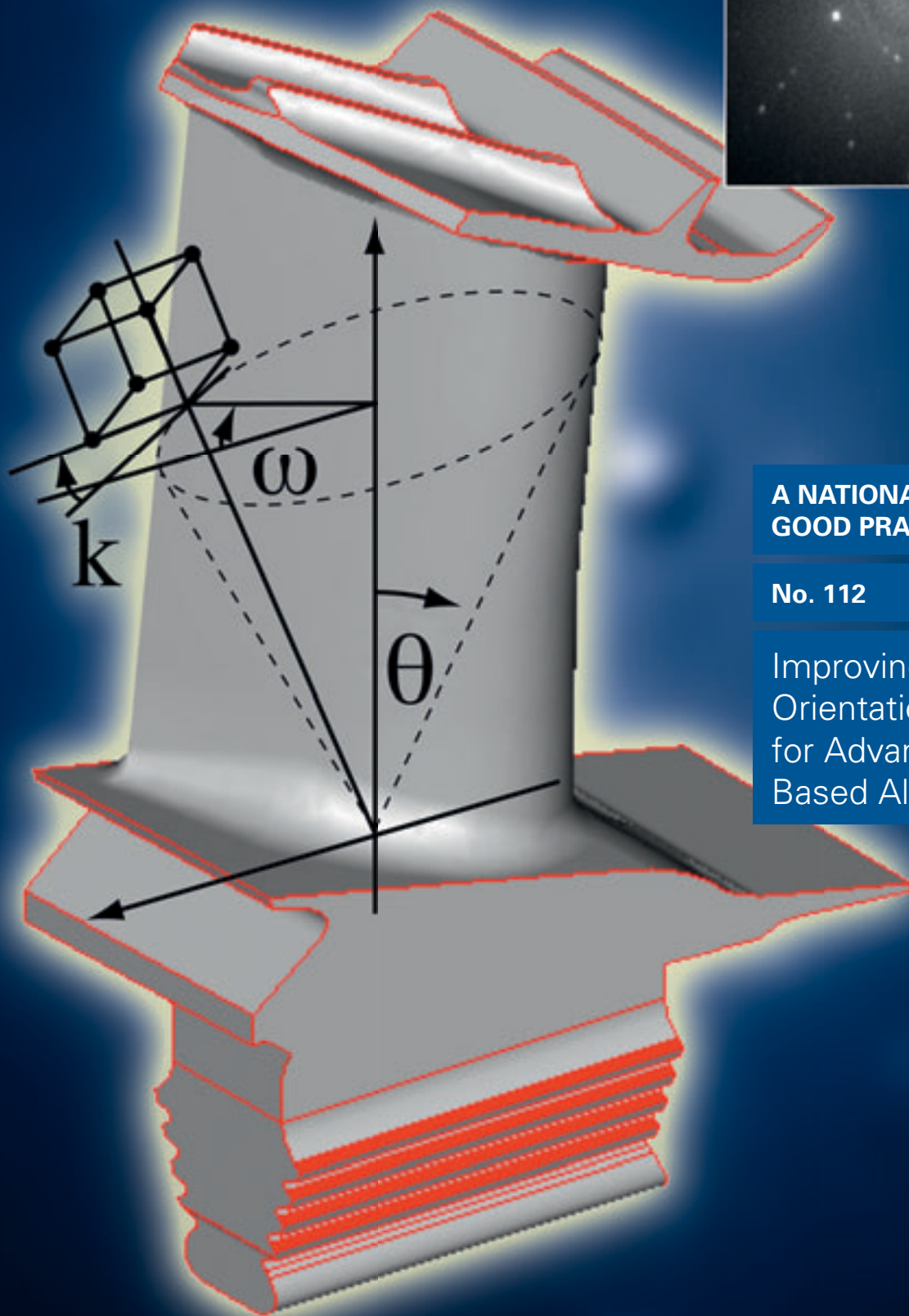
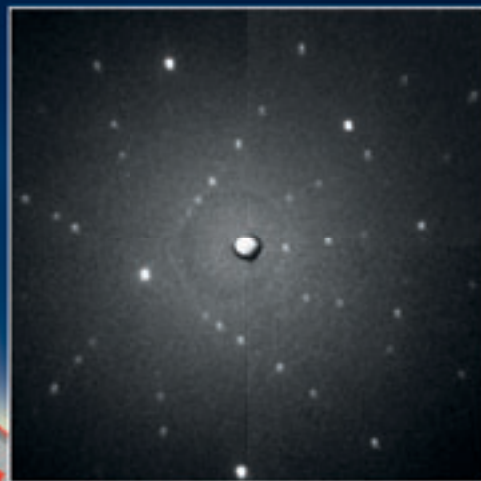




**A NATIONAL MEASUREMENT
GOOD PRACTICE GUIDE**

No. 112

Improving Single-Crystal
Orientation Determination
for Advanced Nickel-
Based Alloys

NPL Measurement Good Practice Guide No. 112

Improving Single-Crystal Orientation Determination For Advanced Nickel-Based Alloys

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Abstract:

This Good Practice Guide describes the determination of single-crystal orientation measurements for single-crystal nickel-base castings by back and side-reflection X-ray diffraction (Laue). These measurements check the quality of the castings against specifications set by the OEMs for the foundries.

For quality control three parameters are determined:

1. The angle between a reference direction in the component, defining the preferred direction of solidification, and the closest $\langle 100 \rangle$ direction. (θ).
2. Less commonly, the angle between a second reference direction in the plane perpendicular to the original reference direction (α) and/or a $\langle 001 \rangle$ direction (κ).
3. For single crystals with more than one grain a measurement of the misorientation or disorientation between adjacent grains (R).

The guide contains information about the definition of orientation parameters; principles of back-reflection and side-reflection Laue systems; calibration standards; system alignment; calibration; validation and uncertainty of measurements with a final section on making the measurements.

The definition of primary angles is based upon European practice but a comparison with two definition conventions used in the USA is included.

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Improving Single-Crystal Orientation Determination

Contents

Executive summary

1.	Background	1
1.1	The need for single-crystal orientation measurement.....	1
1.2	Selection of measurement method appropriate to industry requirements	2
2.	Definition of orientation parameters	5
2.1	Primary orientation of main grains.....	5
2.1.1	Introduction	5
2.1.2	Directly measured primary orientation angles (European convention)	6
2.1.3	Viewing conventions and consequences of differences	7
2.1.4	Derived primary orientation angles (European Convention)	8
2.1.5	Calculation of derived primary orientation angles (European convention).....	11
2.1.6	Primary orientation angles (US Convention).	13
2.1.7	Other derived primary orientation angles.....	14
2.2	Grain boundary R-values.....	15
2.2.1	Introduction	15
2.2.2	Two angle, REL, (R-2cos) R-value definition	15
2.2.3	Single-angle (R-1cos) R-value definition.	16
3.	Principles of the back-reflection Laue (BRL) method	17
3.1	Basic principles	17
3.2	Practical points with the BRL method and single-crystal orientation measurement.....	18
4.	Industrial variants of the back-reflection Laue system.....	20
4.1	Image collection	20
4.2	System geometry	20
4.2.1	Introduction	20
4.2.2	Critical alignment considerations for side-reflection systems	22
4.3	Pattern analysis protocol	24
4.3.1	General information	24
4.3.2	Charting methods	24
4.3.3	Overlay mask matching.....	25
4.3.4	Marking of individual diffraction spots	27
4.3.5	Difficult diffraction patterns	27
4.3.6	Brief comparison of computer aided methods	28

5.	Calibration standards.....	29
5.1	Nomenclature	29
5.2	Use.....	29
5.3	Materials.....	29
5.4	Recommended shape.....	29
5.5	Recommended alignment	29
5.6	Identification	30
5.7	Traceability.....	30
5.8	Care of standard.....	30
5.9	Safety mounting	30
6.	System alignment guidance.....	31
6.1	Introduction	31
6.2	Key features of the Laue pattern of a silicon single-crystal standard	32
6.3	X-ray beam alignment	32
6.3.1	True back-reflection systems.....	33
6.3.2	Side-reflection systems	33
6.4	Detector centring (alignment of DPN) and rotation (detector alignment).....	33
6.4.1	Systems with fixed specimen axis, SA	34
6.4.2	Systems with fixed detector axis, DPN.....	34
6.5	Co-planar alignment of all critical system axes	35
6.5.1	Assessment of SSCS diffraction pattern symmetry	35
6.5.2	Adjusting system coplanar alignments.....	35
6.5.3	System alignment verification.....	37
6.6	Final alignment verification, side-reflection geometry systems only	37
7.	System calibration guidance	39
7.1	Purpose of system calibration.....	39
7.2	Calibration.....	39
7.3	Validation	40
8.	Evaluation and statement of system's measurement capability.....	42
8.1	Introduction	42
8.2	Conducting an assessment.....	42
8.3	Orientation parameter evaluation data sets	42
8.3.1	Specimens	42
8.3.2	Measurements	43
8.4	System measurement consistency statement	44
8.4.1	Consistency assessment: repeatability (and comparing with other facilities).....	44
8.4.2	Consistency assessment: reproducibility (and comparing with other facilities).....	45
8.4.3	Establishing expected precision statistics – repeatability and reproducibility - (for specimens and parameters).....	46
8.5	Independent consistency assessment (general guidelines)	47

8.5.1	Precision (or uncertainty) statement.....	47
8.5.2	Bias (deviation) statement.....	47
9.	Practical limitations on system measurement capability (uncertainty).....	48
9.1	Uncertainty statement.....	48
9.2	Measurement uncertainty limitations: collaborative assessment	48
9.3	Measurement uncertainty: sensitivity studies.....	49
9.4	Customer-defined measurement uncertainty limitations.....	50
10.	Guidance on control of a measurement system.....	51
10.1	Methods	51
10.2	Fixtures.....	52
10.2.1	Measurement system	52
10.2.2	Component fixtures.....	52
10.3	Operators	52
11.	Guidance on comparison of systems.....	54
12.	Completing measurements.....	56
Appendix 1: Definition of primary angles (European convention).....		59
Appendix 2: Definition of primary angles (US convention).....		60
Appendix 3: Definition of angle omega (additional derived primary orientation angle, European convention)		61
Appendix 4: Details of R-value conventions		63
A4.1	Different R-value definitions.....	63
A4.2	Two-angle, REL, (R-2cos) R-value definition	63
A4.2.1	Formal definition.....	63
A4.1.2	Calculation method	64
A4.2	Single-angle (R-1cos) R-value definition.....	69
A4.2.1	Formal definition.....	69
A4.2.2	Calculation method summary.....	69
Appendix 5: Assessment of SSCS diffraction pattern symmetry		70
A5.1	Horizontal axis asymmetry.....	70
A5.2	Vertical axis asymmetry	70
A5.2	Practical considerations.....	72
Appendix 6: Details of system validation parameters.....		73
A6.1	Examples of validation specimen data sets	73
A6.1.1	Primary orientation values	73
A6.1.2	R-values.....	74

A6.2	Multiple facility comparison (data pooling).....	75
A6.3	Nominated master facility (datum system).....	76
A6.3.1	Measurement system precision (or uncertainty).....	76
A6.3.2	Measurement system bias.....	76
Bibliography.....		77

Executive summary

A critical element in the quality assurance of single-crystal alloy engine components is the crystallographic orientation, which should be within defined limits in order to assure a certain level of performance, notably creep resistance. Normally, orientation characterisation is made using the backscattered Laue X-ray diffraction method. The procedure, which initially was made using a wet X-ray film method, has over the years been superseded by progressively quicker and more efficient methods, including the use of dry Polaroid film, scintillation detectors, and most recently CCD detector systems. Analysis methods, once visual/manual, have been semi-automated.

There are two critical areas:

- (1) The determination not only of the general deviation of the crystallographic axis from the components reference direction or axis, but also the rotation of the crystal about that axis, which is assuming increasing importance in critical design.
- (2) The determination of the orientation difference between grains in crystals which by chance have two grains or more, and comparison of those differences with a maximum acceptable level.

This guide has been initiated because of concerns about the reliability of the orientation measurements made in different establishments, perhaps using different equipment or different ways of analysing the Laue pattern. It has been compiled with the assistance of experts in the industry. Subjects include:

- (1) Definition of terms and angles, including angular relationships;
- (2) Good practice in set up and calibration of X-ray systems;
- (3) Identification of X-ray patterns and their measurement;
- (4) Differences between European and US conventions for angles (with a focus on the UK/European convention);
- (5) Assessment of uncertainties of measurement, supported by an interlaboratory exercise.

This guide is not intended for operators, but more for those in control of industrial quality assurance activities and who need to interpret the data obtained from quality assurance measurements.

SAFETY NOTE:

Operation of X-ray equipment may have safety implications. This Good Practice Guide does not purport to address all of the health and safety issues associated with its use. It is the responsibility of the user of this Guide to comply with local Health and Safety Law/rules when adopting this document. Neither the authors of this Guide nor the National Physical Laboratory take any responsibility for inappropriate exposure resulting from procedures recommended in this Guide.

1. Background

1.1 The need for single-crystal orientation measurement

Cast single-crystal (SX)¹ nickel superalloy components were first introduced in gas turbine aero-engines during the early 1980s as high pressure turbine blades. In this application, the anisotropic control of the crystal orientation enhanced the creep endurance (stress rupture response) of these components, enabling their service operating temperatures (based upon an engine's turbine entry temperature, TET) to be raised by over 50°C to approximately 1600°C. This represented significant performance and fuel saving benefits, which have been further enhanced by both alloy development and application of SX in lower stage turbine blades. During the past decade, exploitation of both the alloy's refractory benefits and different aspects of anisotropic property control have extended the use of these SX castings to stators (nozzle guide vanes, NGVs), as well as heat shields and other structural components in aero-engines and power generation turbines.

For these high integrity applications, knowledge of the orientation of each SX component is an engineering control requirement, necessitating orientation measurement(s) on every casting. Generally, for rotating components (blades or rotors), restrictions are imposed on the alignment of the $\langle 001 \rangle$ crystal direction relative to a significant component direction, or the alignment of the $\{001\}$ crystal plane relative to a defined component reference plane. With static components (NGVs, stators and structural members) different formats for crystal alignment control also exist, *e.g.* specification of the alignment of the $\langle 111 \rangle$ crystal direction relative to a significant component direction.

Theoretically, the desired and enhanced creep (or other property) performance is only achieved in a 'single grain' casting, *i.e.* one in which there are no grain boundaries. However, casting process capability, especially for larger and more complex shaped components, very rarely generates a boundary-free component. Thus the turbine engine manufacturers have evolved component-specific quality acceptance standards for allowable grain boundaries. These quality standards define limits on a grain boundary's location, on grain size / shape, and on so-called R-value, which is an assessment of the difference between the orientations of grains on either side of a boundary.

For a detailed chronological article on the development of back scattered diffraction technique for measurement of orientation and misorientation of single-crystal turbine blades in the period from their inception to about the year 2000, the reader is referred to Jones [1].

¹ Strictly they are multiphase coherent aligned dendritic microstructures that exhibit anisotropic property variation equivalent to those of a 'conventional' single-crystal.

1.2 Selection of measurement method appropriate to industry requirements

Both the casting method and quality control require that every single-crystal component is treated as a batch of one, which as a minimum requires an assessment and measurement of its (primary) orientation.

The requirements for a primary orientation measurement method were first specified 30 years ago. They are:

- (i) The measurement method shall be rapid. (a measurement cycle time 5 minutes or less);
- (ii) The measurement process shall be accurate (capable of angle measurement resolution of 1° or less);
- (iii) Operators need not have ‘detailed crystallographic knowledge’;
- (iv) The measurement system shall be able to handle different component types and sizes;
- (v) The measurement equipment shall be robust;
- (vi) The measurement equipment shall be safe, conforming to local health and safety rules;
- (vii) The measurement system shall be capable of collecting orientation data for R-value grain boundary assessment.

Capital cost of equipment and running costs are also important considerations.

