EASI-STRESS

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EUROPEAN ACTIVITY FOR STANDARDIZATION OF INDUSTRIAL RESIDUAL STRESS CHARACTERIZATION

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EASI-STRESS

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CHADA
FE Finite Elements
GV Gauge volume
LRI Large-scale research infrastructure
NQL Neutron Quality Label

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Executive summary

A common framework for residual stress measurement techniques at Large-scale Research Infrastructures (LRIs) has been prepared. The framework will serve as a platform to enable validation and benchmarking of the different techniques and will aid in the development of harmonised data output from the LRIs for better reproducibility between the facilities and easier and more reliable exploitation of measurements from an industrial point of view. The construction of the framework is based on the adaptation of the CHADA structure, which is the standard structure for representing materials characterization data in Europe. The commonalities between the different techniques are highlighted, and the final output of workflows and compiled metadata descriptors for residual stress measurements at LRIs are presented.

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1. Introduction

In the last few decades, it has become a common knowledge that residual stress is an important design factor for engineering structures (Hutchings et al., 2005). A detailed survey conducted to UK industries in 1998 revealed that the majority ranked residual stresses as a medium to high importance to their businesses, with an even split regarding the interest for bulk and near surface residual stresses (Kandil et al., 2001). Presently, a large number of residual stress measurement techniques are accessible for material scientists and engineers with its own characteristics, resolutions, and limitations (P.J. Withers & Bhadeshia, 2001). Among those methods, strain scanning techniques at Large-scale Research Infrastructures (LRIs) using neutrons and synchrotron X-rays have gained tractions in the recent years. This is made possible by the development of high flux sources, detector technologies, and data analysis tools (Webster, 2003), and also due to the increase in the complexity of parts and fabrication processes in engineering components, which necessitates high-quality residual stress measurement methods.

Neutrons and X-rays are complementary and each have distinct characteristics. They both require large, high capital cost LRIs that are often used on a competitive, timeshare basis. Therefore, it requires a thorough preparation and decision-making process to match each case study with the suitable methods and techniques available at the LRIs to optimise the available resources. Equally important, a harmonised framework is required in order to find the relevance between each stage of the workflow of these complementary techniques, and to help defining the metadata that describes each measurement. This framework should lead to the standardisation of the output (data format), which will guarantee thorough and consistent reporting. This is crucial, not only to ensure the repeatability, verification, and benchmarking between the two techniques and towards other laboratory-based methods and simulations, but also to facilitate the interpretation and exploitation of the result in industrial context (e.g., input for FE-analysis).

Among the objectives of the EASI-STRESS project is to provide answers to those needs. In the Work Package 3 (WP3), specification of various LRI instruments and technique is defined and user advisory group is established to ensure the optimum allocation of resources for each measurement. This report is prepared as a part of EASI-STRESS WP4 deliverables to define and introduce a common framework for neutron diffraction and synchrotron X-ray diffraction for residual stress measurement. The framework will serve as a platform for future activities in the work packages, including:

- Selection of common data format
- Development of standardised data analysis method and software
- Implementation of these protocols and software on benchmarking and demonstrator samples.

The framework will be largely based on the CHADA, a recently developed concept for data documentation in materials characterisation (Romanos et al., 2019). Workflows and metadata descriptions in the CHADA format will be prepared for each technique, with an example for a particular method presented in this report. Commonalities between the methods are drawn, and finally the recommendations for future activities are presented.

2. Background

Generalized concept & workflows for residual stress measurement at LRIs

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The general concept for both neutron diffraction and synchrotron X-ray diffraction for stress determination is similar: measurement of changes in the interatomic distances (lattice spacings) of crystalline materials averaged over a certain gauge volume (GV), i.e., the volume from which the measured information is obtained. From the neutron or X-ray diffraction patterns, which spread as a function of diffraction angle or wavelength, the lattice spacings are determined using Bragg's law. Strains are determined by the differences of lattice spacings in the specimen and in the stress-free reference (so-called d_0). Using Hooke's law of elasticity, the macroscopic stresses are then calculated from the strains measured in multiple components.

