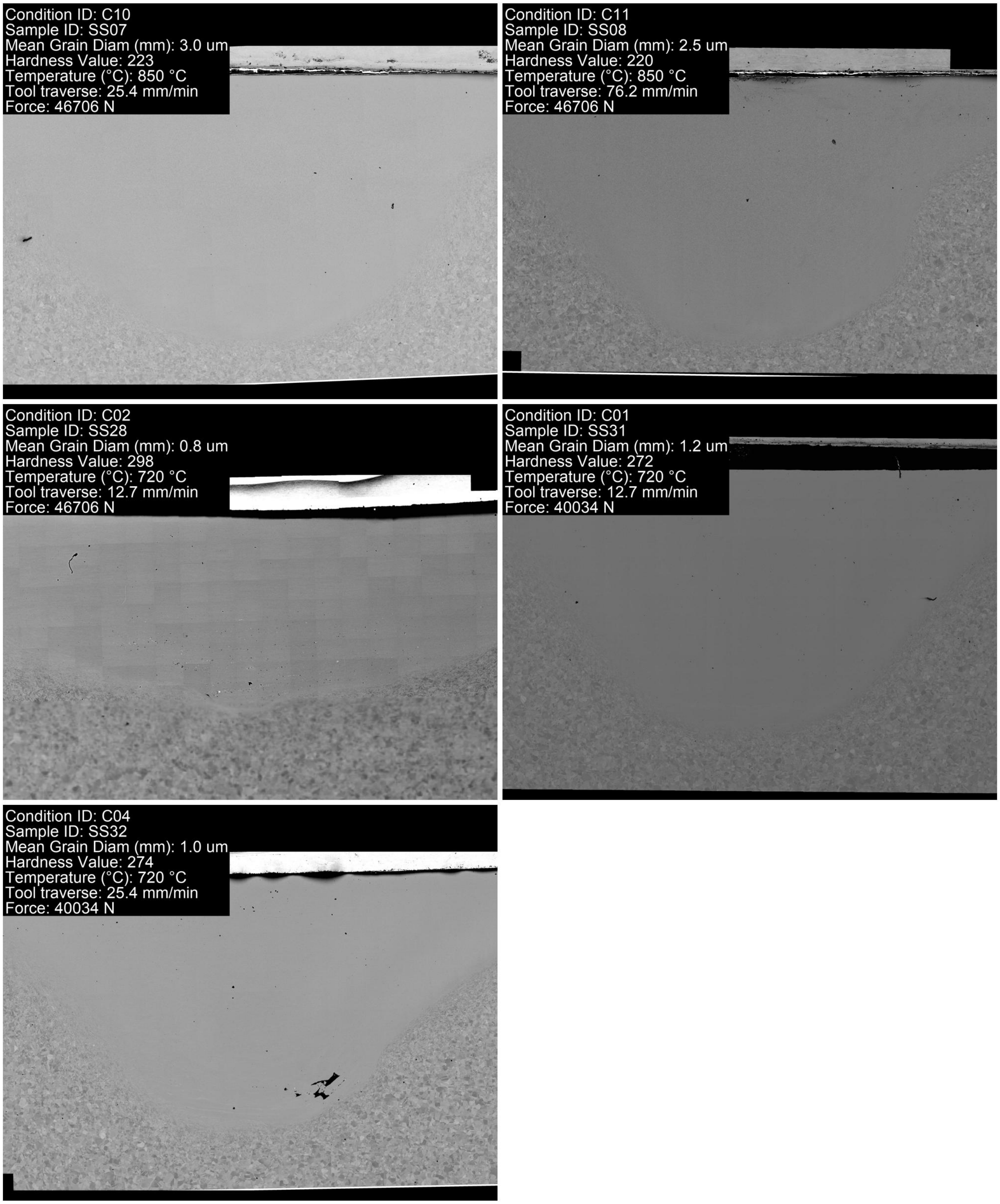

Figure 7.25. Stir Region Collage: Image number 4

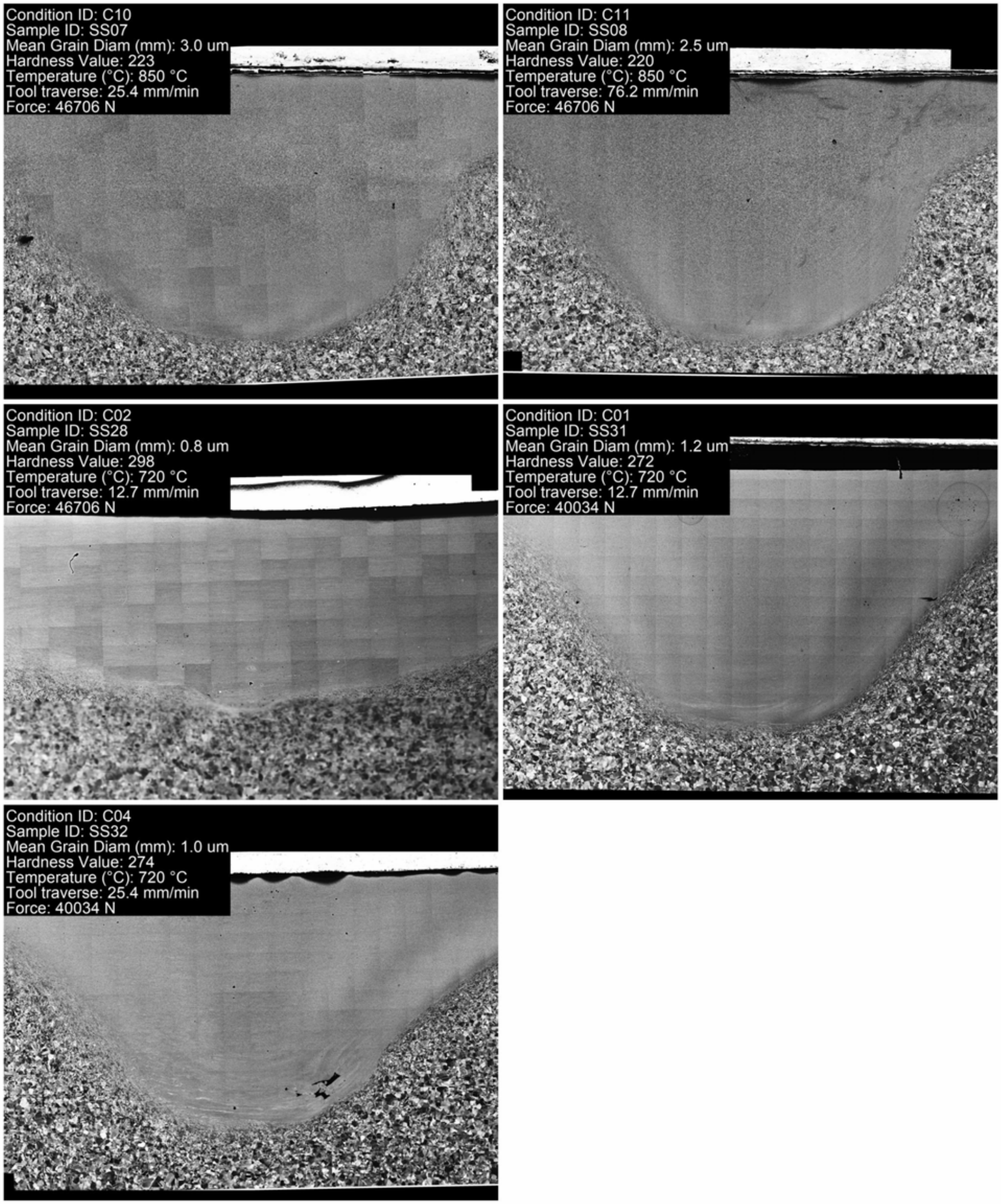

Figure 7.26. Stir Region Collage: Image number 5

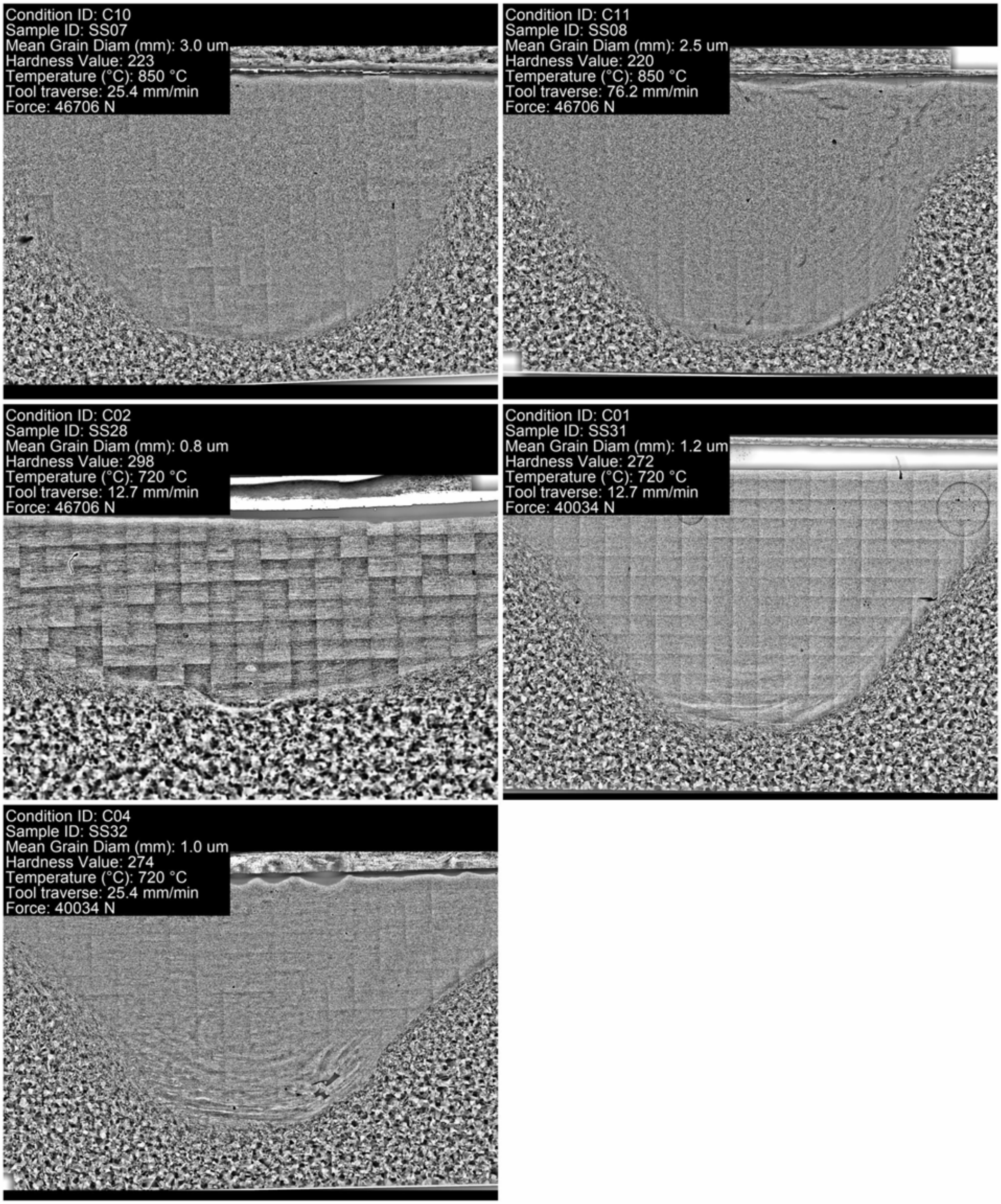

Figure 7.27. Stir Region Collage: Image number 6

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Attachment E: MCPC Round 1 Material Characterization Results for Modality ULTRASONIC

Y Guo K Nwe
DR Todd N Conway

Attachment E contains 19 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

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. or 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:

- There were three scans performed for the sample SS08 (Specimen MCPC0038) and the data is in the folders SS08_0018, SS08_0050, and SS08_0051, respectively. The data in the folder SS08_0018 was later determined to be wrong and invalid. The data in the folders SS08_0050 and SS08_0051 is valid.

- There were two scans performed for the sample SS07 (Specimen MCPC0033) and the data is in the folders SS07_0020 and SS07_0023, respectively. The data in both folders is valid.

- There were two scans performed for the sample SS32 (Specimen MCPC0158) and the data is in the folders SS32_0009 and SS32_0024, respectively. The data in both folders is valid.

