**Georgia Institute of Technology**

**Marcus Characterization Facility**

**Standard Operating Procedure for Residual Stress by X-Ray Diffraction**

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**1.0 Purpose**

The measurement of residual stress using the Empyrean X-Ray Diffractometer.

# 2.0 Scope

This work instruction covers residual stress measurements using the Empyrean X-Ray Diffractometer for aluminum samples around .1” to 2” diameter or edge length in planar view.

Any changes to the procedures defined here must be reviewed and accepted by the laboratory lead. Non- standard test methods should be discussed and approved by the customer, and documented by LIMS, email, and/or written in the lab data sheet.

Only fully train personnel or personnel in training supervised by a fully trained personnel are allowed and authorized to run this test. Fully trained personnel will have a unique log in ID and password for the XRD software system (Data Collector) they will use to log into and run this test.

# 3.0 Abbreviations and Definitions

EHS Environment, Health and Safety

XRD X-Ray Diffractometer/Diffraction

PPC Parallel Plate Collimator Monocap 0.3 Monocapillary Incident Optic FWHM Full Width Half Maximum

# 4.0 Environment, Health and Safety Considerations

The X-Rays from the XRD should be contained within the system itself. The doors must be closed for the system to open the X-Ray shutters, and the glass of the doors are leaded to prevent leakage.

The windows of the XRD tube are beryllium, and, if the tube is ever to be taken out, rotated, or handled, the windows should never be touched.

Several accessories, such as the Chi-Phi-Z-Axis stage, are heavy, and care should be used when moving them, as for changing of the stage. A cart should be used to move accessories from the XRD to other areas of the lab, such as the XRD cabinet.

No specific environmental condition is required for a normal stress measurement.

# General

* 1. **Equipment**

Empyrean X-Ray Diffractometer Copper X-Ray Source in Point Focus

Polycapillary X-Ray Lens Parallel Plate Collimator Point Detector

Height Sensor

3 – Axis or 5 – Axis Stage Cradle or Spinner Stage Sample Leveling Press

Torx Drivers

3 Nm Torque Limiting Wrench 10 Nm Torque Limiting Wrench

# Consumables

Modeling Clay or Double-Sided Tape for sample mounting

# Reference/ Supporting Documentation

Empyrean Manual (software document on PC)

XRD Sample Size calculator (‘XRD Sample Size v2.xls’)

# Tolerances

+/- 0.05 mm Z

+/- 0.5° Ω

# Required Accuracy/ Precision

Highly textured samples will give ambiguous results. Therefore, this method should only be used on modestly texture samples.

Samples should be relatively flat, such that the curvature of the sample will not create a shadowed measurement. Smaller beam sizes can be used to compensate for slight curvatures.

Uncertainty of measurement can be estimated from the stress free tungsten reference sample.

# 6.0 Instrument Calibration/ Verification

The XRD does not have a method to calibrate.

Reference samples are used for integrity verification whenever any servicing is made and/or if any measurement shows any question of the quality of measurement. The reference sample to be used are the stress free tungsten reference sample supplied by Panalytical.

The Torque Limiting Wrenches are not calibrated and are for reference only to tighten the bolts to approximate levels of torque.

# Method Detail

* 1. **Sample & Preparation**

Samples should be relatively flat and between about 0.1” to 2” wide (diameter or smallest edge). Smaller samples will require the use of the microdiffraction optic. If batch running of several samples are to be done (using the 5 – Axis stage), the samples will need to be less than 10 mm in thickness.

Samples should have minimal preparation done, as to preserve the internal strain. Measurements should be done as far away from sheared/cut edges of the samples as possible.

Samples, once received from customers, are stored in the XRD task request storage shelves. Samples should be marked/labeled or placed in marked/labeled bags to identify them. Samples may be moved out of the shelves once they are close to being run, as long as they will not be confused with a separate test.