The two methods differ in some aspects: the differences are mainly the consequences of the beam source being used. Neutrons penetrate well within most relevant engineering materials at wavelengths around 1.5-3 Å, providing diffraction angles near 90° and consequently a near-cubic GV. This means the GVs remain similar for measurement of multiple strain components. To reproduce equivalent penetration depth, high energy synchrotron X-ray typically corresponds to wavelength of 0.05-0.25 Å are required, providing diffracting angles below 15°. This means very high lateral resolution can be achieved along selected directions, with a penalty of poor resolution on the other directions. Additionally, modern synchrotron X-ray sources can provide very intense narrow beams compared to neutrons, which leads to a possible faster measurement and higher strain and spatial resolution.

Despite their differences, neutron diffraction and synchrotron X-ray diffraction share similar components in their measurement workflows since both are diffraction-based methods. Commonalities between the two methods are as follows:

- Diffraction patterns are recorded as a function of diffraction angle, time-of-flight, or wavelength. In order to provide an accurate correlation between these measured parameters and lattice spacings, calibration measurements using a set of standard samples are usually required.
- The GV is defined by means of defining optics (slits, radial collimators). The GV generally needs to be aligned/placed accurately at the center of the instrument before measurements in order to: i) facilitate efficient measurement of multiple strain components, ii) ensure the positional accuracy of the measurement points. Therefore, some form of instrument alignments needs to be carried out.
- To obtain strain, measured lattice spacings from specimens are compared against reference values given by the lattice spacings of stress-free samples.
- To facilitate measurement on series of measurement points (scans) and on multiple strain components for stress mapping, specimens are usually mounted on top of sample manipulator system, which may provide translation, tilt, and rotation (e.g., xyz-table, hexapod, cradle, robot arm, etc.).
- Algorithms for data analysis usually consist of data reduction, fitting of the diffraction spectrum, strain calculation, and finally stress determination

Considering the similarities between the two methods, generalized metadata can be defined to describe experimental parameters.

There are sub-categories/techniques currently available for both neutron diffraction (monochromatic and time-of-flight technique, and more recently-developed Bragg edge imaging) and synchrotron X-ray diffraction (conventional θ/2θ scanning, 2D transmission, and white beam), each of which has certain advantages and disadvantages. Therefore, it is crucial to precisely define the measurement problem in the very first step, in order to select the most appropriate probe. This includes the

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definition of the measurement region and resolution, expected result, and pre-characterization of specimens (grain size, phase, presence of texture, etc.).

Introduction to CHADA

CHADA is a standard structure for representing materials characterization data, representing a novel approach for the definition of terminology, classification, and metadata for materials characterization methods (Romanos et al., 2019). CHADA is one of the outputs of the OYSTER project, with the aim of developing standard procedures for the classification of data, the classification of experimental protocols and the definition of standard taxonomies and ontologies for data representations and linking. Since this structure is designed for integration into an Open Innovation Environment (digital platform designed within the European Materials Characterization Council – EMCC), it would make sense to build the common framework for neutron diffraction, synchrotron X-ray diffraction, and any other materials characterization methods at the LRIs based on the CHADA structure.

Based on CHADA, there are four types of concepts proposed for the classifications of the different steps of an entire characterization workflow, namely:

- **Sample (or "user case")**, representing a volume of probed material, and the information on the surrounding environment, which interacts with the probe and generates a measurable information.
- **Method,** representing a physical process or sequence of processes by which the metrological chain is defined.
- **Raw data**, which is a set of data given directly as output from the metrological chain.
- **Data processing,** representing any process or sequence of processes by which the data are analyzed to arrive to the final shape.

A visual representation of a general characterization experiment (with keywords and colors) is shown in Figure 1. The construction of the metadata structure of any materials characterization process can be based on the four concepts mentioned above. While the scheme also defined the fundamental vocabulary that describes the main elements of a characterization experiment, it can be further refined for any specific technique.