The candidate measurement technology options that have been assessed are summarised in Table 1.1. These potential methods are reviewed periodically by the industry, and to date none have emerged as a viable alternative to the initially selected measurement technology, the X-ray diffraction Back-Reflection Laue Method (BRL method).

The BRL method was preferred as it provided discrete non-topographical orientation information at spatial and angular resolutions appropriate to the nickel superalloy microstructure. Further, it is a static and non-destructive measurement which is suitable for development into a rapid and flexible production assessment technology.

However, it is a **surface only, single-point** (approximate spatial resolution 2 mm diameter with a depth resolution 100 µm) **measurement method**, thus all measurements are assumed to be representative of orientation either of a nominated region or of the whole of the casting. The BRL method can therefore be only one element integrated into overall inspection processes, and allowances have to be made for this assumption.

Figure 1.1 shows a schematic of the quality assurance steps that are typically taken on a single-crystal component.

Table 1.1: Summary review of single-crystal orientation measurement technology options (for use with cast nickel turbine components)

Measurement technology	Principles	Comments
X-RAY DIFFRACTION (XRD) Back-Reflection Laue	Uses white X-radiation: known symmetry diffraction pattern with pattern position only dependent on crystal orientation.	Surface measurement. Static arrangement. Minimal production component preparation. (Lateral resolution 2 mm diameter; depth resolution 100 μm .)
X-RAY DIFFRACTION (XRD) Rocking curves	Uses monochromatic X-radiation; oscillation of crystal to attain diffraction peak overlap with reference crystal.	Surface or transmission measurement. Very high angular sensitivity. Movement of specimen fundamental to the method.
ELECTRON BACKSCATTER DIFFRACTION (EBSD)	Imaging of backscatter electrons to give Kikuchi lines which are indexed for crystallographic information.	Normally performed in a modified Scanning Electron Microscope. Analysis occurs in evacuated chamber. Chamber size can limit the size of casting. Requires good surface preparation. Lateral resolution better than 100 nm and depth resolution about 50 nm.
ULTRASONICS	Change in orientation alters velocity of sound wave.	Other physical properties alter velocity. Difficult to discriminate. Transmission or surface methods possible.
OPTICAL REFLECTIVITY	Intensity of reflected beam of light dependent on crystal orientation (due to the dendritic macrostructure of nickel superalloys).	Requires etched surface for optimum response. Surface only method. Intensity changes and very dependent upon surface condition, so complex to quantify. (Method used to locate grain boundaries).

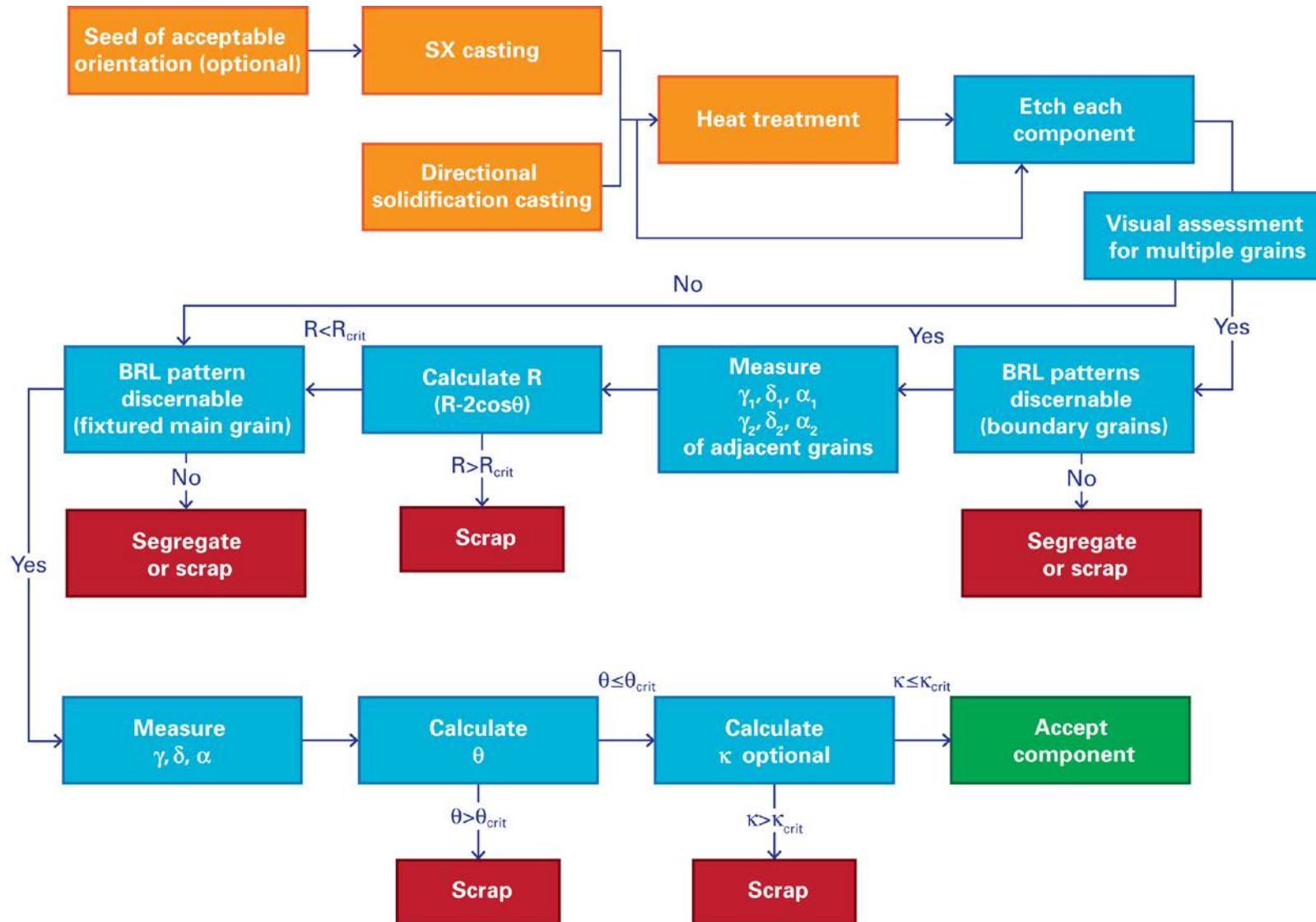


Figure 1.1: Flow diagram for quality assurance of single-crystal orientation.

2. Definition of orientation parameters

2.1 Primary orientation of main grains

2.1.1 Introduction

The term ‘primary orientation’ covers the systematic group of angles used unambiguously to define and describe the orientation of the single-crystal (or grain), relative to a reference system. For turbine components, the reference system comprises a reference plane, RP, and reference direction, RD, lying within the reference plane; see Figure 2.1.

Nickel superalloys have a face centred cubic (FCC) dominant crystal structure. As FCC is a highly symmetric crystal system, it can be represented solely by crystal direction types $\langle 001 \rangle$, $\langle 011 \rangle$ and $\langle 111 \rangle$, and crystal plane types $\{001\}$, $\{011\}$ and $\{111\}$. Primary orientation describes the alignment of the crystal $\langle 001 \rangle$ direction closest to the component reference direction, RD, and the rotation of the crystal’s $\{001\}$ plane parallel with this closest $\langle 001 \rangle$ direction, from the component reference plane, RP.

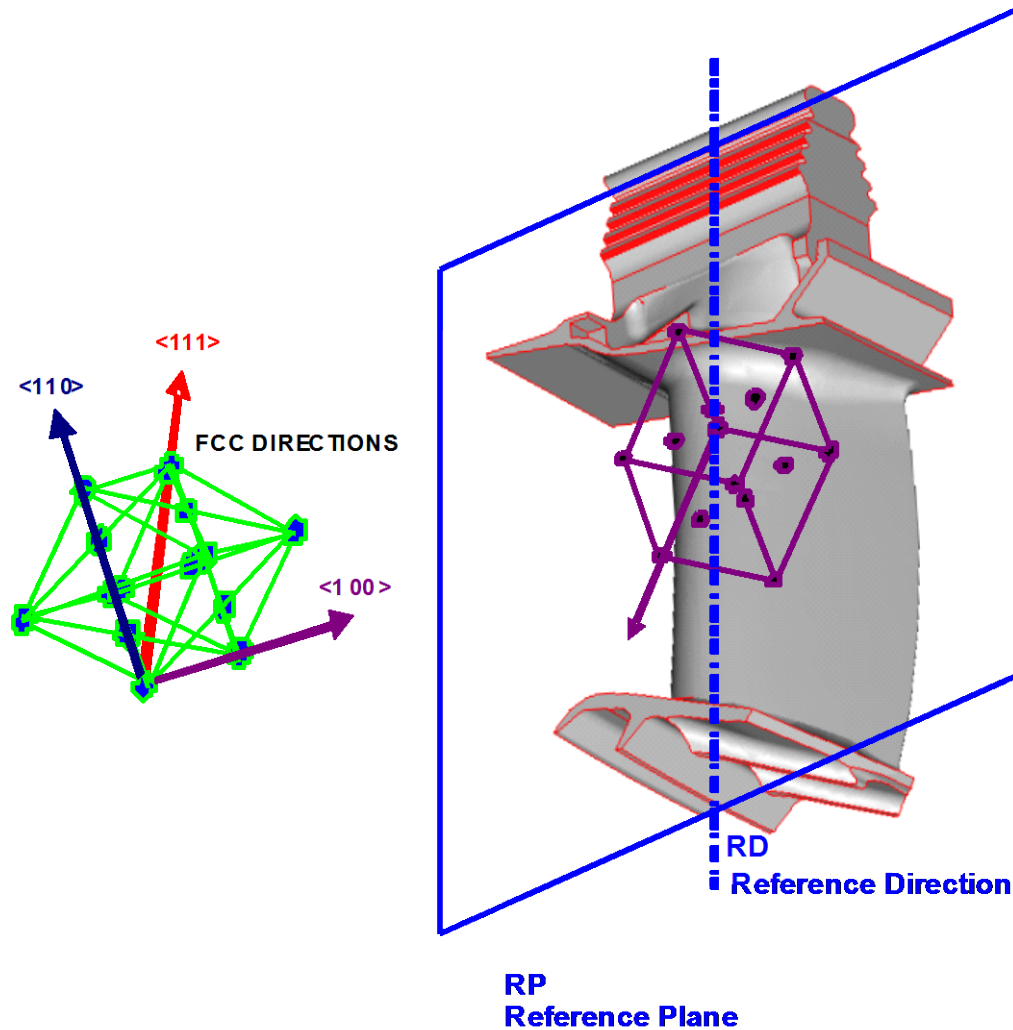


Figure 2.1 Component reference system (reference direction and reference plane).

2.1.2 Directly measured primary orientation angles (European convention)

Historically, the starting position for primary orientation definition was the analysis of a back-reflection Laue (BRL) film, as described in classical diffraction texts, *e.g.* Cullity [2], and standards *e.g.* ASTM E82-91 [3]². The classical (wet film) symmetrical BRL method generates a pattern, shown schematically in Figure 2.2. (The BRL method is described more fully in section 3.1).

Figure 2.2 is a typical of a diffraction pattern from a crystal aligned with one of its $\langle 001 \rangle$ directions inclined close to the measurement reference direction, RD, the film / image centre. The zone representative a crystal's $\{001\}$ plane is also identified. The component reference plane, RP, is denoted by the horizontal of the film. *It is assumed that the wet film, a transparent image, is viewed looking **towards** the component in the experimental alignment.* Some primary orientation angles can be measured directly on a pattern using a Greninger chart and basic knowledge of crystal symmetry [2, 3]. These are the angles gamma, γ , delta, δ , and alpha, α , as shown in Figure 2.3, where directions for positive rotations are indicated (starting at RP, alpha is measured to the first $\{001\}$ zone encountered in the clockwise direction).

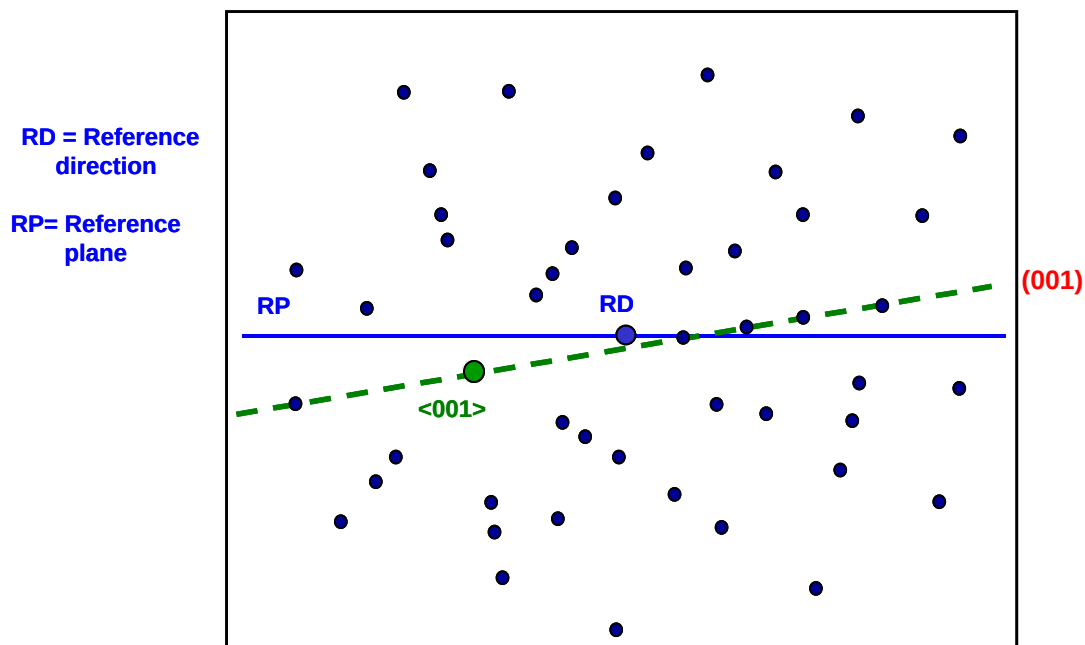


Figure 2.2: Schematic of a classical (wet film) back-reflection Laue (BRL) pattern.

² Two further standards exist for this technique, but are specifically focused on silicon technology:

ASTM F847-02 “Standard test methods for measuring crystallographic orientation of flats on single-crystal silicon wafers by X-ray techniques”. Withdrawn 2003, transferred to SEMI (www.semi.org) as SEMI MF847-0705.

ASTM F26-87a (1999) “Standard test methods for determining the orientation of a semiconductive single crystal”. Withdrawn 2003, transferred to SEMI (www.semi.org) as SEMI MF26-0305.