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
SS01	C03	1.56	0.0552
SS02	C05	2.04	0.073
SS03	C06	2.16	0.0964
SS04	C07	2.42	0.1292
SS05	C08	2.82	0.1634
SS06	C09	3.36	0.2211
SS07	C10	5.72	0.5405
SS08	C11	4.52	0.3971
SS28	C02	1.34	0.0671
SS31	C01	1.40	0.0519
SS32	C04	1.42	0.0471

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.

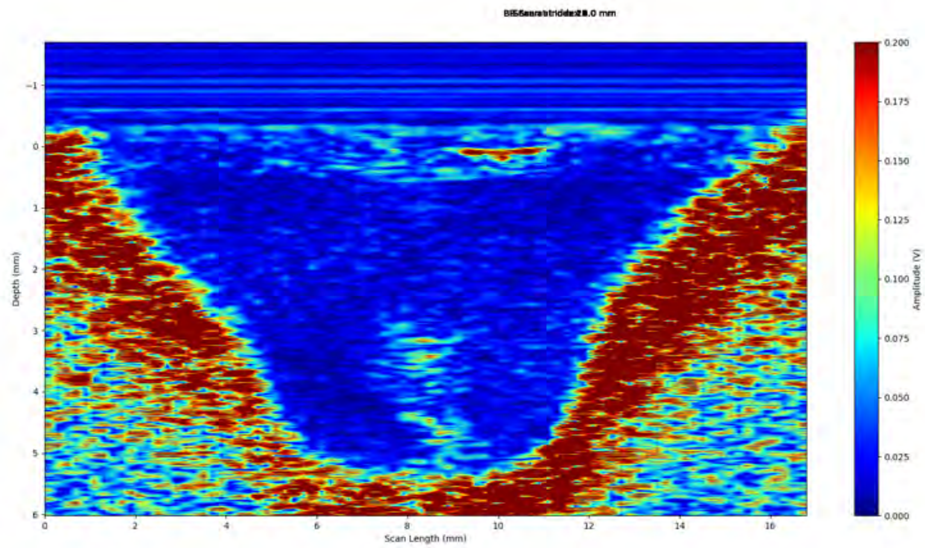