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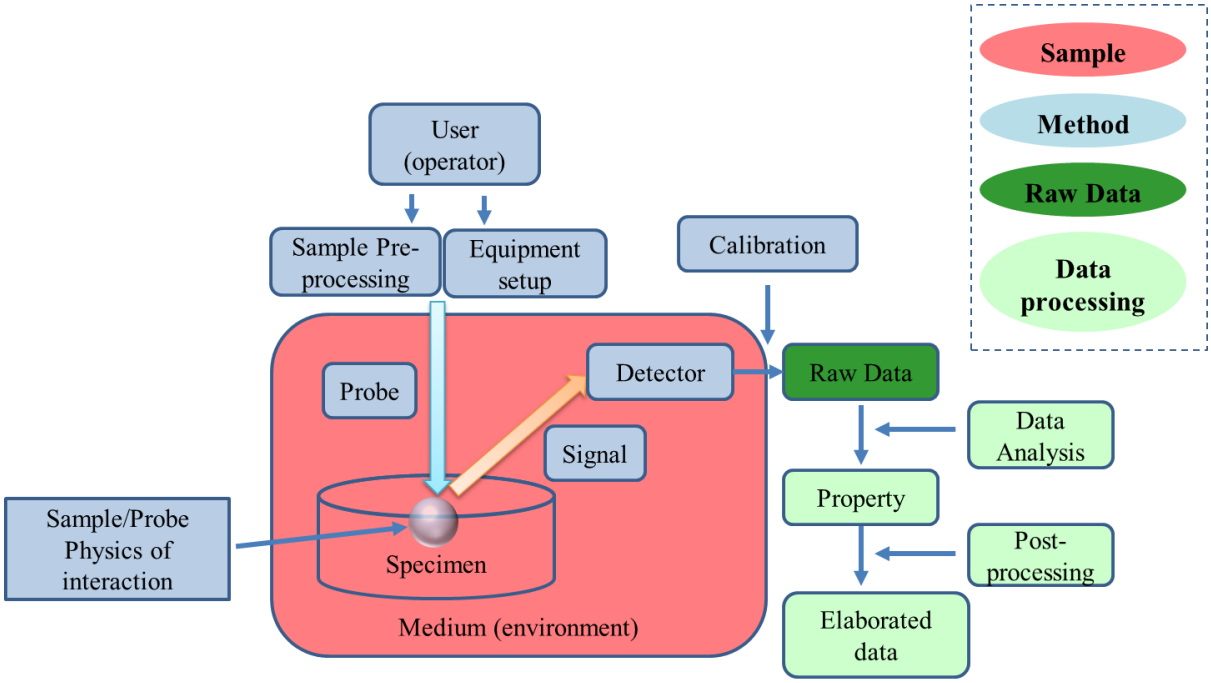

Figure 1 Visual representation of a general characterization method based on CHADA structure, from (Romanos et al., 2019).

Challenges and scopes

The various methods for non-destructive residual stress measurement at LRIs have different levels in terms of technological development. For example, neutron diffraction for bulk residual stress measurements has already gone through various standardisation efforts (VAMAS TWA20, RESTAND AH 138, BrightnESS2 WP2) and standard documents are already available (ISO 21432:2019, Neutron Quality Label – NQL). On the other hand, despite numerous output has been generated using synchrotron X-rays (Ganguly et al., 2006; Philip J. Withers, 2003), as of yet, no general stress measurement guidelines have been developed for the methods.

Due to these differences in technological development levels, there are different challenges in adopting the CHADA structure and defining the workflows and metadata descriptors for each residual stress measurement techniques, before finally compiling them in a common framework. For example, producing the workflows and list of metadata for neutron diffraction only require a small adaptation from the ISO 21432:2019 and NQL documents. Meanwhile, producing the same components for synchrotron X-rays still depends on the knowledge base of the instrument scientists, which might varies depending on the type of the synchrotron X-rays techniques and instrumentations available at each facility.

Therefore, this report serves as a starting point in the conception of the common framework for residual stress measurements on LRIs. Finalised workflows, algorithms, and metadata for synchrotron X-rays, will be completed in as part of the development and standardisation work in WP3 and WP6 of EASI-STRESS. This would also allow future inclusion of recently developed strain mapping techniques such as Bragg edge neutron imaging.

3. Common framework for residual stress measurements at LRIs

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Adoption of CHADA

The adoption of the CHADA structure for residual stress measurement by neutron diffraction, i.e., workflow and compiled metadata according to CHADA classifications, are presented in Figure 2 and Table 1, respectively. This adoption is strictly based on the ISO 21432:2019 standard and NQL documents. The listed procedures, which comprises of sample description, preparation for measurement, the actual data acquisition and recording requirements, and, finally, the calculation of stress, were arranged into four levels in the workflow (Figure 2), namely:

i) sample & measurement preparation,
ii) instrument calibration & alignment,
iii) sample & d_0-reference data acquisition, and
iv) data reduction & post processing.