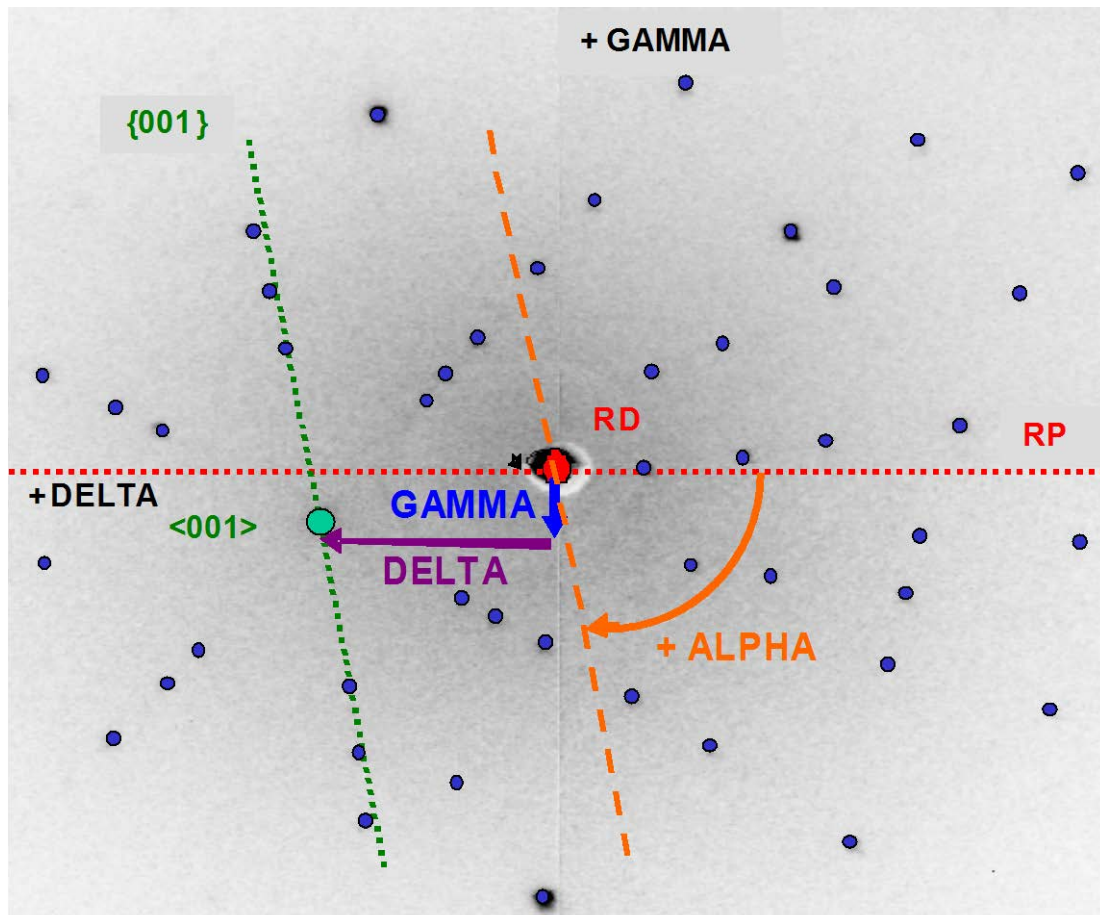


Figure 2.3: Directly measured angles on a BRL diffraction pattern – European convention (crystal $\langle 001 \rangle$ orientated close to the reference direction).

2.1.3 Viewing conventions and consequences of differences

The definitions for primary orientation angles apply when viewing the wet film looking towards the component [2]; this is termed the ‘classical viewing convention’. An alternate film viewing convention would be to observe the Laue diffraction pattern on a film when looking back at the film from the specimen. This is the case when instant ‘Polaroid’ film substitutes for the wet film. These different viewing options are illustrated in Figure 2.4. If the same film (same crystal alignment) is viewed from both of these alternative positions, the *observed* positions of the diffraction pattern are different, Figure 2.4. So, to obtain the same values for directly measured primary orientation angles, adjustments will need to be made to the sense (direction of rotations) for the directly measured angles, gamma, delta and alpha. However, if such adjustments (corrections) are not made within the alternate view (and the sense / directions for rotations as classically defined are applied), different numerical values for these directly measured angles result.

Even though viewing convention differences may appear to be a theoretical point, it is nonetheless one of crucial importance to the materials engineer in the single-crystal turbine industry, because both the classical viewing and the partially adjusted, alternate viewing conventions are used to specify primary orientation. To make a clear distinction, these are

often referred to respectively as the EUROPEAN CONVENTION (classical viewing) and US CONVENTION (alternate viewing).

In sections 2.1.4 and 2.1.5 of this guide full details of primary orientation parameters using the European convention are provided. Details of the differences between the conventions are then described in section 2.1.6.

Note that real-time Laue systems generally display images as though viewing a Polaroid film but calculate the angles to simulate the selected convention.

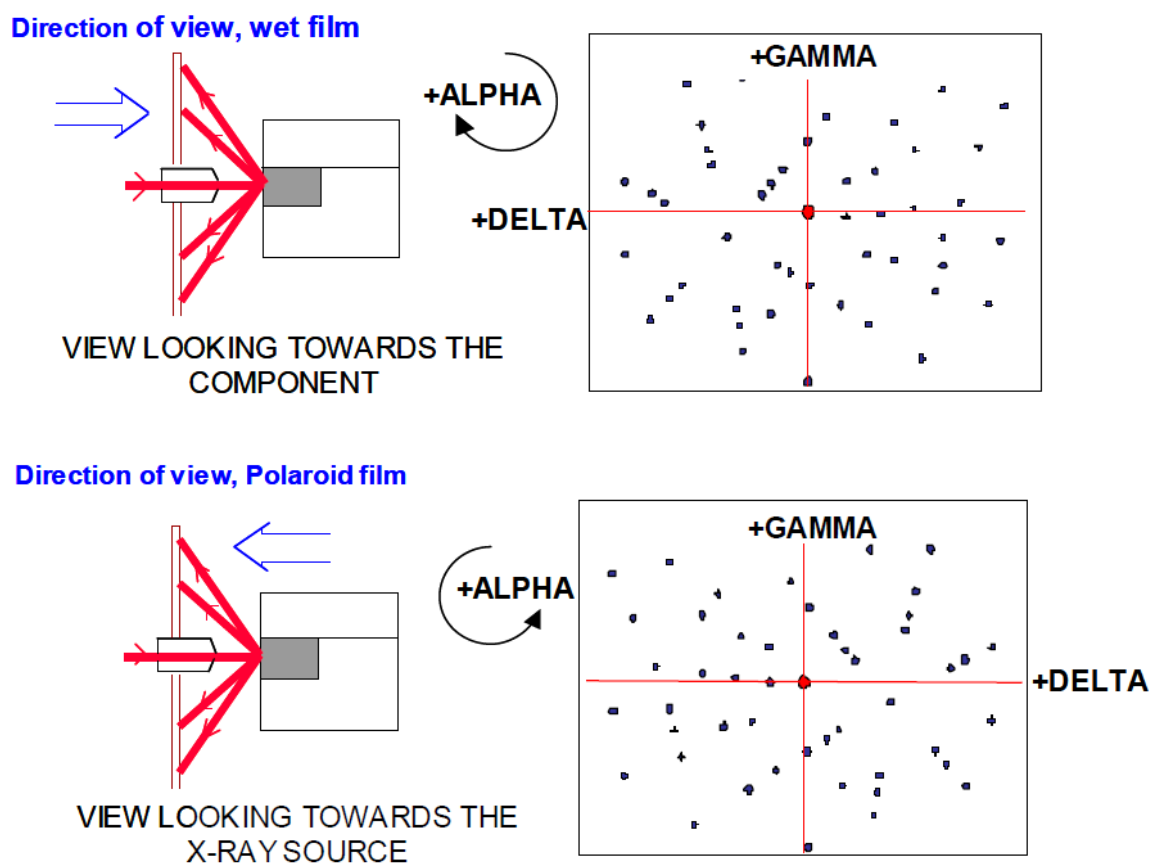


Figure 2.4: BRL diffraction film viewing options.

2.1.4 Derived primary orientation angles (European Convention)

For any BRL diffraction pattern (especially non-001 patterns), the Greninger chart can be used to collect systematic data from the pattern, which can be used to construct a universal crystal angle orientation diagram, the Stereographic Projection, Figure 2.5 (see [2], pp.70 -86 for detail of construction and format of a stereographic projection, and [2], pp.471 - 487 for method of constructing a stereographic projection from directly measured BRL information). On such a stereographic projection, the crystal $\langle 001 \rangle$ directions and $\{001\}$ planes are

displayed along with the measurement references, RD and RP. The formal written definitions of all six primary angles are based on their measurement on a stereographic projection: these are detailed in Appendix 1.

Six angles are used in the turbine industry, the additional three parameters helping to relate the crystal orientation to either key component geometry features or to anisotropic variation of a material property within the crystal's FCC unit cell. A visual interpretation of the orientation angles is shown in Figure 2.6. The additional three angles are:

- (i) Theta, θ : the mean deviation, regardless of direction, of the nearest crystal $\langle 001 \rangle$ from the component reference direction, RD.
- (ii) Kappa, κ : a rotation, equivalent to alpha but with its rotation axis being a $\langle 001 \rangle$ crystal direction.
- (iii) Rho, ρ : a statement of the position of the component reference direction, RD, within the FCC unit cell.

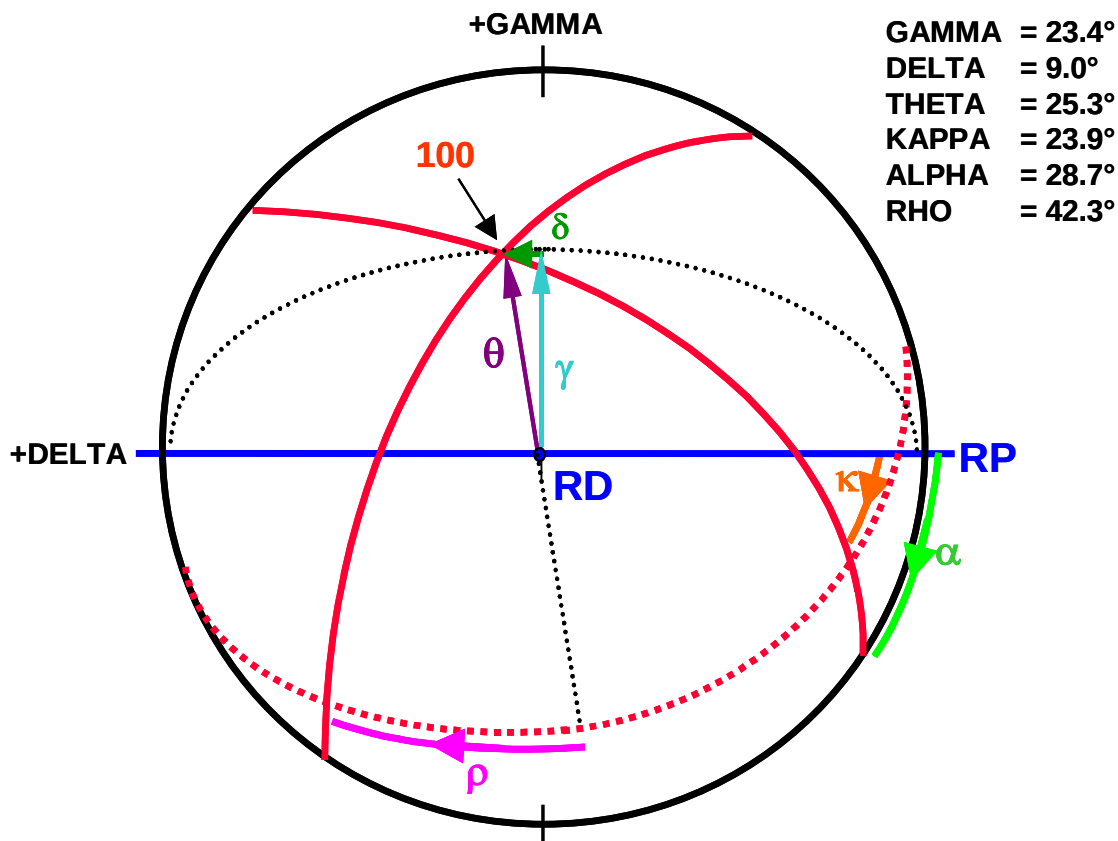


Figure 2.5: Stereographic projection illustration of crystal orientation, denoting European convention primary orientation angles as viewed on a wet film.

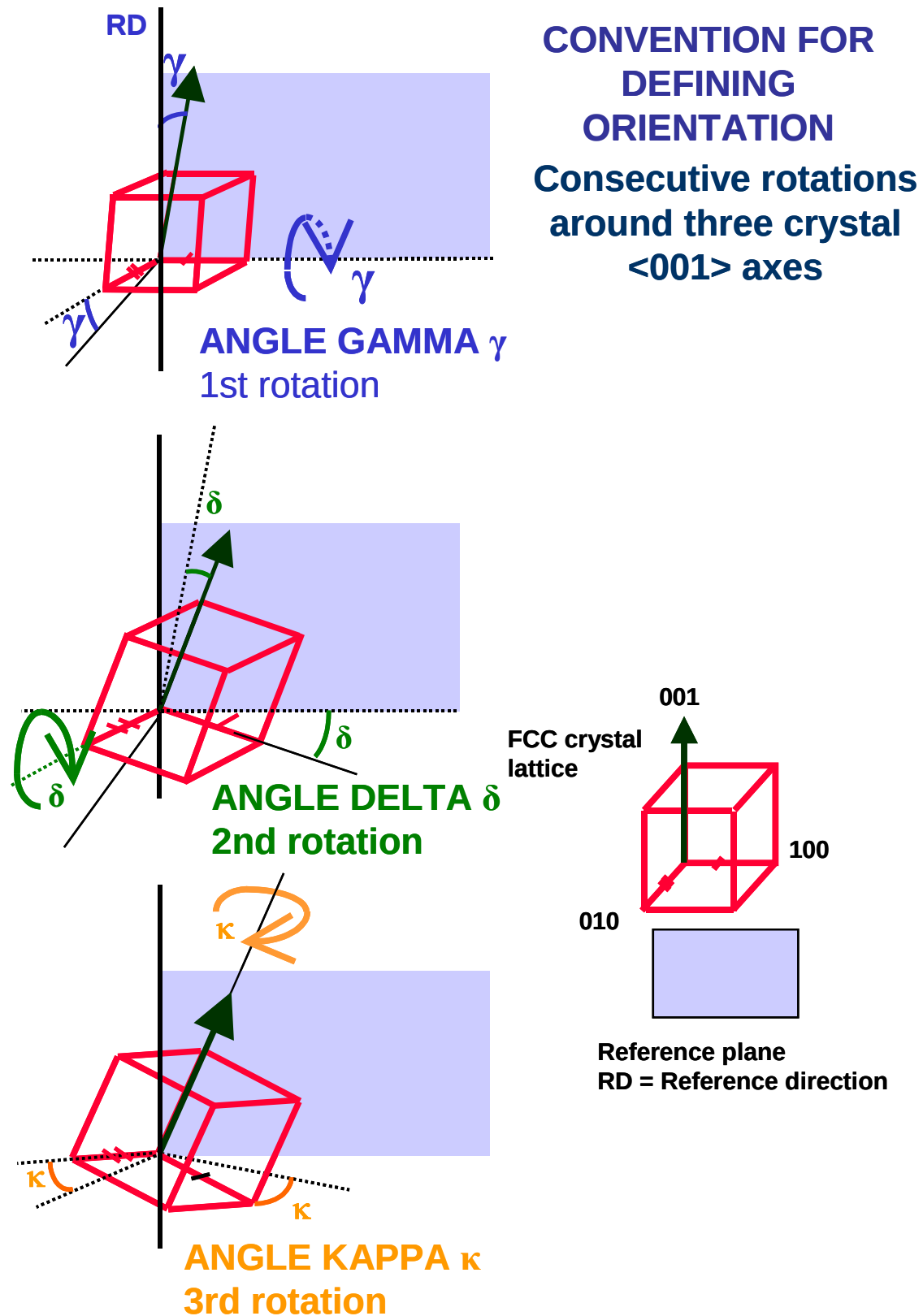


Figure 2.6(a): Visual representation of primary orientation angles: gamma, delta and kappa.