Figure 8.1. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C03, Sample SS01

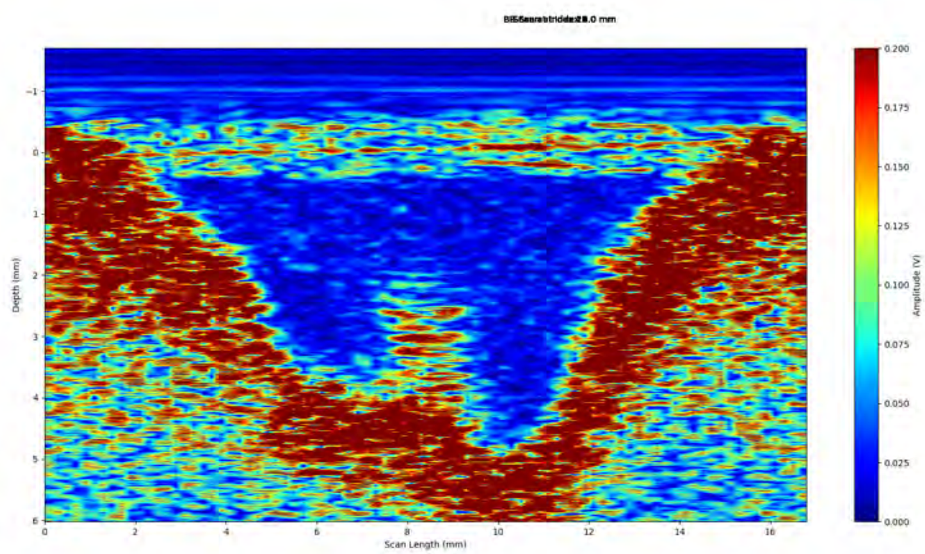

Figure 8.2. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C05, Sample SS02

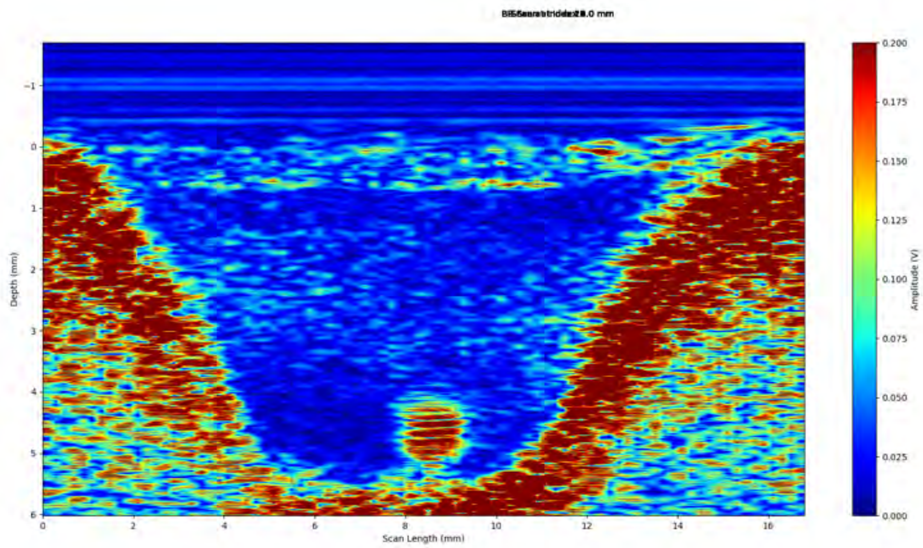

Figure 8.3. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C06, Sample SS03

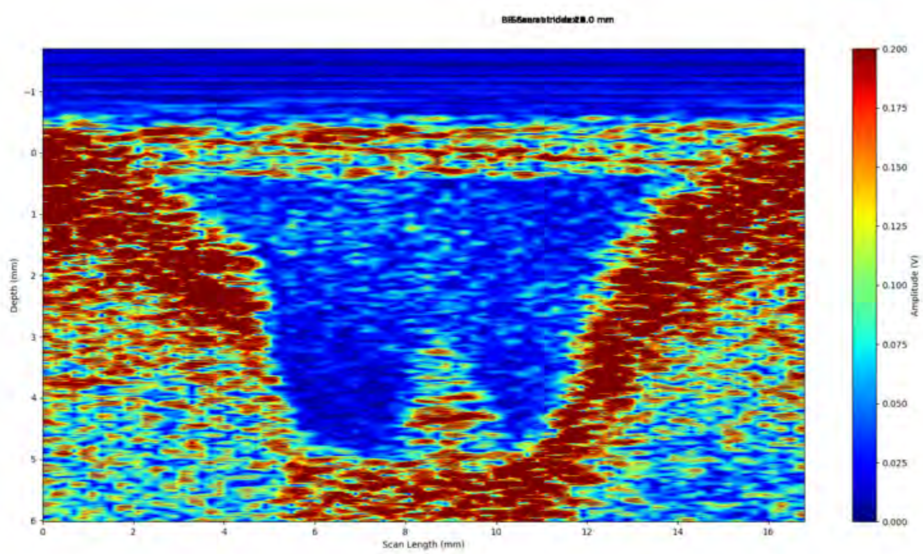