Each block in the workflow is color-coded according to the type of CHADA concept and represents a set of information that is stored in the metadata. In Table 1, the compiled metadata based on CHADA format is presented: the first column comprises CHADA elements, which compile a group of relevant metadata, and the second column is the description of each element. For the purpose of this report, a column is added to provide a detailed list of metadata. The examples presented in Table 1 is not an exhaustive list, but rather selected examples of the metadata for each CHADA element.

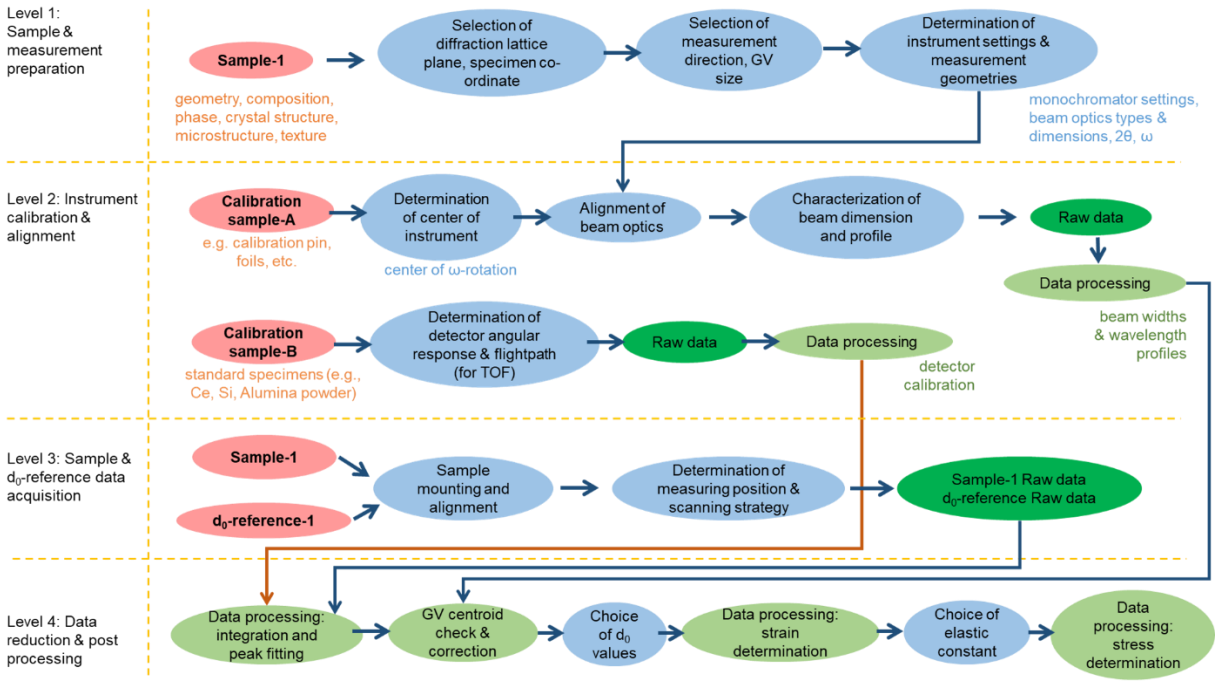

Figure 2. CHADA workflow for residual stress measurement by neutron diffraction

Table 1. Compiled metadata of residual stress measurement by neutron diffraction, based on CHADA structure

Keyword	Description	List of metadata
User case	Specimen; diagram of specimen showing dimensions, fiducial marks or reference locations and specimen co-ordinates; diffraction condition	Sample name Sample geometry & references Material