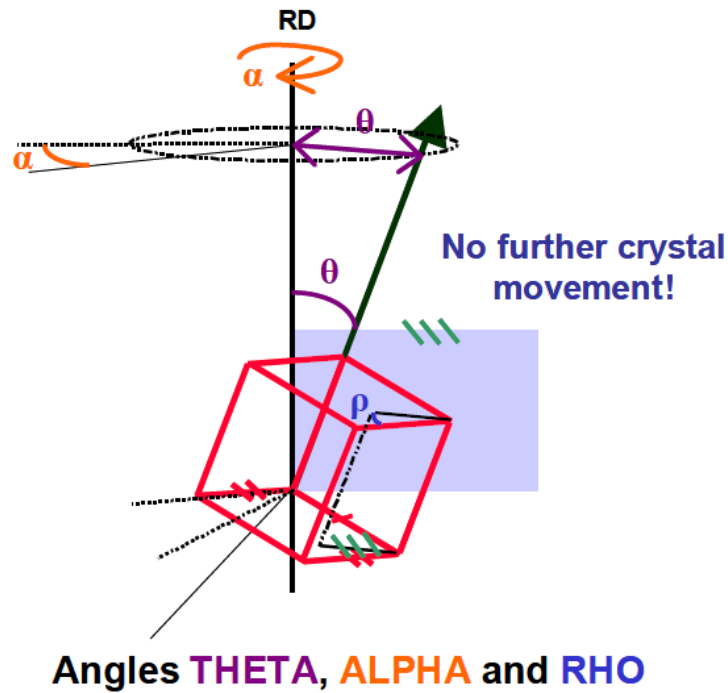


Figure 2.6(b): Stereographic projection illustration of crystal orientation (European convention): *theta*, *alpha* and *rho*.

2.1.5 Calculation of derived primary orientation angles (European convention)

The formulae for calculation of angles *theta*, θ , *kappa*, κ , and *rho*, ρ , from the measured angles *gamma*, γ , *delta*, δ , and *alpha*, α are shown in Table 2.1.

Table 2.1: Calculation of derived angles from directly measured angles, primary orientation (European Convention) [10]

Derived angle	Formula	Notes:
<i>theta</i> (θ)	$\cos \theta = \cos \gamma \cdot \cos \delta$	
<i>kappa</i> (κ)	$\kappa = \kappa_{\text{TRUE}} - \text{atan}(\tan \gamma \cdot \sin \delta)$	* See below for definition κ_{TRUE}
<i>rho</i> (ρ)	$\rho = \text{atan}(\tan \gamma / \sin \delta) - \kappa_{\text{TRUE}}$	* See below for definition κ_{TRUE}
<i>kappa</i> _{TRUE} (κ_{TRUE})	$\tan(\kappa_{\text{TRUE}}) = \cos \gamma \cdot \tan \alpha / (\cos \delta - \sin \gamma \cdot \sin \delta \cdot \tan \alpha)$	* See below for definition κ_{TRUE}

The formulae within Table 2, make use of an angle κ_{TRUE} . This is a parameter required in consistent 3-D mathematical analyses of the primary orientation angles. κ and κ_{TRUE} have the same rotation axis ($\langle 001 \rangle$ direction nearest to the reference direction) and rotation direction (clockwise) within their definitions, but differ in the datum for the rotation. The rotation datum for kappa is the reference plane whereas for κ_{TRUE} , the datum is the trace of the $\{001\}$ that is both normal to this rotation axis and nearest to the reference plane before applying the κ rotation, see Figure 2.7.

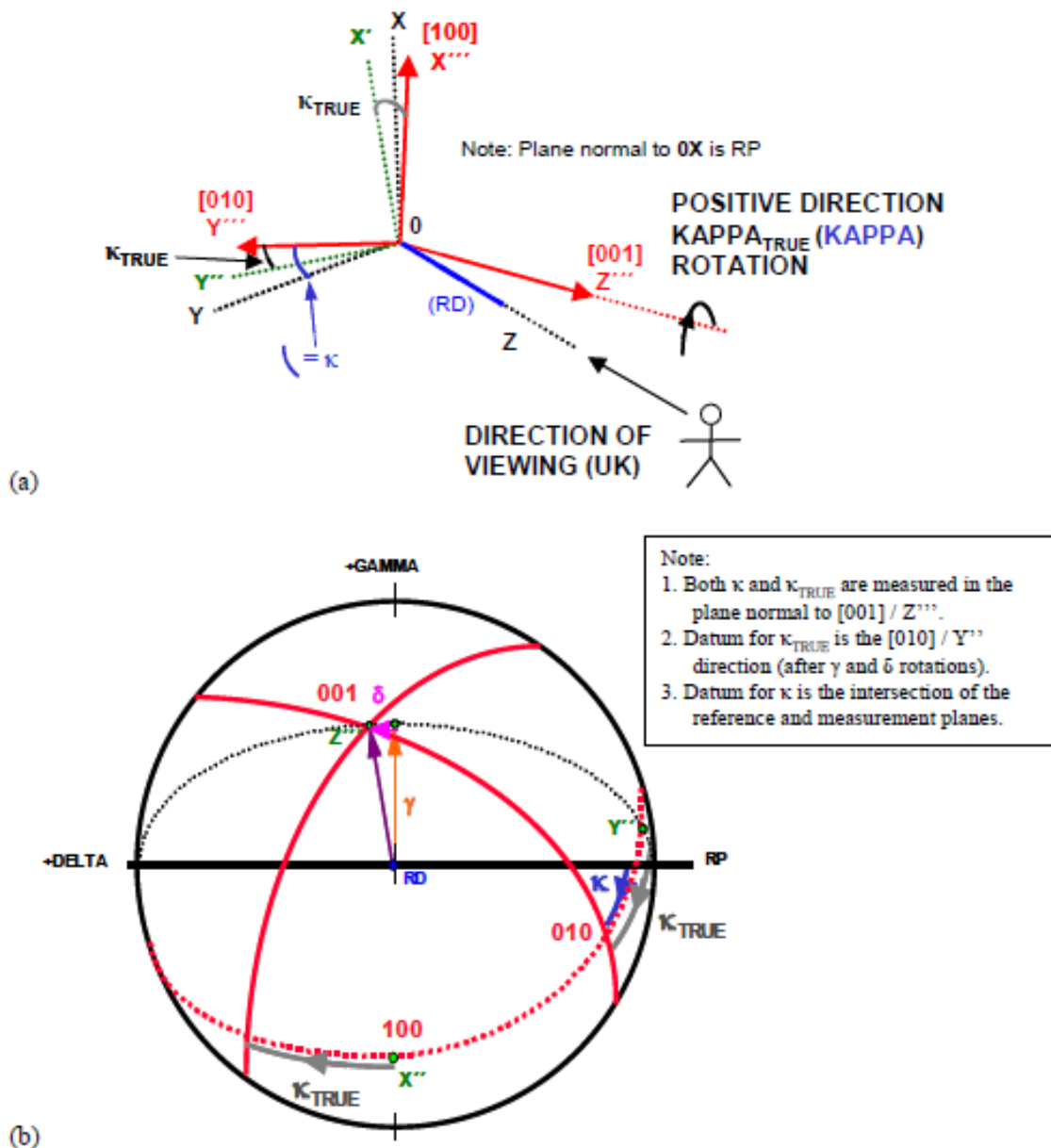


Figure 2.7: Datum differences for angles κ and κ_{TRUE} . (a) visual representation, (b) stereographic projection.

2.1.6 Primary orientation angles (US Convention).

As described in section 2.1.3, the direction of view of the BRL pattern (and hence direction of viewing of a component) is critically important. When the same crystal alignment is viewed from the alternative viewing positions the *observed* positions of the diffraction pattern are different, Figure 2.4. To obtain the same values for primary orientation angles, adjustments will need to be made to the sense (direction of rotations) for the directly measured angles.

In the US convention, the BRL pattern (hence component) is viewed looking back at the film from the specimen, *i.e.* towards the X-ray source. However, the definitions of positive sense directions for two of the directly measured angles (designated gamma and delta in the US convention) are the same as the equivalents in the European convention. However, the definitions of positive sense directions of the directly measured angle beta is altered. So without any adjustments to account for the different viewing, the values of the directly measured angles are different between the US and European conventions.

If, however, the positive sense rotations of the US vs. European conventions are compared, when viewing the component examined surface, gamma and delta have equivalent positive directions, and hence the same numerical values, but European alpha and US beta have opposite positive senses, and hence different numerical values.

The formal written definitions for primary orientation angles in the US Convention are detailed in Appendix 2.

In addition to the different directions of viewing the component and the film, there are other systematic differences between the European convention and this US convention (US1).

- (i) Use of different names for some ‘equivalent’ primary orientation angles.
- (ii) Definition of a different number of angles (six in the European convention, four in the US convention).

The conventions are compared in Table 2.2. Significantly,

- (a) US1: γ has the same numerical value as European γ .
- (b) US1: δ has the same numerical value as European δ .
- (c) US1: α has the same numerical value as European θ .
- (d) US1: β is analogous to European α but has a different numerical value (it approximates to the complement of the angle at small values of gamma and delta).

Mathematical relationships exist for the angles in the different conventions, so orientation data can be transformed between the conventions.

Table 2.2: Comparison of European and US conventions for definition of primary orientation

Attribute	EUROPEAN convention	US convention (US1)
VIEWING CONVENTION	LOOKING TOWARDS THE COMPONENT.	LOOKING TOWARDS THE X- RAY SOURCE.
SENSE OF ANGLES	ROTATIONS, AS PER CLASSICAL DEFINITION.	ROTATIONS, AS PER CLASSICAL DEFINITION.
NUMBER OF ANGLES	6	4
ANGLE NAMES: ¹ = equivalent measurement and value ² = equivalent measurement, different value.	GAMMA, γ^1	GAMMA, γ^1
	DELTA, δ	DELTA, δ
	THETA, θ^1	ALPHA, α^1
	ALPHA, α^2	BETA, β^2
	KAPPA, κ	-
	RHO, ρ	-

It is important to note that the two conventions are both used in the single-crystal turbine casting industry, and that this can lead to confusion for users and suppliers of primary orientation data. One needs to explicitly define and know the convention requested and / or supplied, especially when comparing components and materials or defining manufacturing process controls.

A second, less common, US convention also exists. It has the same viewing direction and direction of rotations for gamma and delta as US1 but does not have a prescribed direction of rotation for beta: beta can be clockwise (positive) or anti-clockwise (negative) and is selected to have the smaller absolute value. To differentiate, it is called the US2 convention.

2.1.7 Other derived primary orientation angles.

Other primary orientation control angles can be derived from the directly measured angles and are of use to either the supplier foundry (where controlled and seeded orientation in the investment casting process can reduce or eliminate secondary grain defects) or to the turbine engine designer (component specific property control applications). Appendix 3 gives an example of one such angle, omega, ω , that gives a clear description of where the nearest <001> crystal direction is situated within a non-symmetric component cross-section profile.

2.2 Grain boundary R-values

2.2.1 Introduction

The R-value, a single number expressed in units of angle ($^{\circ}$), is a quantitative assessment of the difference between the orientations of grains adjacent to a boundary. It is also variously commonly described as ‘misorientation’ or, more correctly, ‘disorientation’, and is the minimum angular displacement between two grains. The two terms are used interchangeably but ‘disorientation’ is strictly correct [6]. The R-value is used in the SX turbine industry as a ‘figure of merit’ for grain boundaries and will have some correlation with the performance of the boundary within a component.

If grains on either side of a boundary are designated A and B, the R-value can be interpreted as a summary of movements (rotations) required to align grain A with grain B. So, any R-value has a pre-requisite that the orientations of each grain relative to the *same arbitrary reference system* need to be known. (The arbitrary reference system is usually not the formal component references, as described in section 1.2, as a boundary can occur at any position on the surface of a component).

Various different R-value definition conventions have been defined in the last thirty years. However, two conventions are recommended:

- 1) the two-angle, REL, (R-2cos)
- 2) the single-angle (R-1cos)

These are chosen because they are physically more rigorous than others, and are consistent over the complete possible numerical range for the parameter (0 to 62.8°).

2.2.2 Two angle, REL, (R-2cos) R-value definition

PHI (φ) and TAU (τ) are two minimum rotations which, when applied consecutively, transform grain A’s orientation to the alignment of grain B.

PHI (φ) is the minimum rotation of the primary grain required to align one $\langle 001 \rangle$ axes of grain A with an $\langle 001 \rangle$ axis of grain B. The axis of the rotation is perpendicular to both of these $\langle 001 \rangle$ axes and is therefore the intersection of the two planes defined by these axes.

TAU (τ) is the minimum rotation of grain A, about the aligned pair of $\langle 001 \rangle$ axes (one from both A and B grains), to achieve alignment of the remaining two pairs of $\langle 001 \rangle$ axes.

φ and τ are combined to generate the R-value, R:

$$R = \arccos(\cos \varphi \cdot \cos \tau)$$

A visual representation of the rotations, their illustration on a stereographic projection, and a fuller description of the calculations for φ , τ and the R-value (from directly measured orientation angles) are given in Appendix 4.

2.2.3 Single-angle (R-1cos) R-value definition.

A vector (axis) and an angle are both defined such that a rotation by the angle about the vector, bring the lattices of the two grains, A and B, into coincidence. The axis angle pair selected must be the minimum angle of the 24 different possibilities. The R-value, R, is the magnitude of this minimum angle.

(This approach is analogous to the angle / axis pair for misorientation and Rodrigues vectors approaches used in texture analysis studies [5].)

A visual representation of the rotation and a fuller description of the axis angle pair (from directly measured orientation angles) are also given in Appendix 4.

It is recommended that users and suppliers always indicate the method used for R-value calculation (using either the R-1cos or R-2cos coding). Use of any other definition convention has risks, not only of confusion, but because many of the other methods generate different values for the R-value parameter for the same physical boundary and are also generally not consistent across the complete R-value numerical range.

3. Principles of the back-reflection Laue (BRL) method

3.1 Basic principles

The main features of a true (BRL) experimental geometry, Figure 3.1, are:

- (i) Use of a narrow, parallel beam of white X-radiation, *i.e.* the beam is collimated to minimise angular dispersion (spreading). Molybdenum or tungsten X-ray targets are usually used, operating in ranges 25 – 40 kV and 15 – 40 mA.
- (ii) A fixed orientation of the incident X-ray beam relative to the diffracting surface of the specimen.
- (iii) A fixed position of the specimen.
- (iv) The specimen axis, SA, is coincident (and parallel) with X-ray beam axis, XA.
- (v) The diffraction (Laue) pattern is registered on a detecting plane (*e.g.* film) in a back-reflection position.
- (vi) The detecting plane, DP, is normal to both the specimen axis, SA, and X-ray axis, XA.
- (vii) The plane containing the specimen axis, SA, X-ray axis, XA, and the detector plane normal, DPN, is defined as the system measurement plane, MP.
- (viii) The specimen measurement position, XMP to detecting plane, DP, distance (along the axis DPN), is defined as the system working distance, S. It needs to be known and either fixed or empirically derived by a calibration routine. This is necessary to obtain quantitative angular measurements (*i.e.* orientation of a single crystal in a component).

The principles of orientation measurement in a BRL method are:

- (a) Diffraction occurs in three dimensions, emanating from a volume of material (diameter approximately 2 mm and depth approximately 100 μm) at the measurement surface, XMP (Figure 3.1).
- (b) The detecting plane registers the diffraction poles (spots), each generated by a set of crystal planes from within the single crystal that satisfy the Bragg diffraction conditions (Figure 2.3).
- (c) The poles form a pattern that:
 - (i) is characteristic of the symmetry of the FCC crystal system;
 - (ii) has a position dependent only on the alignment of the crystal relative to the measurement reference system.
- (d) Due to the geometry of the BRL system, the diffraction poles (spots) within the pattern are aligned along hyperbolae, referred to as ‘zones’.