Figure 8.4. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C07, Sample SS04

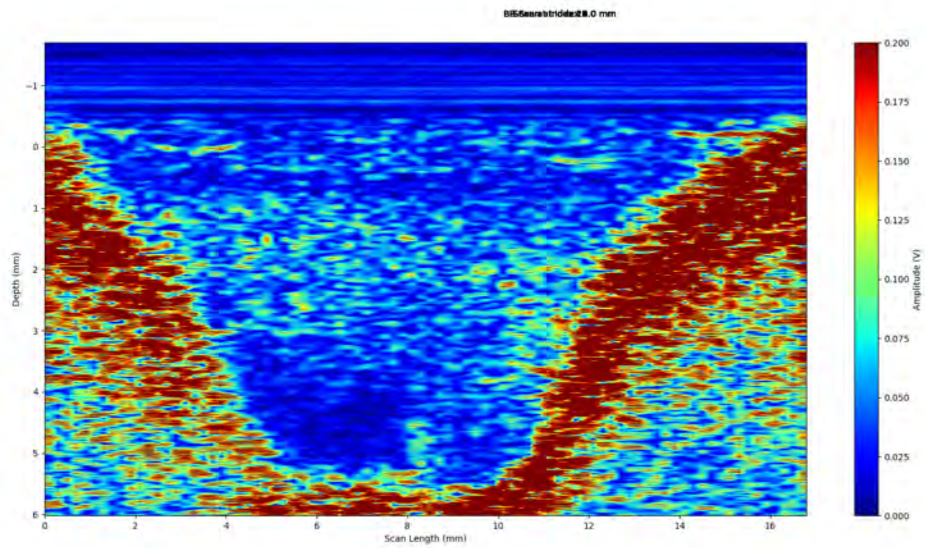

Figure 8.5. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C08, Sample SS05

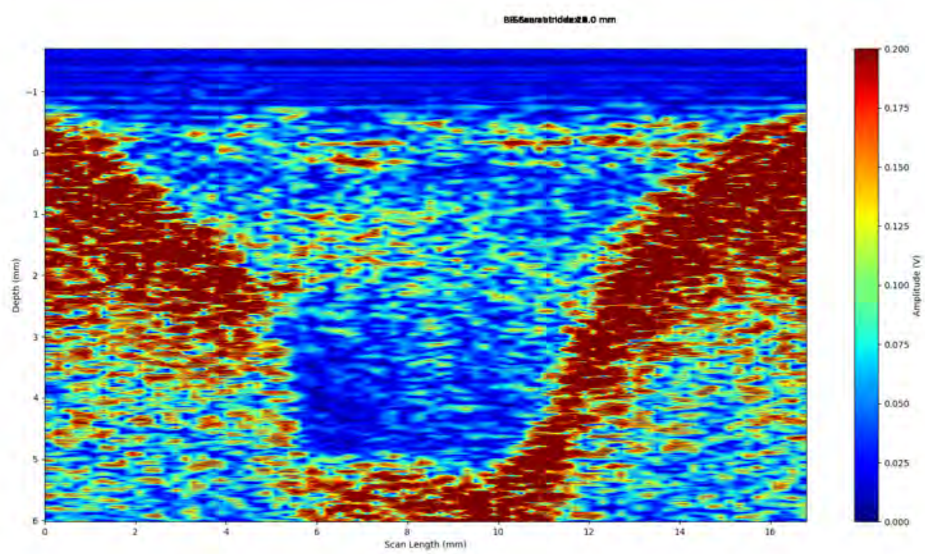

Figure 8.6. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C09, Sample SS06

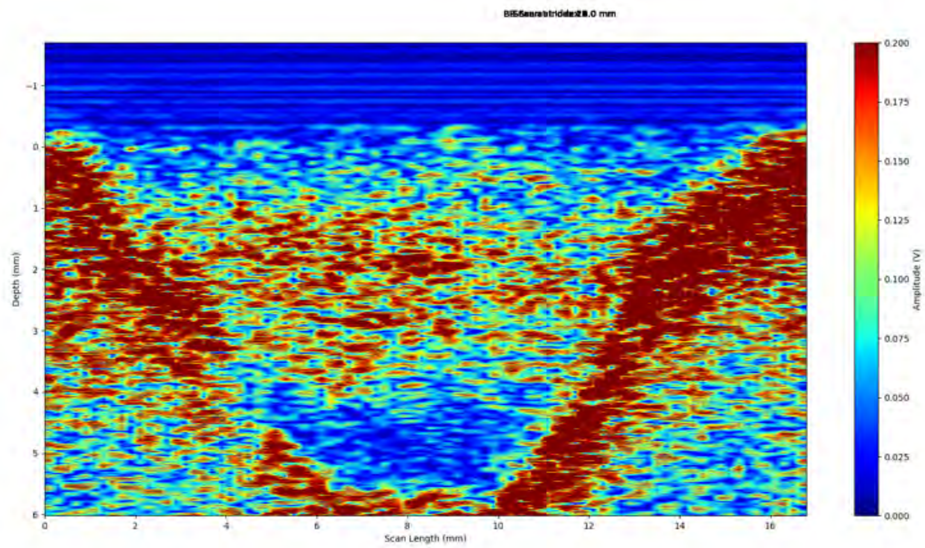