Samples should be mounted to the sample stage plate (with the plate removed from the stage cradle first) for the 3 – axis and 5 – axis cradles or the bulk sample ring holder for the spinner stage using clay, tape, or an equivalent method to hold the samples without deforming them. Mounted samples should be

leveled using a leveling press. For the 5 – Axis stage, samples must be mounted such that they are less than 10 mm from the top of the plate.

Samples should be aligned, when mounted, such that the direction of deformation (such as the rolling direction or axial (ironing) direction) or direction of interest, is in line with the X axis of the stage (the X axis is parallel to the beam path when φ = 0°, and is the direction of σxx).

Care should be taken to prevent the samples from being mounted too far off the plate, as during scanning this could cause collision of the sample with parts of the instrument. The stage plate can be removed/installed using a torq driver, and should be placed back on with the guide pins in the correct hole locations. Installation/Removing/Changing of the XRD accessories are usually installed by MCF Staff or by advances users.

Samples may be identified by them being in individual bags or by direct markings on the samples with a marker. Marking are samples may be made as long as the marking does not deform the sample.

Samples may be marked with simplified identification numbers, center points, and alignment arrows to assist in identification and alignment during measurement. For highly reflective samples, which can cause the height sensor to incorrectly determine the sample height, the surface may be smudged with clay or similar non-crystalline substance to reduce the reflectivity, as long as the amount is trivial.

# Testing Frequency

4 Phi steps should be used for each measurement.

# Instrument Preparation

The XRD should be set up in point focus with the Copper source. The incident optic should be the Polycapillary X-Ray Lens or the monocap. The diffracted optic should be the PCC (with Nickel Beta filter) with the point detector, set in detector path 1. The X-Ray Source generator should be set to 45 kV/40 mA.

The X-Ray sample mode must be in Reflection mode. If this is not the case, change the sample mode by opening the Sample Stage option under Instrument Settings in Data Collector. Press Change Sample Mode to switch to Reflection. Care must be taken to avoid instrument collisions, as the optics will shift to be horizontal to the stage. If needed, remove all accessories first before changing modes. The stage should be the spinner stage before changing modes. If a stage change is required, do so after the mode is already in Reflection mode.

For a large (thick) sample, the 3 – Axis stage cradle must be used. The different leg pegs can be used to raise the sample to the sufficient height for measurement.

For batch scans, the 5 – Axis stage cradle must be used. This stage is limited to samples less than 10 mm in thickness.

If using the monocap, care must always be taken to prevent the optic from colliding with the stage or sample.

Installation/Removing/Changing of the XRD accessories are covered in WI-ME-2000, including the configuration for using a monocap.

# Running a Test

For each sample, the correct Z position (height) must be recorded. For the 5 – Axis stage, the (X, Y) positional coordinates must also be recorded. For the 3 – Axis stage, (X, Y) will be (0, 0). The spinner stage will not require height adjustments, and any procedural steps relating to height adjustment can be ignored.

Using the smallest sample as the sample size, find the beam height and width using the XRD Sample Height calculator which will not be larger than that size. Adjust the X-Ray Len’s height/width knobs to the correct values for these samples. Note that the 2ϴ for these scans will need to be set to approximately 137.4°, and the Chi angle will be 0°. If using the monocap, the beam size will be approximately 1 mm.

Before positional alignments, the 2ϴ offset alignment should be done, if it has not already been set beforehand. With the samples completely out of the beam path (and all XRD parameters at 0), make a Manual 2ϴ scan of 0.5° range. The copper 0.2 attenuator and alignment slit should be inserted in the X- Ray Lens, and the 0.4 Rad Soller Slit should be put into the PPC. Move the position of the line to the top of the peak, and set the fine calibration offsets to 0. Remove attenuator, slit, and soller before texture measurements. If using the monocap, this step is skipped, as the monocap should not be used at 0° 2ϴ.

To find the (X, Y) positions for each sample, the alignment camera can be used. Move the stage until part of the sample to be measured (normally the center) is under the crosshairs. Record the (X, Y) values for each sample in the Data Sheet.

Two methods are acceptable for finding the Z position for each sample: using the Height Sensor Method or the Split Beam Method. The split beam method should not be used with the monocap.