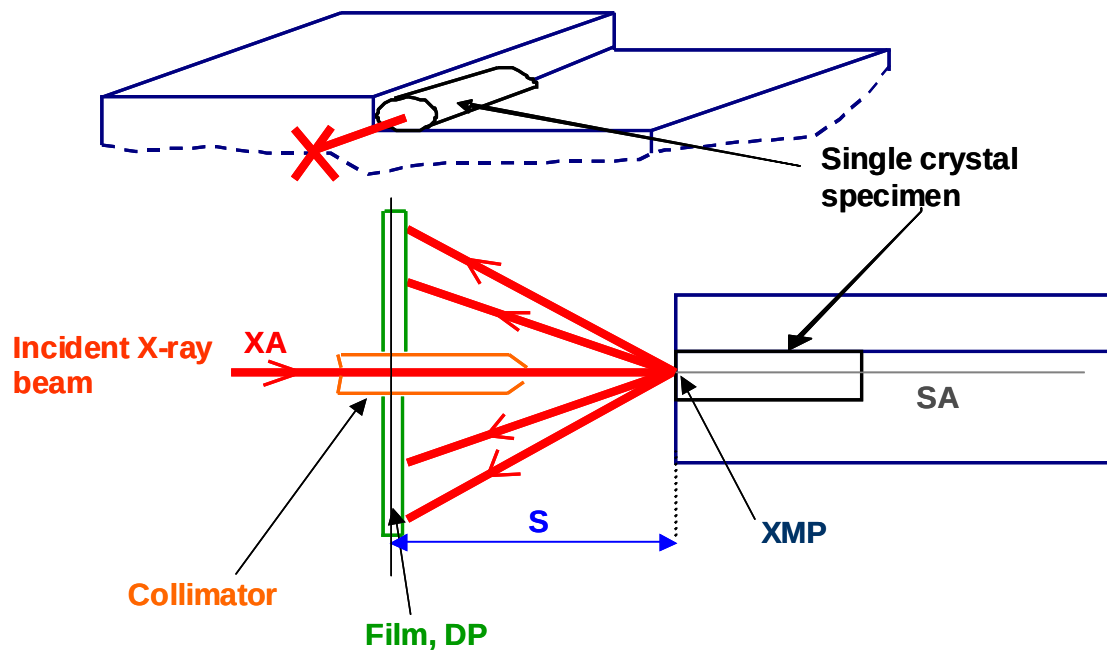


Figure 3.1: Back-reflection Laue geometry.

- (e) The position of the diffraction pattern (translation and rotation) depends upon the ORIENTATION OF THE CRYSTALLITE relative to the incident X-ray beam.
- (f) The position of the 001 pole of the Laue pattern, relative to RD and RP indicates the *gamma*, γ and *delta*, δ angles. The rotation of a 001 zone within the plane of the Laue pattern indicates the *alpha*, α , rotation – European convention (Figure 2.3).
- (g) Pattern analysis involves unambiguous identification of an 001 pole and an 001 zone, then measurement of gamma, delta and alpha and the subsequent calculation of the remaining primary orientation angles.

3.2 Practical points with the BRL method and single-crystal orientation measurement

- (a) The BRL method yields information on the alignment of the FCC crystal relative to the X-ray beam (and to a plane defined within the detection plane, measurement system reference plane, MP). As the X-ray axis, XA, is coincident with the specimen axis, SA, orientation information relates to the alignment of the crystal relative to the specimen axis, SA, and the measurement system reference plane, MP.
- (b) The end user is only concerned with the alignment of the crystal relative to the component (specimen) references, *i.e.* component reference direction, RD and reference plane RP - see section 2. (In addition, some information may be required on crystal alignment relative to the component shape – also see section 2.) To obtain this information from a BRL measurement system, the component references, RD and RP, must be aligned with the measurement systems' specimen axis, SA and

measurement plane, MP respectively. Individual fixtures (jigs) for each component type (part number) enable this.

- (c) Diffraction pattern analysis to identify significant crystal direction(s) and plane(s) for subsequent measurement of the orientation parameters requires identification of RP and RD on the detection plane with identification of positive and negative directions for gamma and delta. These must be consistent with:
 - (i) measurement system geometry, SA and MP.
 - (ii) component alignment, RP and RD and
 - (iii) orientation definition convention.
- (d) To generate a clear and easily distinguished diffraction pattern, the surface layer of material at the measurement position must be free from mechanical strain. The strain-free condition should extend to a depth of approximately 100 μm (*i.e.* equivalent to the X-ray beam penetration depth). Whether material removal by chemical or polishing methods is required depends on individual component surface conditions and the prior manufacturing processing operations. Component dimensional tolerances and restrictions also influence material removal options.

4. Industrial variants of the back-reflection Laue system

4.1 Image collection

The original BRL method collected the Laue diffraction pattern on a wet (transparent) film. The first, classical, convention for orientation definition viewed this film from the X-ray source looking towards the specimen (see viewing conventions and definitions, section 2.1.3).

To speed image generation, Polaroid film replaced wet film. Such films can only be viewed in one direction, *i.e.* looking towards the X-ray source or away from the test surface, which differs from the ‘conventional’ viewing of a wet film described above (also see viewing conventions and definitions, section 2.1.3). Appropriate adjustments to diffraction pattern analysis are required to attain equivalent orientation angles.

In order to meet the requirements for increased production volumes of SX castings, real-time Laue diffraction pattern image capture was introduced enabling faster measurements. The 2-D real-time area detectors that have been developed for this measurement application are mainly incorporated in side-reflection Laue systems. Only the more expensive detector types overcome the challenge of directing the X-ray down the centre of the device to produce a real-time back-reflection system. The adoption of side-reflection requires an extension of the theory to allow for the change from back to side-reflection geometry (see section 4.2).

4.2 System geometry

4.2.1 Introduction

The geometry a true BRL method is shown in Figure 3.1, whilst examples of a systematic geometry adaptation for side-reflection systems are shown in Figures 4.1 and 4.2. Because the adaptations are systematic, and can be mathematically defined, orientation measurements equivalent to those obtained in a true BRL geometry are feasible.

The main *DIFFERENCES* between side-reflection and back-reflection systems are shown in Table 4.1.

Real-time true back-reflection systems are now becoming available, which, due to their larger detector size, can be used at working distances in the range 30 – 100 mm.

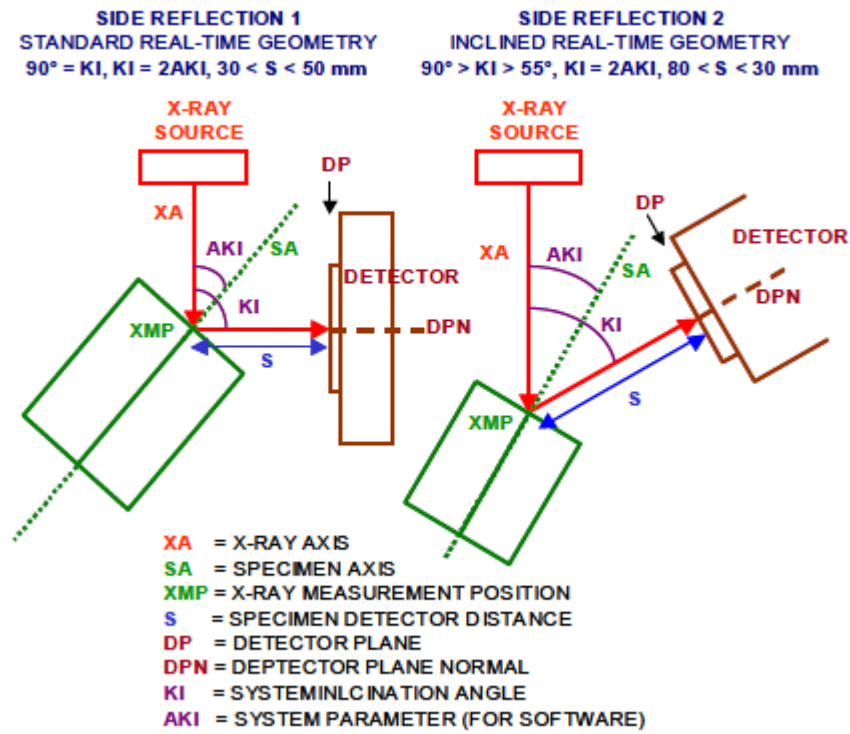


Figure 4.1: Side-reflection Laue geometry variants.



Figure 4.2: Example side-reflection real-time system.

Table 4.1: General comparison: back-reflection and side-reflection Laue systems

System feature	Back-reflection *	Side-reflection **
X-ray beam axis	The X-ray beam axis, XA, specimen axis, SA, and detector / film normal are all parallel and coincident.	(i) The specimen axis, SA, X-ray beam axis, XA, and detector plane normal, DPN, are co-planar (ii) The specimen axis, SA, is inclined at an angle AKI to the X-ray beam axis, XA. (iii) The detector plane normal, DPN, is inclined at an angle AKI to the specimen axis, SA, and is also inclined at an angle KI to the X-ray beam axis, XA. (iv) $KI = 2.AKI$.
Detectors	Most use film.	Real-time detector mainly used.
Diffraction pattern analysis	Off line either manual or semi automatic.	Normally rapid, integrated computer aided: matching a computer simulation with the pattern.
Geometry constraints	$AKI = KI = 0$ *	$KI = 90^\circ$, $AKI = 45^\circ$, $S = 30$ mm. ** or: $90^\circ > KI > 55^\circ$, $AKI = \frac{1}{2} KI$, $80 \text{ mm} > S > 30 \text{ mm}$. ** This latter side-reflection system geometry was evolved to increase the available space to accommodate larger components.

(Note: * refers to Figure 3.1 and ** refers to Figure 4.1)

4.2.2 Critical alignment considerations for side-reflection systems

Side-reflection Laue systems shall conform to the following alignment principles:

- (i) A narrow, parallel beam of white radiation, *i.e.* the beam is collimated to minimise angular dispersion (spreading). Beam collimation diameter should be of the order of 0.8 mm.
- (ii) A fixed specimen position.

- (iii) The specimen axis, SA, and X-ray beam axis, XA, are coplanar and define the measurement plane, MP.
- (iv) The orientation of the incident X-ray beam relative to the diffracting surface of the specimen is fixed. The angle between the X-ray beam axis, XA, and the specimen axis, SA, (within the measurement plane, MP) is AKI.
- (v) The plane of the detector, PF (DP), is normal to the plane defined by the specimen axis, SA and X-ray beam axis, XA, *i.e.* normal to MP.
- (vi) The specimen axis, SA, X-ray beam axis, XA, and detector plane normal, DPN, all intersect at a point XMP. This intersection, within the measurement plane, MP, defines the measurement position on a specimen surface.
- (vii) The detector plane normal, DPN, is inclined at an angle AKI to the specimen axis, SA, and is also inclined at an angle KI to the X-ray beam axis, XA (all angles are likewise within the measurement plane).
- (viii) $KI = 2.AKI$.
- (ix) The diffraction (Laue) pattern is registered on a detecting plane.
- (x) The specimen - detector distance (system working distance), *S*, needs to be known and either is fixed or is empirically derived by a calibration routine. This can be in the range 30 mm to 80 mm.

All angular alignments shall be achieved to an angular tolerance of $\pm 0.1^\circ$ for side inclination Laue systems.

Note: These values are only given as a guide for initial system set-up. Any system build should have some capability of adjustment to set all system elements relative to the X-ray beam axis, XA. Final system alignment is based on visual feedback from X-ray beam information and standard pattern information – see section 6.

Notes regarding the working distance *S*:

- (a) In the original manual analysis of true BRL films, pre-calibrated analytical charts (Greninger charts) were used to make direct measurements on Laue films. Hence, having the working distance, *S*, fixed exactly at 30 mm was paramount to achieving unambiguous pattern identification and consequent orientation angle information.
- (b) At a working distance of $S = 30$ mm, gamma / delta ranges of $\pm 30^\circ$ (theta angle 30°) could be attained on an active film area of approximately 90 x 110 mm (see Figure 2.2).
- (c) With the introduction of computer aided analysis protocols for Laue pattern identification (see Section 4.3), the working distance *S*, is effectively a scaling parameter within analytical software. So, provided that the actual working distance for a particular hardware set-up can be unambiguously entered into the software by a calibration routine (see sections 5, 6 and 7), the exact value of *S* need not be known.
- (d) Practical limits exist for the working distance *S*. They include:

- (i) The flux of the incident X-ray beam (increases in S , reduces image intensity as the inverse of the square of the distance).
- (ii) The active area of the detector (as a minimum detected image area of theta angle $\pm 22^\circ$ is the norm for most computed aided pattern analysis protocols). This minimum needs to be achieved at the chosen S for any given detector active area.
- (iii) The need for a large component envelope, to prevent component collisions with system hardware (especially with large components during R-value measurements) prescribes larger values of S .
- (iv) The requirement to have all key systems axes intersecting at XMP on side-reflection geometry systems (see section 4.2.2.)

4.3 Pattern analysis protocol

4.3.1 General information

The logical phases of any BRL pattern analysis are:

- (i) Unambiguous pattern symmetry identification.
- (ii) Definition of the position of the pattern (location and rotation) relative to measurement reference system.
- (iii) Application of the outputs of (i) and (ii) to calculate the primary orientation angles, *i.e.* γ , δ and α .

Pattern analysis protocols fall into one of three categories: charting methods, full pattern overlay matching, and marking of individual diffraction poles (spots). Variants exist within each category.

4.3.2 Charting methods

The first category, charting methods, [3] and [4] (and also reviewed in section 2.1), has an analysis cycle time of over 15 minutes and requires the analyst to have a detailed understanding of crystal symmetry. In summary the main steps of these methods are:

- (i) Using a Greninger chart to read manually the positional parameters for each zone (line of spots) of the BRL pattern.
- (ii) Plotting the positions of each zone on a stereographic projection.
- (iii) Measuring angular relationships between zones on the stereographic projection.
- (iv) Use of data from (iii) and standard cubic system interplanar angle tables to index the stereographic projection (of the crystal orientation) and identify all $\langle 001 \rangle$ directions and $\{001\}$ planes.
- (v) Measuring the primary orientation angles on the stereographic projection.

4.3.3 Overlay mask matching

A computer generated overlay mask, matching to the measurement system geometry and appropriately scaled to its key dimensions, defines the second category. The most common mask format is a spot pattern, such as in Figure 4.3. Masks consisting of lines representing the zones of a diffraction pattern have sometime been used, but they can be subject to operator error when rotationally matching the pattern.

The operator has the option of using one of three masks, drawn up for the 001, 011 and 111 symmetry patterns, *e.g.* Figure 4.4. The selected mask overlay is translated and rotated within the computer overlay plane until a match with the diffraction pattern image is achieved. Its final position is indicated by the analysis software. A specialised space-mouse (more common) or keystroke commands are use to move the mask. Mask overlay methods are dominant in systems with real-time detectors.

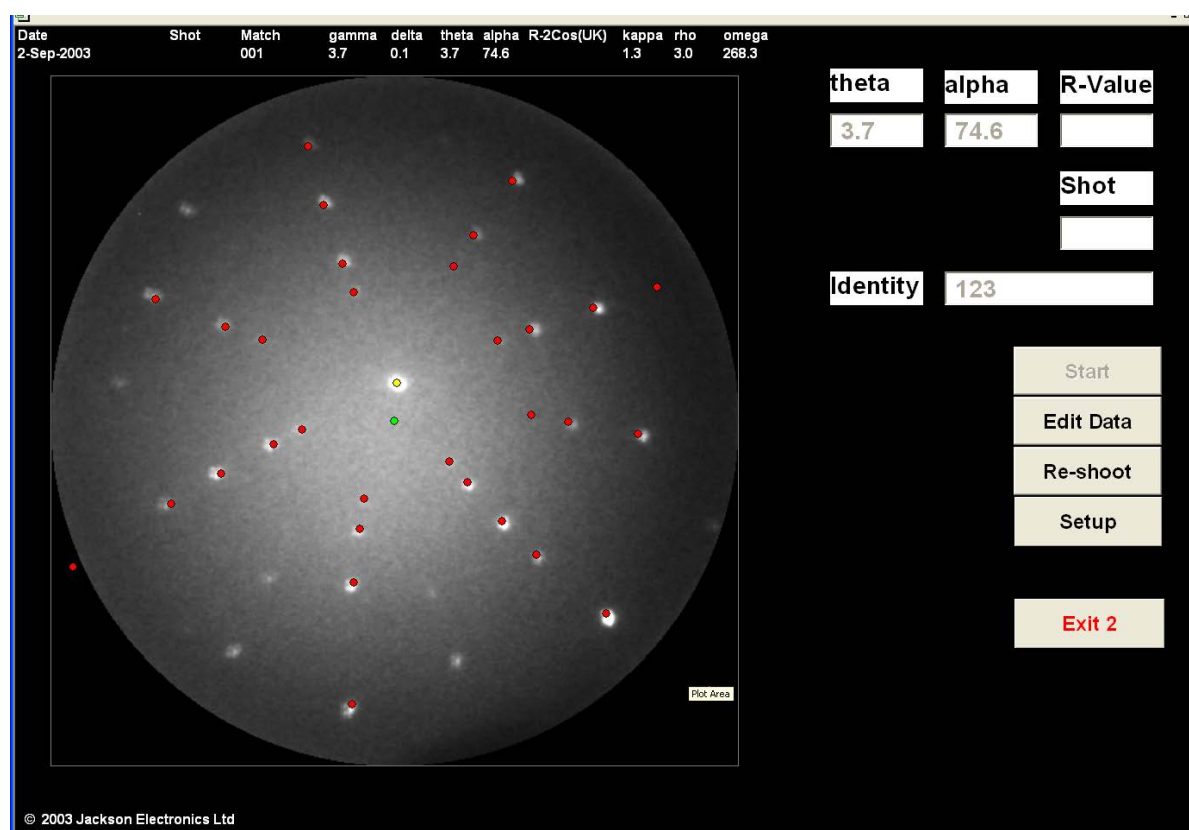


Figure 4.3: Mask and pattern matching in a real-time side-reflection Laue system set up for the European convention.