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		Composition & phase Grain size *hkl* Measured strain component GV dimension …
Reference sample	Stress-free or reference lattice spacing. Can be measurement in a material at a position known to contain a negligible stress, measurement on a powder, which is representative of the material being examined, measurement on a small coupon, cut from a large block of material, or other approaches (including possible extra investigation using other techniques).	Description of d_0 measurement (e.g., small cut of sample, dimension 5 mm × 5 mm × 5 mm)
Calibration sample	For calibration: standard sample with known lattice parameter, diffract neutrons strongly, small intrinsic peak widths (e.g., Ce, Si, Al2O3 powder); For alignment: calibration pin (Ø < 2 mm) and foils (< 1 mm)	Details of calibration sample (e.g., pin with Ø 1mm; foil)
Medium	Mostly air, can be on load-rig for in-situ loading, can be inside a furnace in vacuum or filled with inert gas	Temperature Pressure Load …
User (operator)	Human operator (individuals responsible for instrument, user), experiment details	Instrument responsible details User details Begin & end date of experiment …
Sample/probe physics of interaction	Measurement of lattice spacing inside bulk material, by illuminating a defined volume of material (gauge volume) with neutrons and measuring the angular/ temporal position of a specific sets of constructive interference of the neutrons scattered by the corresponding crystal lattices according to Bragg's Law. For a monochromatic instrument, the specimen is illuminated by a monochromatic neutron beam of a fixed wavelength and its lattice spacing can be determined from the observed Bragg angle. At a time-of-flight (TOF) instrument, the incident beam contains neutrons spanning a range of velocities (i.e., wavelengths) arriving at the sample in pulses. All lattice planes normal to the scattering vector will diffract neutrons to the detector, and diffraction corresponding to each hkl peak is produced by different families of grains. Lattice spacing can then be solved using de Broglie relationship and Bragg's Law.	Type of methods (e.g., monochromatic; Time-of-flight)
Equipment setup	Neutron source and location, name and type of instrument (monochromatic or TOF). Optics components in incident and diffracted beams: for slits: height, width and distance to the reference point shall be specified; for radial collimators: focal length, foil length and thickness, angle between foils, all aperture dimensions and collimator oscillation parameters shall be quoted. For monochromatic instruments: the type of monochromator, its crystal and reflection used, the type of detector, the distance from the monochromator to the reference point; the resolution of the detector. For TOF instrument: the total flight path, the type of detector, the angular range of the detector; the time resolution or channel width; the incident intensity as a function of the wavelength. For both: the wavelength/ wavelength range and how it was determined; the vertical and horizontal gauge intensity profile (if critical to the measurement), the distance form the detector to the reference point	Neutron source & instrument name Monochromator type *hkl* used incident wavelength (or range) … Beam optics type Optics focal length and aperture dimension (GV size) … Sample manipulator & accuracy Instrument coordinate system …
Calibration	Instrument calibration: detector angular response and neutron flightpath (unique for TOF instruments) must be calibrated. Instrument alignment: axis of the primary and secondary beam optics must be aligned to the center of instrument (ω-rotation axis) with min. accuracy of 10% of the	Standard d-spacing vs 2θ or TOF plot Primary optics alignment Secondary optics alignment Reference point vs ω-center of rotation

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	GV width. GV characterization: GV width and intensity profile shall be characterized.	Beam width Beam intensity …
Detector	Position sensitive detector (PSD), variations of scintillator material, dimension, resolution, angular coverage.	Detector type Detector distance to sample Area Pixel size …
Raw data output	Neutron counts as a function of wavelength (monochromatic instrument) or time-of-flight (TOF instrument) at each measurement points (described by coordinates and angles) Maximum counts are observed on the specific wavelengths or time-of-flights where the Bragg's Law is satisfied.	Total count Counting time Measurement position (e.g., x, y, z, rotation) Measurement direction (e.g., ω, X, ψ) …
Raw data analysis (data reduction)	Integration of detector reading, followed by peak fitting. Bragg's Law is applied to translate fitted peak position to lattice spacing. Single peak fitting using Gaussian or Voigt function provides lattice spacing of particular *hkl*. Full-pattern (multiple peak) refinement using Rietveld or Pawley refinement provides lattice parameter.	Fitting function Background function …
Data analysis	Elastic strains are calculated by the change in lattice spacing/parameter of the sampled specimen and stress-free reference sample. Stresses are calculated from these strains. Normal stresses at a point can be determined from strain components along three mutually orthogonal co-ordinate axes. Commonly the co-ordinate axes are assumed to be coincident with the principal directions of deformation, thus theses tresses are the principal stresses. When principal directions of the stress state are not known, determination of strain in at least six independent orientations is generally needed. Diffraction elastic constants are required to calculate the stresses, and diffraction elastic constant associated with each hkl should be used.	d_0 reference value(s) Diffraction elastic constants …
Post processing	Measurements of stresses at multiple points can be interpolated to produce a map	Interpolation function …
Property	Result of the measurements	Lattice spacing/parameter Strains Stresses …