Figure 8.7. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C10, Sample SS07

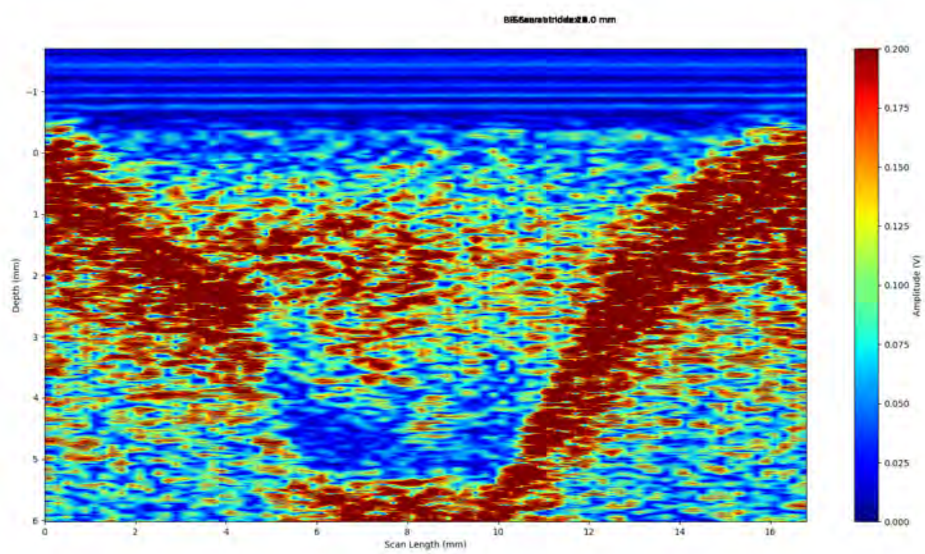

Figure 8.8. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C11, Sample SS08

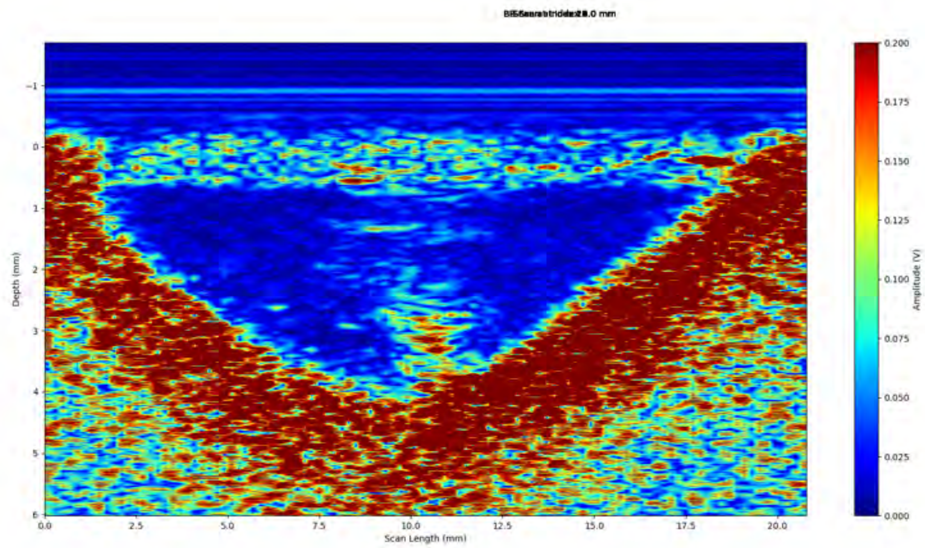

Figure 8.9. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C02, Sample SS28

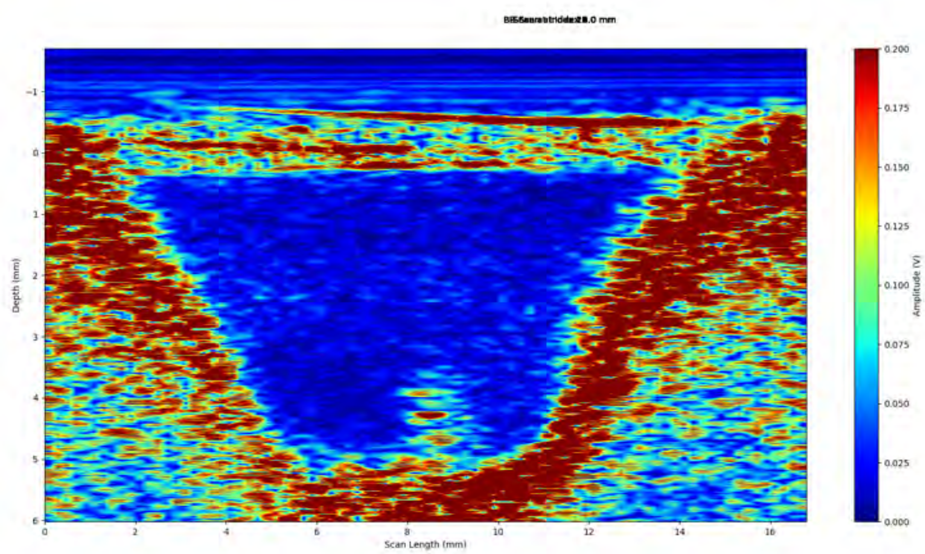