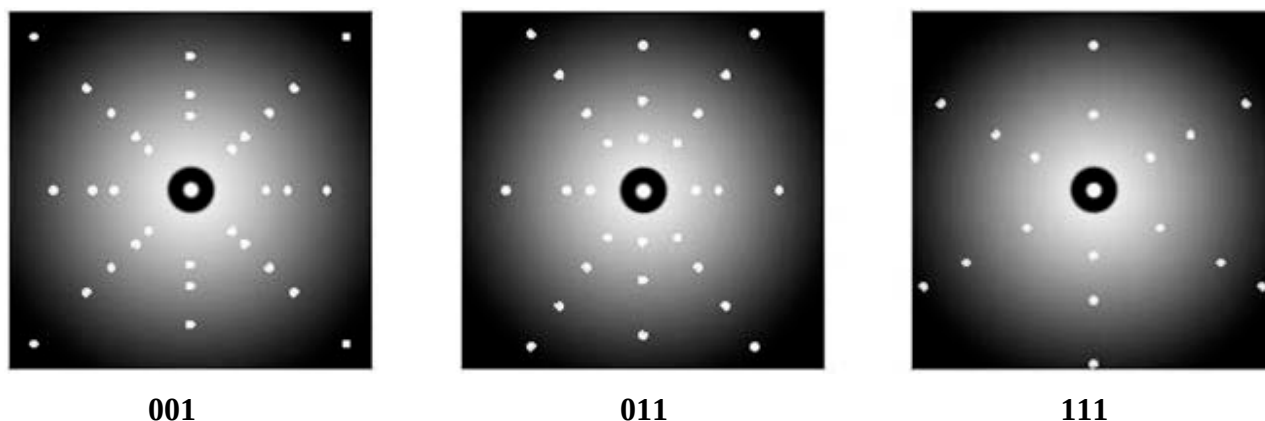


Figure 4.4: Overlay mask symmetry types.

The sequence of recommended steps when matching an overlay mask is.

- (i) Identify a prominent centre of symmetry (intersection) of the Laue image.
- (ii) Translate the 001 overlay mask so that its centre coincides with the noted Laue image centre.
- (iii) Rotate the mask around its centre to determine whether a match of all its zones (lines) with those of the Laue image is possible; consider only line position at this stage.
- (iv) If a line position match is obtained for the 001 overlay, check for pole (spot) matching. Further rotation of the mask may be required to achieve mask and image spot coincidence.
- (v) If an 001 mask match does not exist, select the 011 pattern (at the same Laue image centre). Rotate the 011 mask around its centre to determine whether a match is possible of all its zones (lines) with those of the Laue image; at least four different rotational position options will need to be tested. A rotational position match will also achieve pole (spot) match.
- (vi) If an 011 mask line position match does not exist, select the 111 pattern (at the same Laue image centre). Rotate the 111 mask around its centre to determine whether a match is possible of all its zones (lines) with those of the Laue image; consider only line position at this stage.
- (vii) If a line position match is obtained for the 111 overlay mask, check for pole (spot) matching. Further rotation of the mask may be required to achieve mask and image spot coincidence.
- (viii) If a match is not achieved for any of the three overlay mask symmetry types then another prominent centre of symmetry (intersection) of the Laue image needs to be selected and the pattern matching sequence ((i) to (vii)) repeated.
- (ix) Any initial mask match will need to be refined by fine adjustments so as to achieve an overall optimum fit.

4.3.4 Marking of individual diffraction spots

The final category is the marking of individual diffraction spots. Their coordinates within a calibrated field are entered into analysis software. For negative or positive film, a digitiser pad and either a pen or cursor, are used for computer data entry (Figure 4.5). Real-time systems using this type of protocol are rare, but usually use a box cursor for spot marking. Some systems request spot data entry in specified groups and order (*e.g.* along a diffraction zone line), both to minimise the spot data set size and to simplify the pattern symmetry identification routine.



Figure 4.5: Spot marking pattern analysis, back-reflection Laue films.

4.3.5 Difficult diffraction patterns

A clear diffraction pattern implies uniform crystallinity over the region probed by the incoming X-ray beam. However, this does not always occur. Indistinct patterns cannot be reliably indexed. The usual procedure is to re-etch the component in order to remove any surface layers which may contribute to an indistinct pattern but if this fails, the component often has to be quarantined. Examples of indistinct patterns are shown in Figure 4.6.

Split or doubled spots usually indicate two possible local orientations at the X-ray beam site, usually a consequence of sub-grain formation. Reliable matching may require the test location to be moved.

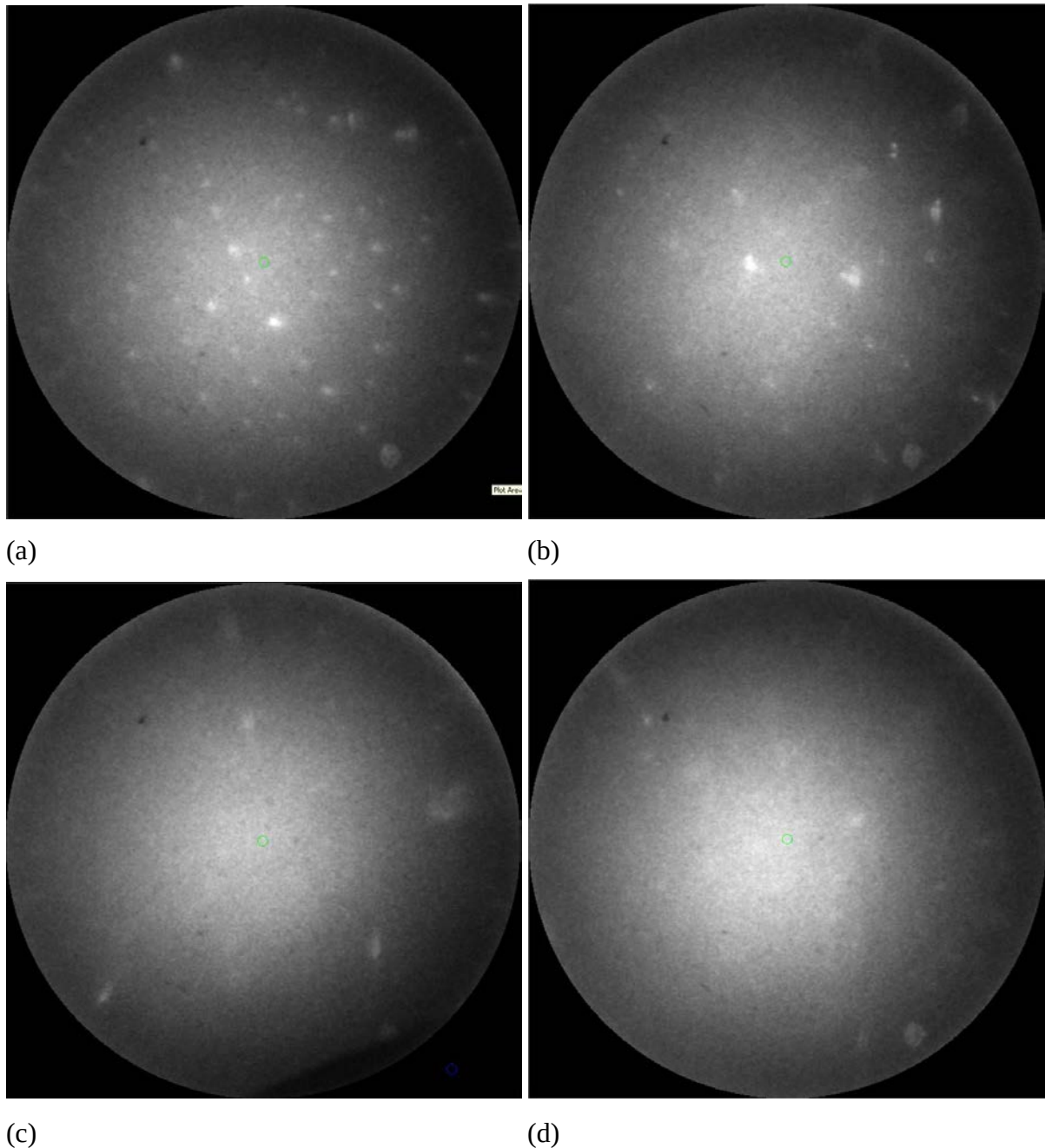


Figure 4.6: Patterns of varying clarity recorded using a real-time side-reflection system. (a) and (b) are probably indexable by an experienced operator, and both show spot splitting, but (c) and (d) are very doubtful.

4.3.6 Brief comparison of computer aided methods

In 4.3.3, steps (i) and (ii) of BRL pattern analysis using mask matching are straightforward separate operator functions entered sequentially into the software. In contrast, individual spot marking (4.3.4) is a more complex analytical procedure because both pattern symmetry and its position are derived from the spot coordinate data set alone. This is a potentially more fallible process, and so requires embedded software fail-safe checking routines. As the operating protocols and operator interactions of these two categories of computer aided methods are quite different, these factors are also thought to have significantly different influences on the precision and repeatability of orientation measurement.

5. Calibration standards

5.1 Nomenclature

Because silicon is normally used, the term silicon single-crystal standard, SSCS, is prevalent.

5.2 Use

The principle uses of a calibration standard are:

- (i) To confirm correct relative alignments by observation of its pattern symmetry;
- (ii) For scaling of analysis software to match the actual system working distance, S;
- (iii) Quality assurance.

5.3 Materials

The recommended material for a calibration standard is a **silicon single crystal**. This material is chosen because:

- (i) It is extremely stable at ambient and elevated temperatures;
- (ii) It has an FCC structure which is the same structure as single-crystal materials used in the turbine industry, and its unit cell size is very accurately known;
- (iii) It can be manufactured in a high-purity and defect-free condition; hence it has the potential to generate high-quality Laue diffraction patterns.

5.4 Recommended shape

The recommended shape for a standard is a cuboidal block with a recommended size of approximately 30 x 60 x 8 mm, to enable it to be readily aligned in typical fixtures used by the industry.

5.5 Recommended alignment

The required alignment of the silicon crystal relative to the features of the standard shall be:

- (i) A crystal $\langle 001 \rangle$ axis must be parallel (ideally $\pm 0.05^\circ$, $\pm 0.1^\circ$ worse case) with the longest axis of the standard block and
- (ii) All $\{001\}$ planes should be parallel (ideally $\pm 0.05^\circ$, $\pm 0.1^\circ$ worse case) with all faces of standard.

This alignment is chosen so as to give a diffraction pattern from any plane of the specimen (equivalent to being aligned with one of its axes parallel with the SA of a Laue measurement system), which is appropriate for both measurement system alignment confirmation and for analysis software scaling. See section 6.1 for further details.

5.6 Identification

A single-crystal standard should be uniquely identified and ideally have a nominated and labelled measurement surface.

5.7 Traceability

The standard either:

- (i) shall be traceable to a National Measurement System. A standard such as NIST SRM1994 provides this traceability;
- (ii) or, shall be otherwise independently certified as conforming to its specified crystal orientation alignment, *e.g.* independent certification from the standard's manufacturer where an alternative (non-BRL) higher resolution method for orientation measurement has been used, such as rocking curves, or standard testing within an aligned and verified BRL system.

Standards can be self-checked on an aligned and calibrated system by obtaining images from both of the 6 mm x 30 mm faces, in all possible specimen alignments. The movement between the different standard locations involves rotations of 180° around all of the standard's axes. A truly acceptable standard will not have any discernable movement of the generated diffraction pattern between any of its alignments.

5.8 Care of standard

A silicon single-crystal standard is very brittle, and therefore needs to be handled with extreme care, avoiding local impacts. However, the standard can tolerate local chipping provided that:

- (i) regions of the nominated measurement surface remain undamaged and accessible when used in the BRL measurement system;
- (ii) the damage does not prevent specimen lying flat in an appropriate measurement system alignment fixture.

5.9 Safety mounting

Since silicon is more transparent to X-rays than nickel alloys, it is recommended that a silicon single-crystal standard is permanently mounted in a robust holder that has an X-ray opaque upper covering (*e.g.* lead).

6. System alignment guidance

6.1 Introduction

System alignment is separated into three distinct stages for any system, plus an additional recommended step for side-reflection systems:

- (1) X-ray beam alignment;
- (2) Detector centring and rotational alignment;
- (3) Confirmation of co-planar alignments of all the critical axes;
- (4) Additional axes alignment verification for side-reflection systems

All the basic and critical alignments (sections 3.1 and 4.2) shall be addressed within the design and construction of any measurement system. An effective system design shall have some capability of adjusting at least the X-ray collimator and detector positions.

This system alignment guidance covers general practical procedures to check and refine the system alignment relative to the X-ray beam optics (hence the final diffraction pattern).

All the alignment adjustments described in the following sections are generally achieved on a ‘trial-and-error’ basis, because for safety reasons it is generally not possible to complete adjustments with X-ray beam switched on. Due to this iterative approach, it is advised that good experimental practices are observed (*i.e.* records / notes made of all changes).

The geometry alignment requirements for any system type define the relative alignments of 3 independent axes: *i.e.* the X-ray axis, XA, the specimen axis, SA and the detector plane normal, DPN. The first adjustment (section 6.2) defines the position of XA. Depending upon the mechanical build of a system, full free movement may be possible with both the DPN and SA axes, resulting in very complex system coplanar alignment adjustments (section 6.4). Ideally, one of these should be mechanically fixed or linked to the X-ray axis, XA (*e.g.* in a traditional film system, the SA is linked to the collimator mounting whilst in many contemporary side-reflection systems there is a link between the detector housing, hence DPN and the collimator mounting, XA).

As systems differ in their detailed arrangements, it is only possible to provide generic guidance for each of these three or four stages. Alternate procedures that complete equivalent verifications may exist for some bespoke systems. All stages need to be completed iteratively to achieve an optimised system alignment.