For the Height Sensor Method: This method is reliable as long as the sample is not too reflective, and it can be used even if the arrangement of samples on the plate have multiple samples in line with the beam path. The Height Sensor should be installed and plugged in. For each sample, move to its position (if applicable), and align its height with the height sensor. This should be done at least twice, until two consecutive measurements are within +/- 0.05 mm of each other. Record Z for each sample in the Data Sheet. Remove the height sensor before texture measurement. When aligning the height using the monocap, remove the monocap optic first, in order to help prevent collisions. Return the monocap after the correct height is determined.

Split Beam Method: This method is used when more accurate heights are required, as well as when the sample level is important and the Ω offset must be set as well. For this method, multiple samples cannot be in line with the beam at the same time. The copper 0.2 attenuator and alignment slit should be inserted in the X-Ray Lens, and the 0.4 Rad Soller Slit should be put into the PPC. For each sample,

move to the sample position and set Z to the approximate height of the sample. Run a manual Z height scan with a range of 2 mm. If the scan shows a straight line above the background level only, then the sample is too low still. Raise the Z 2 mm and scan again. If the scan shows a straight line which is only a background level intensity, the sample is too high. Lower the Z 2 mm and scan again. Once a scan shows the line decreasing from a high to low value, the sample height is between the two positions where the line is high and low. Place the Z value where the intensity is approximately ½ the counts between the difference of the approximate high intensity and the approximate low intensity. At this Z, run a manual Ω scan with a range of 2°. If no peak is apparent, increase the range to 4°. If no peak is still apparent, the Z may be incorrect (repeat the Z alignment above) or the sample may be too curved or not leveled for Ω to be corrected (leave Ω offset to 0 in this case). If a peak is observed, move the line to the top of the peak. Repeat the Z alignment above, and then repeat the Ω alignment. This should be done at least twice. Once the values for Z and Ω do not change (to a tolerance of +/- 0.05 mm for Z and +/- 0.5° for Ω), record the Z and Ω offset value in the Data Sheet.

Before residual stress measurements are made, remove all slits and attenuators from the optics. For any given sample, move the stage to its recorded position and offset (X, Y, Z, Ω offset). A stress measurement is needed for each aluminum sample at [422], which is approximately 137.4° 2ϴ. Run a simple, Manual 2ϴ measurement for each sample at that angle, and determine the exact peak location, as well as the range needed to cover the whole peak. Record the peak location, range, and the Full Width Half Maximum. Create a Stress Measurement in Chi tilt axis, with Positive + Psuedo Negative tilt range. Set the max Psi limit to 15°. The scan axis type should be 2ϴ-Ω, with actual Ω at start, in continuous mode. 4 Phi steps should be used. The Step Size should be smaller than 0.1 \* the measured FWHM. Use the range measured in the manual scan. Scan times can vary, depending on the intensity of counts which is wanted, but 1 second is usually adequate. Each test scan should have a unique name in each task request which includes the name of the sample.

For batches, each sample location will need to be added to a General Batch Program. Each sample will have a Batch Setting of its (X, Y, Z, Ω offset) and then a Stress Measurement program for each sample individually. The smallest step size, and largest range of all the samples can be used. If using the monocap, care must be taken that none of the batch settings will result in collision with the optic.

For each task request, the 2ϴ offset, beam height and width, and the smallest sample size is to be recorded in the Data Sheet. Any errors or additional comments should also be recorded in the Data Sheet.

After all samples are scanned, the XRD should be put into standby mode by lowering the generator to 45 kV/ 20 mA.

# Data Handling, Analysis & Reporting

Residual Stress measurements are calculated using Panalytical’s Stress software.

Samples are retained and stored in the small sample storage racks, with the Task Request Number marking each set of samples, unless otherwise requested by the customer.

# 8.0 Revision History

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| **Revision** | **Revision Date** | **Summary of Changes** | **Author** |
| O | 10-21-2017 | Initial creation of document | David Tavakoli |
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