Figure 8.10. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C01, Sample SS31

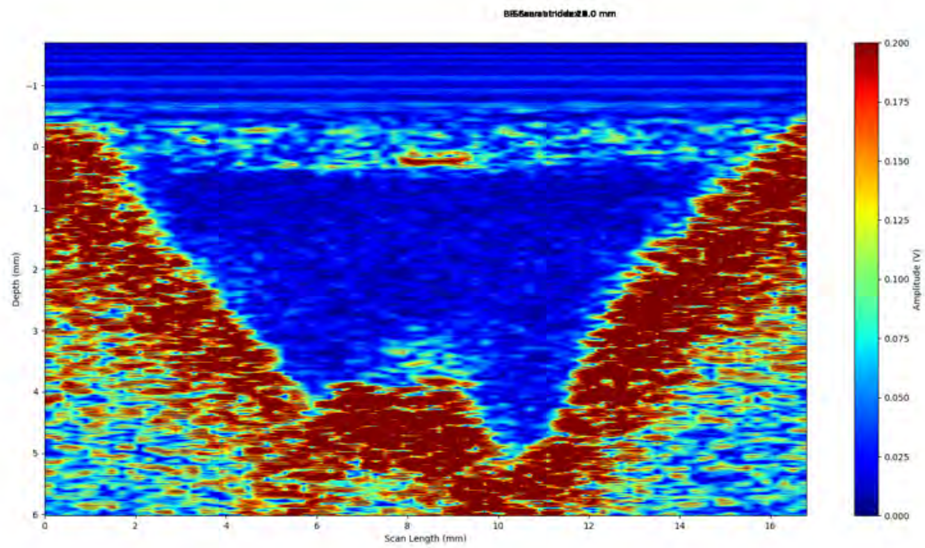

Figure 8.11. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C04, Sample SS32

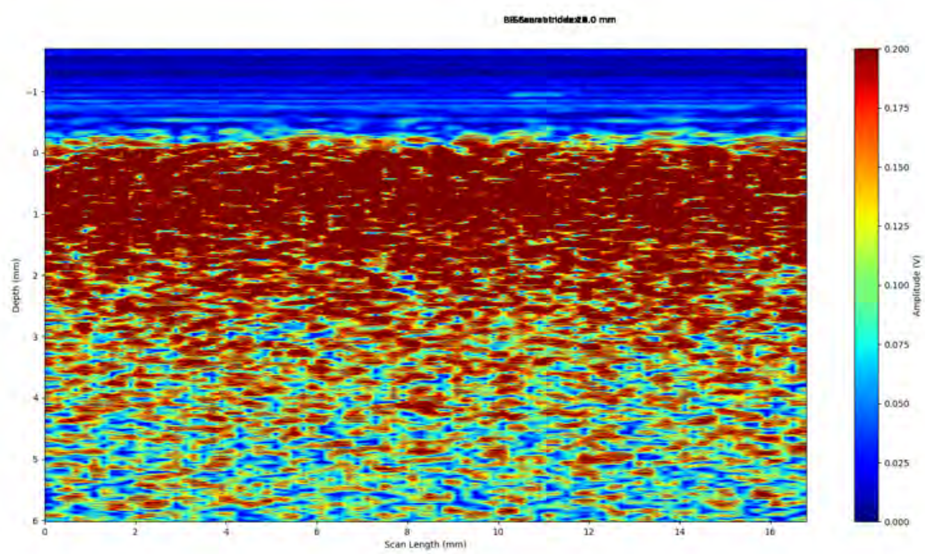

Figure 8.12. Transverse wave grain noise (GN) configuration (mid-specimen): Condition C00, Sample SS36

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