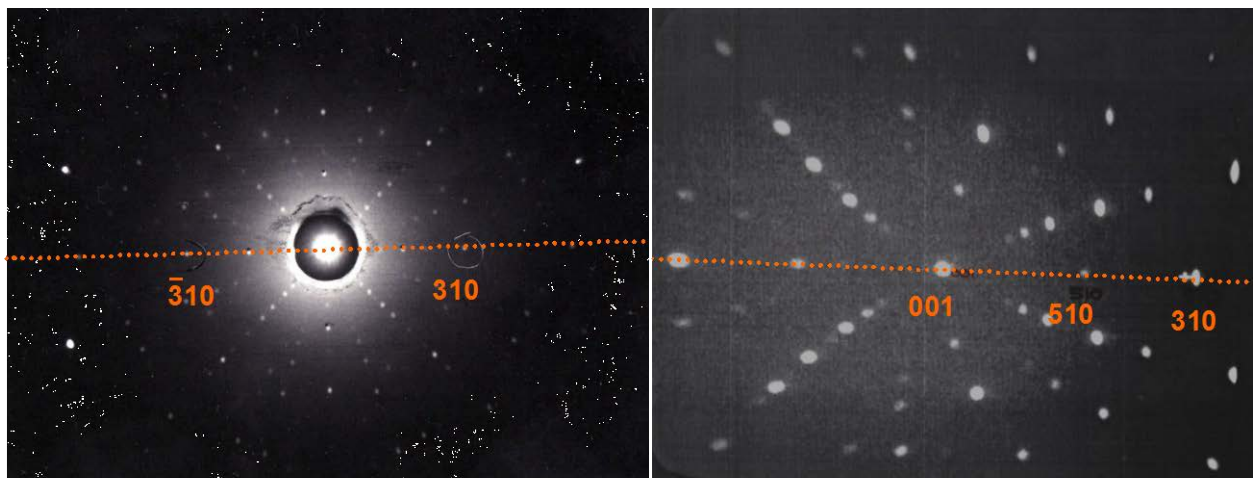
Personnel undertaking system alignment are strongly advised to use their system manufacturer’s notes as the primary source of information and direction. The following sections are provided as an explanation of the main stages of system alignment and are only intended for use in cases where appropriate manufacturer documentation is not available.

6.2 Key features of the Laue pattern of a silicon single-crystal standard

The 001 diffraction pattern generated from the silicon single-crystal standard (SSCS) is used in system alignment and calibration. Examples of such patterns are shown in Figure 6.1.

The key features of this pattern are:

- (i) A central 001 diffraction spot (or pole);
- (ii) A horizontal and straight line, of spots (all within the (001) crystal plane), known as the 001 zone;
- (iii) Within this 001 zone, a symmetrical spacing of diffraction spots (poles) on either side of the 001 diffraction pole;
- (iv) Horizontal plane reflection symmetry (termed horizontal symmetry) within the total diffraction pattern;
- (v) Other lines of spots (zones) pass through the 001 diffraction pole. These zones (which are only straight lines for a diffraction image collected in a true BRL system geometry) all have symmetrical spacing of diffraction spots (poles) either side of the 001 diffraction spot;
- (vi) The position of the 310 or 510 diffraction spots on the 001 zone (see Figure 6.1).



..... 001 DIFFRACTION ZONE

Note: both images are rotated. In perfectly aligned system the 001 diffraction zone lies HORIZONTAL

Figure 6.1: Silicon single-crystal standard Laue patterns (a) BRL system (b) side-reflection Laue system.

6.3 X-ray beam alignment

Purpose: To ensure that the X-ray beam emanating from the spot focus X-ray source is aligned coaxially with the mechanical beam guide (collimator) of the measurement system; together they define the measurement system X-ray axis, XA.

Beam characteristics will depend on the combination of X-ray source geometry (*i.e.* the size and shape of the X-ray focal spot), the controlling diameter of the collimator and the collimator design.

The scope of alignment adjustments will depend on the mechanical design of a system. Any or all of the following: collimator height, collimator horizontal shift, (*i.e.* (x, y) movement) and collimator pitch (collimator angle relative to the detector plane normal, DPN), need to be optimised and adjusted in turn. They will be an iterative sequence of optimising adjustments.

Assessment of beam / collimator coaxial alignment is achieved by either of the following:

- (i) Using a fluorescent screen with cross-wires (or an alternative scintillation target) to centre and maximise the intensity of the direct X-ray beam;
- (ii) Assessing and optimising the brightness of the Laue pattern from the SSCS when it is securely positioned in its appropriate fixture at the system working distance S (its position is set by either fixtures or gauges). The effectiveness of this method depends upon collimator design.

6.3.1 True back-reflection systems

Beam / collimator alignment assessment can be achieved by either of the methods (i) or (ii) noted in Section 6.3.

6.3.2 Side-reflection systems

All side-reflection systems should have the capability of independent collimator alignment.

In side-reflection systems, the detector plane does not intersect the direct beam path, so that using a relative diffraction pattern brightness assessment approach, (ii) above, is not recommended. The fluorescent screen is positioned either directly on the collimator or at the X-ray measurement point.

6.4 Detector centring (alignment of DPN) and rotation (detector alignment)

Purpose: To ensure that the reference direction RD, and reference plane, RP markers within the detector (hence pattern analysis systems) are co-incident or at the expected alignments with the measurement system specimen axis, SA, and measurement plane, MP, respectively.

Note: Software offsets and scaling should be set to zero prior to mechanical alignment. Working distance should be checked, particularly in the case of side-reflection systems.

To achieve detector centring, initially either the specimen axis needs to be fixed and the detector position, DPN, moved (as should be the case in true BRL systems), or the detector's position fixed and the specimen axis position moved (as in the case of most side-reflection systems). If the system has neither fixed, one of these axes should be considered as fixed and the other's alignment optimised, as described below, and then the other assumed to be fixed and the first axis adjusted. An iterative optimisation process ensues.

Note: Most systems have specimen axis, SA, drive capabilities (x and y movements normal to the specimen axis and sometimes z, parallel to the SA). SA alignment is the positioning of the axis itself, (and consequently the above linked drives) and not the movement / use of these drives.

Any alignment assessment uses the reference pattern from the SSCS. This SSCS must be securely positioned in its appropriate fixture at the system working distance, S.

6.4.1 Systems with fixed specimen axis, SA

The following steps are recommended:

- (i) The detector is horizontally and vertically shifted to obtain coincidence of the marked centre of the detector with the SSCS's pattern central 001 pole (spot).
- (ii) With (i) achieved, the detector may need to be rotated to achieve alignment of its marked horizontal (or equivalent) specimen reference plane, RP (also the trace of the system measurement plane, MP) with standard's pattern horizontal straight-line 001 zone.

Iterations of (i) and (ii) maybe required to attain both alignments. At this stage, the SSCS's diffraction pattern should be symmetrical around both the horizontal and vertical axes of the detector. If this is not the case the adjustments detailed in section 6.5 are required.

Note: if it is difficult to achieve (i) then coplanar adjustment (Section 6.5) should be completed prior to the detector adjustments.

6.4.2 Systems with fixed detector axis, DPN

The following steps are recommended:

- (i) The total SA assembly (inclusive any drive assemblies) is moved (by shimming or other adjustment facilities) either horizontally or vertically to obtain co-incidence of the marked centre of the detector with the SSCS's pattern central 001 pole (spot).
- (ii) With (i) achieved (and assuming that the fixtures are manufactured with the SA assembly measurement plane MP parallel to the plane defined by the X-ray axis and the detector plane normal, DPN), the detector may need to be rotated to achieve alignment of its marked horizontal (or equivalent) specimen reference plane, RP (also the trace of the system measurement plane, MP), with standard's pattern straight-line 001 zone. If however, there is some doubt regarding this fixture assumption, the critical alignment will first need to be confirmed by dial-gauge clocking and /or other standard mechanical metrology methods.

Iterations of (i) and (ii) maybe required to attain both alignments.

6.5 Co-planar alignment of all critical system axes

Purpose: To ensure, irrespective of system geometry type, that the three critical system axes, X-ray axis, XA, specimen axis, SA and the detector plane normal, DPN, are all coplanar and within the system measurement plane, MP.

The definitive evaluation of this condition is made by symmetry observations on the Laue pattern generated by the SSCS. If the coplanar condition is satisfied, the total diffraction pattern should be symmetrical around its horizontal axis (all system geometry types) and also its vertical axis (for true BRL systems only). Note that side-reflection systems will have vertical axis symmetry within its horizontal 001 straight-line zone only.

6.5.1 Assessment of SSCS diffraction pattern symmetry

The practical method of assessing the symmetry of the SSCS diffraction pattern symmetry will depend the pattern analysis facilities associated with a particular measurement system.

However, the principles of all assessment are the same and are detailed in Appendix 6.

6.5.2 Adjusting system coplanar alignments

Note: The following interpretations of the causes of and remedial action for system asymmetry ASSUME that there are no optical inversions within the displayed diffraction image, and that the image is viewed as if looking towards the detector or X-ray source from the specimen. As systems may be different in this respect, a specific system's diffraction image inversions need to be established and interpretations and actions amended accordingly.

- (a) Horizontal SSCS pattern asymmetry indicates that either the specimen axis, SA, or detector plane normal, DPN, is not parallel with the system X-ray measurement plane, MP. The misalignment will be a pitch of the axis (*i.e.* rotation around an axis within the measurement plane that is normal to either SA or DPN).

- (i) **Systems with fixed specimen axis, SA**

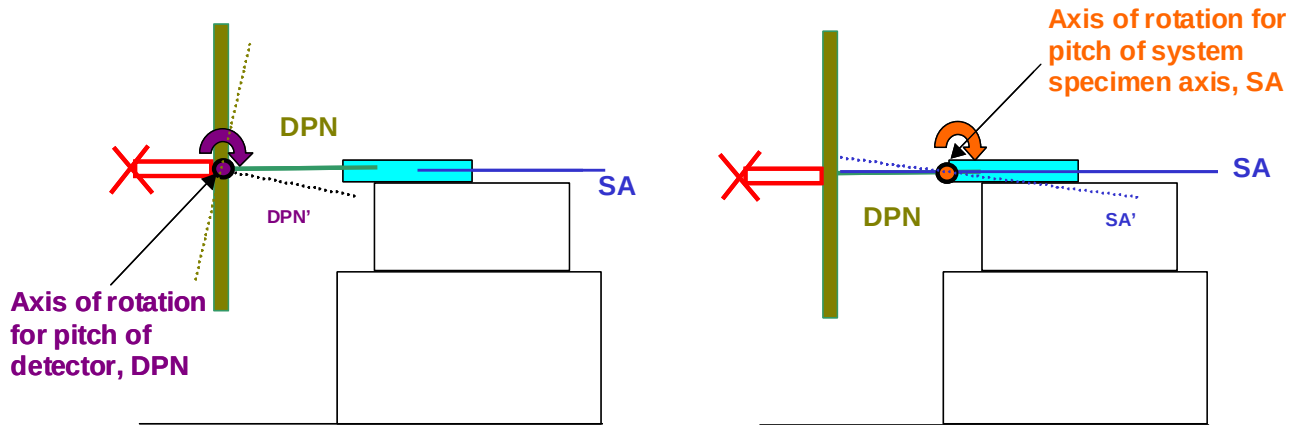
The horizontal asymmetry will be due to a rotation around the horizontal axis of the detector. If from the assessment of 6.5.1, the top half of the observed SSCS pattern is closer to the horizontal centre (*i.e.* smaller) than the bottom half, then the top half of the detector is closer to the specimen than its bottom half. The detector alignment will need to be adjusted to correct the symmetry aberration. Likewise, when the observed pattern bottom half is smaller, then the bottom half of the detector is closer to the specimen than its top half.

- (ii) **Systems with fixed detector axis, DPN**

The horizontal asymmetry will be due to a rotation around the axis within the measurement plane that is normal SA. If from the assessment of 6.4.1, the top half of the observed SSCS pattern is closer to the horizontal centre (*i.e.* smaller) than the bottom half, then, when viewing the SA from detector, its measurement end is pitched below the system measurement plane, MP. The SA alignment will need to be adjusted to correct the symmetry aberration. Likewise, when the

observed pattern bottom half is smaller, the SA's measurement end is pitched above the system measurement plane, MP.

The above for horizontal pattern asymmetry are summarised in Figure 6.2.



(a) Systems with fixed specimen axis (SA) (b) Systems with fixed detector axis (DPN)

Figure 6.2: Sources of system horizontal asymmetry.

- (b) Vertical SSCS pattern asymmetry indicates that either the specimen axis, SA, or detector plane normal, DPN, is not parallel with their specified positions within the system X-ray measurement plane, MP. The misalignment will be a yaw of the axis (*i.e.* rotation around an axis normal to the measurement plane, MP).

(i) **Systems with fixed specimen axis, SA.**

The vertical asymmetry will be due to a rotation around the vertical axis of the detector. If from the assessment of 6.4.1, the left half of the observed SSCS pattern is closer to the central 001 pole (*i.e.* smaller) than the right half, then the left half of the detector is closer to the specimen than its right half. The detector alignment will need to be adjusted to correct the symmetry aberration. Likewise, when the observed pattern right half is smaller, then the right half of the detector is closer to the specimen than its left half.

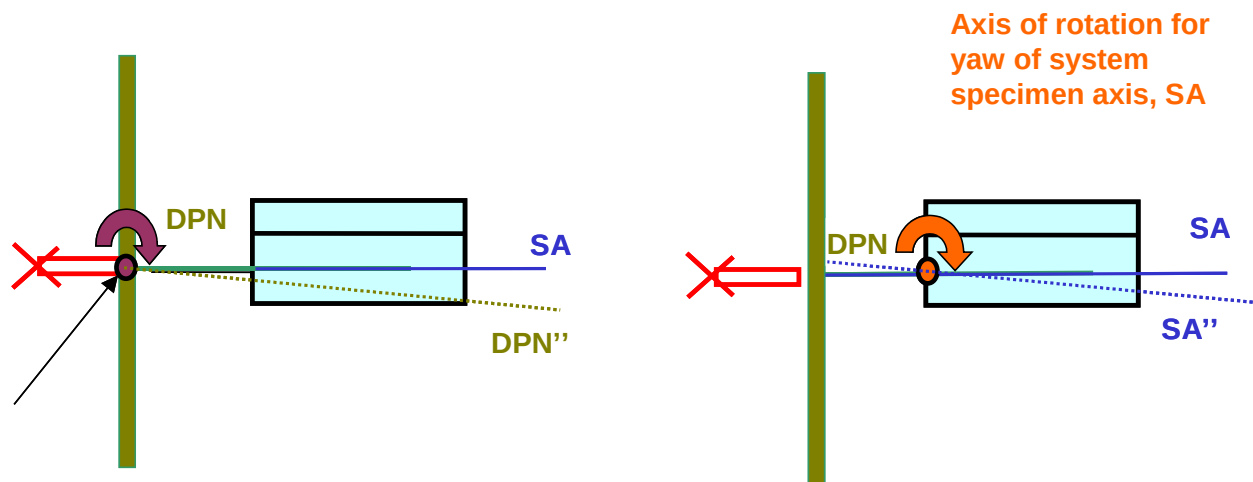
(ii) **Systems with fixed detector axis, DPN**

The vertical asymmetry will be due to a rotation around an axis normal to both the measurement plane, MP and specimen axis, SA. If from the assessment of 6.4.1, the left half of the observed SSCS pattern is closer to central 001 pole (*i.e.* smaller) than the right half, then when viewing the SA from detector, the left hand side of the specimen axis is closer to (yawed towards) the detector. The SA alignment will need to be adjusted to correct the symmetry aberration. Likewise, when the observed pattern right half is smaller, then the right hand side of the specimen axis is closer to (yawed towards) the detector (on viewing SA from the detector).

The above for vertical pattern asymmetry are summarised in Figure 6.3.

As noted earlier in section 6.1, asymmetry correction for systems that have freedom of movement of both SA and DPN is very complex. The alignment of such systems requires a very systematic iteration of all the adjustments noted in this section, 6.5.

For any system, symmetry corrections may shift the centring of the pattern within the detector plane. So, detector re-centring adjustments (section 6.4) maybe required either between or on completion of system asymmetry adjustments.



(a) Systems with fixed specimen axis (SA) (b) Systems with fixed detector axis (DPN)

Figure 6.3: Plan views of system (true BRL geometry example); sources of system vertical asymmetry.

6.5.3 System alignment verification

For all systems, a final practical check to verify system alignment is to slide the detector back along its axis (if practical), or for true BRL systems only to slide the SCCS away from the detector along the specimen axis. In both cases, the SCCS 001 pole should remain at the detector centre. This should also hold true for movements that bring system parts closer together.