A similar CHADA workflow, albeit more generic, has been proposed for synchrotron X-ray diffraction, Figure 3. While it is mostly comparable to that of neutron diffraction, note that additional levels have been added to the beginning and the end of the workflow. The so-called "Level 0" comprise of the definition of measurement problem and expected result to determine the most efficient techniques (conventional θ/2θ scanning, 2D transmission, and white beam). "Level 6" concerns with the reporting of the results. The chart is likely to be expanded in the future, with separate detailed workflows for each synchrotron X-ray diffraction techniques.

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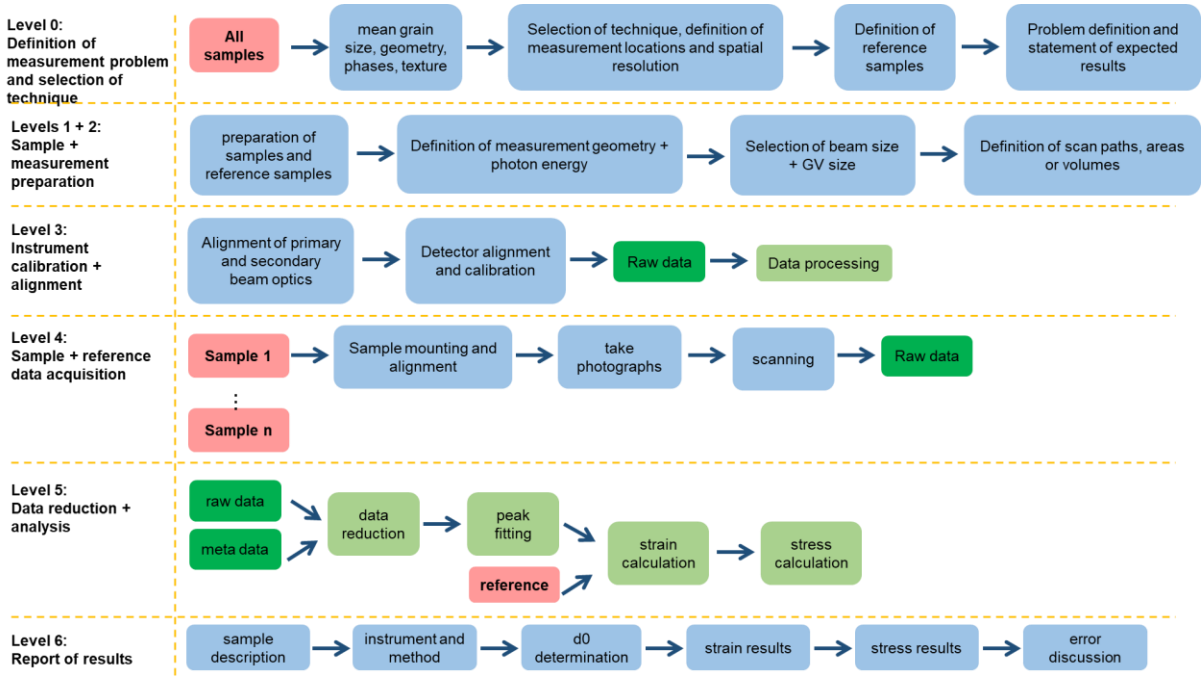

Figure 3. CHADA workflow for residual stress measurement by synchrotron X-ray diffraction

Harmonization of methods at the LRIs

Based on the exercises above, it can be clearly seen that there are plenty of resemblance in the workflow of neutron diffraction and synchrotron X-ray diffraction. Consequently, most of the metadata from both methods can also be categorised under the same keywords based on CHADA elements. Therefore, a common framework for residual stress measurement at LRIs can be defined in terms of CHADA workflow and compiled metadata descriptors as follows.

The workflows are divided into five levels, with an addition of a preliminary level:

i) **Level 0 – Definition of measurement problem and selection of probe.** In this preliminary stage, properties of the sample, especially ones that directly affects the measurement (e.g., geometry, materials, crystal structure, grain size, texture, second phases, etc.) are detailed. Definition of problems and statement of expected results (e.g., expected stress profile, resolution) are provided. The methods of getting the d_0 reference values are also defined. From these information, selection is made of the most suitable probe (neutron or synchrotron X-ray) and technique (monochromatic or TOF for neutron; conventional θ/2θ scanning, 2D transmission, or white beam for synchrotron X-ray).

ii) **Level 1 – Sample & measurement preparation.** Based on the sample properties and definition of problems, design of experiment (e.g., selection of measurement direction and GV size) is performed, which then determines the instrument settings (e.g., incident beam energy/wavelength, beam optics type and dimensions, detector angular position).

iii) **Level 2 – Instrument calibration and alignment.** To verify the instrument setup, a series of calibration and instrument alignment activities are performed (see NQL for neutron methods). Raw data are produced and recorded for metadata and input for further analysis.

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iv) **Level 3 – Sample & d_0-reference data acquisition.** Sample(s) and references are mounted on the instruments. Scans are performed according to the scan strategy. Raw data are produced.

v) **Level 4 – Data reduction, analysis, and post-processing.** Data reduction including fitting the diffraction spectrum with appropriate model, analysis including strain and stress calculation of one or multiphases and with/without external applied conditions (i.e., load, temperature, etc.), post-processing including interpolation of data points and reconstruction of strain and/or stress map.

vi) **Level 5 – Reporting of results.** Comprehensive reporting, including sample and problem description, methods, instrument setup, results, and discussions. For neutron diffraction method, this reporting can refer to the NQL for residual stress determination with neutron diffraction (Ramadhan & Cabeza, 2020).

The visual representation of the workflows described above are provided in Figure 4. Table 2 provides the format of common metadata descriptors for residual stress measurements at the LRIs.

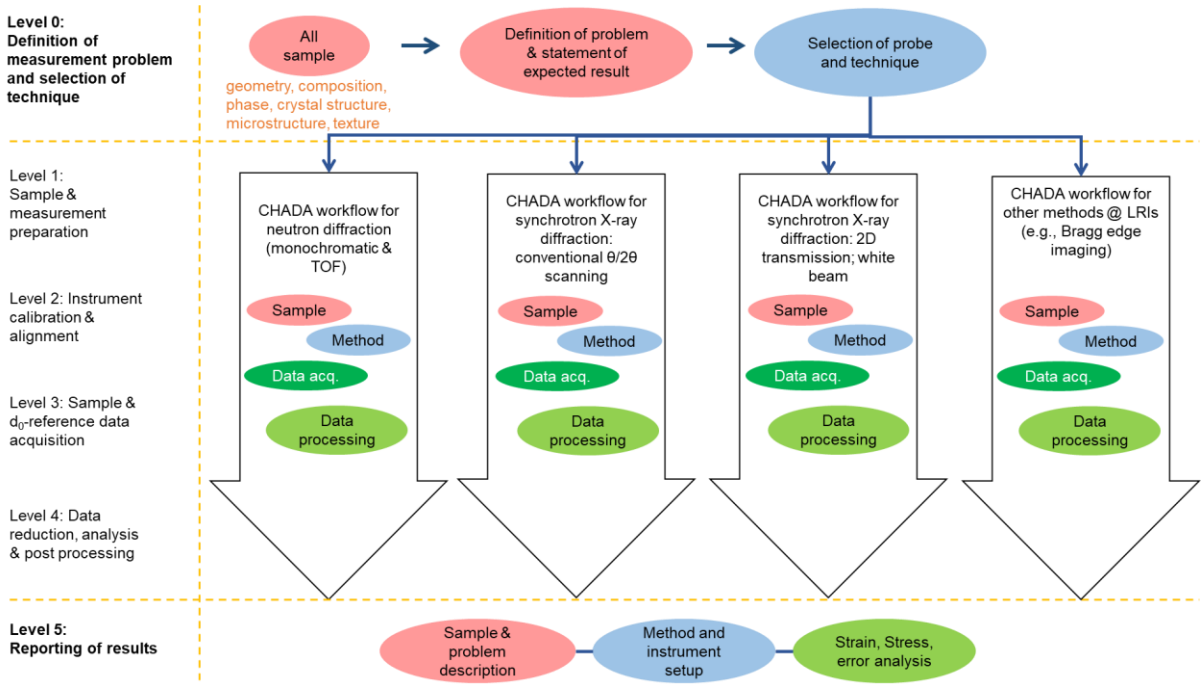

Figure 4. Visual representation of the common framework for residual stress measurement technique at LRIs

Table 2. Compiled metadata descriptors in CHADA format for residual stress measurement techniques at LRIs. The tables are not final nor comprehensive, and only presented for demonstration purposes. Changes are envisioned in the future, with details to be added especially for certain techniques that are under development.

Keyword	Description	List of metadata – Neutron Diffraction (monochromatic)	List of metadata – Synchrotron X-ray: 2D transmission	List of metadata – other methods
User case	Specimen; diagram of specimen showing dimensions, fiducial marks or reference locations and specimen co-ordinates; diffraction condition	Sample name, geometry, references, co-ordinate, material, composition & phase, grain size, possible texture, *hkl*, Young's modulus, other specimen details… measured strain component, GV dimension, …		

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Reference sample	Stress-free or reference lattice spacing. Can be measurement in a material at a position known to contain a negligible stress, measurement on a powder, which is representative of the material being examined, measurement on a small coupon, cut from a large block of material, or other approaches (including possible extra investigation using other techniques).	Description of d_0 measurement (e.g., small cut of sample, dimension 5 mm × 5 mm × 5 mm; metal powder, 100 µm particle size)		
Calibration sample	For calibration: standard sample with known lattice parameter, ideal diffraction pattern For alignment: standard sample with appropriate and known geometries	Calibration sample details (e.g., standard FeCu powder, manufacturer) Alignment sample details (e.g., Pin Ø 1mm)	…	…
Medium	Mostly air, can be on load-rig for in-situ loading, can be inside a furnace in vacuum or filled with inert gas	Temperature, pressure, load, …		
User (operator)	Human operator (individuals responsible for instrument, user), experiment details	Instrument responsible details, username details, begin & end date of experiment, …		
Sample/probe physics of interaction	Measurement of lattice spacing inside bulk material, by illuminating a defined volume of material (GV) with the probe (neutron/ x-ray) and measuring the angular/ temporal position of a specific sets of constructive interference of the probe scattered by the corresponding crystal lattices according to Bragg's Law.	Brief details regarding the specific technique		
Equipment setup	Details of the probe source and incident beam, beam defining optics, sample manipulator details	Neutron source & instrument name, monochromator type, *hkl* used incident wavelength (or range), … Beam optics type, optics focal length and aperture dimension (GV size), … Sample manipulator & accuracy, instrument coordinate system, …	Synchrotron source & instrument name, energy range, … Beam optics type, aperture dimension (GV size), … Sample manipulator & accuracy, instrument coordinate system, …	…
Calibration	Instrument calibration and alignment result	Standard d-spacing vs 2θ or TOF plot, primary & secondary optics alignment, reference point vs ω-center of rotation, beam width & intensity, …	…	…
Detector	Details of the probe detector	Detector type, distance to sample, area, pixel size, …	2D detector type, area, distance to sample, pixel size, noise, …	…
Raw data output	Detected signal at each measurement point	Total count, counting time, measurement	Total count, counting time, measurement	…

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		position (e.g., x, y, z, rotation), measurement direction (e.g., ω, X, ψ) …	position (e.g., x, y, z, rotation), …	
Raw data analysis (data reduction)	Steps necessary to translate detector reading into fit-able spectra, fitting of signal for accurate positional determination of the diffraction features (e.g., Bragg peak, Bragg edge), data filtering (removal of the outliers or non-relevant points) conversion to lattice spacings using Bragg's Law	Fitting function, background function, …	Integration geometry, information on the method of data reduction, fitting function, background function, data filtering criteria …	…
Data analysis	Elastic strains are calculated by the change in lattice spacing/parameter of the sampled specimen and stress-free reference sample. Stresses are calculated from multiple strain components, according to certain assumption. Appropriate diffraction elastic constant associated with each hkl should be used.	d_0 reference value(s), diffraction elastic constants, …	d_0 reference value(s), method ($\sin^2 \psi$, normal component …), diffraction elastic constants, micro-mechanical model …	…
Post processing	Representation of the measured parameters for better readability	Interpolation function to produce a map from multiple measured points, …	…	…
Property	Result of the measurements	Lattice spacing/parameter, strains, stresses, uncertainties		

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