6.6 Final alignment verification, side-reflection geometry systems only

All side-reflection systems require the three main system axes, XA, SA and DPN, to all intersect at a common point, XMP. This should be the position of measurement surface of any specimen. Furthermore, the system working distance, S , has its datum at XMP.

So, if the system is aligned (and preferably calibrated) the following should be observed, assuming no image inversions (see 6.4.2):

- (i) If the SCCS is moved closer (approximately 2 mm nearer) to the detector, thereby reducing S , then the SCCS diffraction pattern should move to the LEFT.

- (ii) If the SSCS is moved further away from detector by an equivalent distance from XMP, thereby increasing S , then the SSCS diffraction pattern should move to the RIGHT.

If the SSCS 001 pole is not on the centre point of the detector, it indicates that the specimen measurement system does not correspond with the common point XMP. The total system will need to be corrected accordingly.

7. System calibration guidance

7.1 Purpose of system calibration

- (i) To confirm correct relative alignments of key planes and directions within a measurement system (see section 4.2).
- (ii) To provide a scaling routine for system analysis software, commensurate with the actual working distance, S .
- (iii) To have a quality assurance role.

The above practice is called **CALIBRATION** and is a mandatory requirement for ALL measurement systems.

It shall also be supported by a lower level, more-frequent, part number specific calibration verification, termed **VALIDATION** of calibration (see section 7.3).

7.2 Calibration

7.2.1 A silicon single-crystal standard, SSCS (see section 5), shall be used for primary calibration. It shall be securely placed in an appropriate fixture (that aligns its long axis parallel with the measurement system specimen axis, SA, and its larger surface parallel with the measurement system measurement plane, MP). Its nominated measurement surface shall be at the system measurement point, XMP, and also be at the working distance, S , from the detection plane.

7.2.2 For BRL systems using manual film analysis, S shall be set at 30 ± 0.5 mm.

7.2.3 For other systems, a calibration routine, may allow for some adjustment for offsets between collimator and detector centrelines. If used, this facility shall be used with caution (movements restricted to less than 0.2° equivalent).

Practical details will vary with different systems, but essentially, if available, this facility will:

- (i) Start with the detector horizontal and vertical axes at a software (0,0) position.
- (ii) Allow the movement of the horizontal and vertical software (detector) axes, which in combination define RP and RD, so that they are coincident with the SSCS diffraction pattern 001 zone and 001 pole respectively.

7.2.4 To provide a scaling routine for any measurement system, the position (or positions) of defined diffraction poles on the SSCS diffraction pole shall be marked in a defined sequence. These are either the 310 or 510 poles (see Figure 6.1 and Section 6.1).

The software is able to generate an angular scaling parameter for the actual system working distance, S . This is achieved from crystallographic first principles: the angle between (001) plane and (310) plane is fixed. Thus, marking of the 001 and 310 pole on the Laue image field gives an angular scale for the image field.

- 7.2.5 The system will retain the above zero offsets and scaling parameter until the next system calibration. Date of calibration and other operational details shall be retained with these calibration data.
- 7.2.6 Frequency of calibration
- (i) Primary calibration check (and re-calibration) as a minimum shall be completed whenever a change to system settings (including working distance setting) is made or suspected.
 - (ii) For measurement system quality assurance, a periodic check of calibration should also be made (with parameters and any actions recorded). The frequency of these periodic checks will depend on system usage and any organisation / customer requirements. The interval between checks should not be greater than a week, and may be as frequent as the start of every shift.
- 7.2.7 For systems where component-specific fixtures exist which do not have any facility to accommodate a SSCS, *i.e.* when the SSCS is held or located in the measurement system within its own fixture, primary calibration fulfils confirmation of the alignment of the system specimen axis and the SSCS only. As each component will have its own measurement fixture, a secondary calibration procedure should be completed as part of the primary orientation measurement to confirm alignment of the designated component reference direction, RD, and reference plane, RP, with the measurement system specimen axis, SA, and measurement plane, MP, respectively.

7.3 Validation

Purpose of undertaking validation: To ensure that for each component type at the time of primary orientation, the designated component reference direction, RD, and reference plane, RP, are aligned with the measurement system specimen axis, SA, and measurement plane, MP, respectively.

- 7.3.1 Validation is specific to each component type (or part number) and applies only to primary orientation measurements.
- 7.3.2 A standard component – known as the **Reference Component** – shall be provided for each component type.
- 7.3.3 Every Reference Component shall have recorded standard orientation data agreed for the measurement facility.
- 7.3.4 A secondary calibration procedure shall include:
- 7.3.4.1 The measurement of orientation of the Reference Component in accordance with the local operating procedures.
 - 7.3.4.2 A comparison of the measurement with previous orientation data of the Reference Component.

Note: The development of a statistical process control chart is strongly recommended.

- 7.3.5 Documentation for validation shall include the acceptance standards (criteria) for secondary calibration. These shall be based on the established precision of the measurement system.
- 7.3.6 Validation shall be carried out at the start and end of measurement of each component type.
- 7.3.7 If the validation results are unacceptable, then suitable preventative actions shall be performed. These shall include re-confirmation of primary calibration. Measurements must not continue for the component type until successful re-validation results are achieved. Subsequent repeats of validation non-compliance should be separately investigated.

8. Evaluation and statement of system's measurement capability

8.1 Introduction

The measurement capability of any measurement system needs to be established. Two aspects of measurement capability should be stated:

- (i) Consistency of measurement using the system ('repeatability').
- (ii) Comparison of measured parameters with those obtained from other facilities ('reproducibility').

These factors should be used by both facility owners and their customers as quality assurance controls for the use of a measurement system.

These factors can be assessed either independently for a laboratory or relative to a master body of data. The latter is recommended. However, this guide describes both assessment routes.

8.2 Conducting an assessment

It is recommended that a method such as that given in ASTM E691 is used for the analysis of the results.

The following sequence of steps provides an example of how to apply this standard to a set of X-ray analysis data.

8.3 Orientation parameter evaluation data sets

8.3.1 Specimens

An evaluation of system measurement requires a reference set of measured data for both primary and R-value orientation parameters separately.

For both, a set of at least three specimens shall be used and shall have the following attributes:

- (i) Alloy type, material condition and surface condition (at nominated measurement location) of all the specimens are representative of nickel superalloy turbine components.
- (ii) Fixtures required for every specimen are nominated and available to all participant measurement facilities.
- (iii) For every specimen, a measurement location or surface and its alignment during measurement are nominated.
- (iv) For primary orientation assessment, the specimens shall cover an appropriate range of angles gamma, delta (and hence theta) and alpha. See Appendix 5 for an example of such a specimen set.

- (v) For R-values assessment, the specimens shall have a range of R-values encompassing expected values below and above the range likely to be measured. See Appendix 5.1 for an example of such a specimen set.

Note: There would be advantages in developing an industry-wide nominated set (or sets) of reference specimens to be available for measurement system assessment purposes, and for the development of a Proficiency Testing (PT) scheme. The existence of a common set would permit a more rigorous statistical assessment of all measurement system capabilities without recourse to a nominated ‘master’ system. The former is recommended. However, this guide describes both assessment routes.

Assessments of intra-laboratory quality (short-term repeatability and longer-term reproducibility) and inter-laboratory correlation (reproducibility) without the nomination of a ‘master’ system should comply with the guidelines from the ASTM Standard E 691-05, a standard which covers inter-laboratory measurement studies [7].

8.3.2 Measurements

(i) Generic measurement requirements

Prepare the system for measurement as per local operating instructions, recording and supplying details of calibration checks and verification.

Use and record the customary X-ray settings (tube type, current and voltage); if any specimens required deviation from the settings to attain Laue images, record and supply details of variations.

(ii) Primary orientation measurement.

- (a) Complete one orientation measurement (or Polaroid shot) for each of the specimens.
- (b) Remove and replace the specimen fixture in system. Repeat one orientation measurement on each specimen, measuring specimens in the same order as in (a).
- (c) Repeat instruction (b) a further three times (so that a total of 5 measurements are obtained for each specimen).
- (d) For film based systems only, complete an analysis of the films to generate orientation parameters in the order of their data collection.
- (e) Measurements must be presented in an agreed convention and format (European convention recommended).
- (f) Gamma, delta and alpha are the directly measured primary orientation angles. Completion of the capability assessment (see section 8.3 and 8.4) for these angles should only be completed as part of understanding the origin of measurement eccentricities.

- (g) Theta and kappa are the primary angles of main interest to the users of the orientation data within the turbine industry. As a minimum, the system validation capability assessment shall be completed for these two angles.

(iii) R-value measurements

- (a) Collect data required for R-value assessment for each of the specimens in turn. Grain measurements should be made either directly on the boundary, or as close as reasonably practicable to the boundary location. Grain measurements taken more than 5 mm from a boundary are not recommended. Use the data to generate the R-value for each assessment.
- (b) Repeat instruction (a) a further 4 times. R-value data repeats for each specimen must not be collected consecutively.
- (c) For film based systems only: complete analysis of films to generate orientation data and R-values in the order of their data collection.
- (d) R-values must be presented in an agreed convention and format, with the R-1cos or R-2cos conventions being strongly recommended.
- (e) The system validation capability assessment shall be completed for the R-value.

8.4 System measurement consistency statement

Note: For all assessment details in this section, spreadsheet calculators are recommended. These should be used with care, with particular attention given to controlling the consistency of significant figures. Guidelines are given in [7].

8.4.1 Consistency assessment: repeatability (and comparing with other facilities)

The consistency of the measurement performance of a system relative to that achieved by other facilities provides a more rigorous assessment of precision and precision statistics for a measurement system. For this, equivalent repeat measurement data for the same specimen data set is required from ideally at least 6 different facilities.

System measurement consistency can be assessed by repeatability. Repeatability is defined [7] as the spread of results obtained with the same method on identical test specimens within the same organisation/facility (termed laboratory in [7]) by the same operator using the same equipment within short intervals of time.

For the test data set defined in 8.3, for every specimen in the test data set, the number of test results, n , is 5. The number of different facilities included in the comparison is denoted by p .

Separately for each parameter for every specimen for a system:

- (i) The average of the test results ($\bar{x}$) shall be calculated.
- (ii) The standard deviation (s or σ_n) shall be calculated.

- (iii) The standard deviation (s or σ_n) is required for every system under scrutiny. These values will be required to be calculated / recalculated for each system. They are used to calculate the repeatability standard deviation (s_r) (see Appendix 5.2).
- (iv) The within-system consistency statistic (k) shall be calculated. These will be required to be calculated / recalculated for each system (see Appendix 5.2).
- (v) The significant level for the consistency statistic, k , at 0.5% significance level, needs to be obtained from standard tables (Table 5 in [7]).

The consistency statistic, k , gives an indicator of the within-facility precision (compared with other facilities). Although its 0.5% significance level will vary with the number of facilities, p , a value of k greater than 1 indicates greater within-facility variability than average for all facilities and any value exceeding the 0.5% significance level will require investigation. Generally, high k values represent within laboratory imprecision whilst very small k values may indicate a very insensitive measurement scale or other measurement problems.

As a range of specimens with different values are being assessed, a pattern or consistent trend with k values for all specimens from a given facility will provide evidence of systemic measurement system problems.

8.4.2 Consistency assessment: reproducibility (and comparing with other facilities)

System reproducibility is another assessment of measurement system consistency. Reproducibility is defined [7] as the spread of results obtained with the same method on identical test specimens in different organisations/facilities (termed laboratory in [7]) by the different operators using different equipment.

For the test data set defined in 8.2, for every specimen in the test data set, the number of test results, n , is 5. The number of different facilities included in the comparison is denoted by p .

- (i) Separately for each parameter for every specimen for all systems, the average of the test results ($\bar{x}$) shall be calculated.
- (ii) Separately for each parameter for every specimen for all systems, the standard deviation (s or σ_n) shall be calculated.
- (iii) For each specimen and parameter, using the averages for all p systems, the 'mean of the means' ($\bar{x}^*$) shall be calculated, (See Appendix 5.2).
- (iv) For each specimen and parameter and every system, the 'cell deviation' (d) shall be calculated, (See Appendix 5.2).
- (v) Overall, the standard deviation of the cell averages (s_{x^*}) shall be calculated, using the d values for all p systems, (See Appendix 5.2).
- (vi) A provisional value for the reproducibility standard deviation (s_R) is calculated, (See Appendix 5.2).

- (vii) The actual value for the reproducibility standard deviation (s_R) must be finalised. It is assigned as the larger of the reproducibility standard deviation (s_R) and the repeatability standard deviation (s_r).
- (viii) The between-system consistency statistic (h) shall be calculated. These will be required to be calculated / recalculated for each system, (See Appendix 5.2).
- (ix) The significant level for the consistency statistic, h , at 0.5% significance level, needs to be obtained from standard tables (Table 5 in [7]).

d is a measure of a parameter's variance compared with the average of its variance for all other facilities. d can be interpreted as a measure of a combination of system alignment, calibration and systematic operating errors. Limits on values of d should be used within system validation and approval activities.

The between-system consistency statistic, h , gives an indicator of the significance of parameter variance compared with the average of the variance for all other facilities. Values of h can be either positive or negative. Although its 0.5% significance level will vary with the number of facilities, p , values of h approaching or exceeding the 0.5% significance levels will require investigation. With the range of parameter values recommended for orientation data sets, trends in h parameters will need to be established and systems that do not conform to the trends will require investigation. (Further detail can be found in the ASTM Standard E691-05 [7]).

8.4.3 Establishing expected precision statistics – repeatability and reproducibility - (for specimens and parameters)

The methods (detailed in sections 8.4.1 and 8.4.2) define how to generate for every system/specimen/parameter combination:

The mean value, $\bar{x}$

The standard deviation, s , and its associated consistency statistic, k .

Its variance compared with other systems, d , and the associated consistency statistic, h .

Furthermore, pooling data from a number of equivalent systems should be used to empirically establish the expected precision (precision statement) for the measurement method. A precision statement should include:

Average of system averages, (*i.e.* 'mean of the means'), x^* .

Standard deviation of the cell averages, s_{x^*} .

Repeatability standard deviation, s_r .

Reproducibility standard deviation, s_R .

95% repeatability limit, r , where $r = 2.8s_r$.

95% reproducibility limit, R , where $R = 2.8s_R$.

See sections 9 and 11 for recommended uses of these precision statistics.

8.5 Independent consistency assessment (general guidelines)

This section covers the less rigorous option for producing a stand-alone set of precision variance (bias) statistics based on test data obtained from just one facility, and then comparison with those obtained from a nominated master facility (datum system). Measurements of all parameters must be obtained from both the system under investigation and the nominated ‘master’ system.

For the test data set defined in 8.3, for every specimen in the test data set, the number of test results, n , is 5.

8.5.1 Precision (or uncertainty) statement

- (i) Separately for each parameter for every specimen, the average of the test results ($\bar{x}$) shall be calculated.
- (ii) The standard deviation (s or σ_n) shall be calculated for each parameter for every specimen for a system (See Appendix 5.3).
- (iii) The precision at 95% confidence level, $\pm 2s$, shall be calculated for each measured angle for each specimen for a system (See Appendix 5.3).
- (iv) A maximum observed precision can be derived for each angle for the system by pooling the results from each specimen. This requires the calculation of the combined standard deviation, σ_{combined} : the maximum observed precision is $\pm 2\sigma_{\text{combined}}$, (see Appendix 5.3.1).

8.5.2 Bias (deviation) statement

- (i) Separately for each parameter for every specimen, the average of the test results ($\bar{x}$) shall be calculated for both system under investigation and the nominated master system.
- (ii) For every specimen and each orientation parameter, the bias between the systems shall be derived as follows:

Mean (subject system) – Mean (nominated master system).

- (iii) The maximum calculated bias (to a 95% confidence level) for the subject system shall be derived for each orientation parameter.
 - (a) Derive the mean bias value of all the 8 specimens, $mean_{\text{bias}}$.
 - (b) Derive the standard deviation within the bias values for all the 8 specimens, σ_{bias} .
 - (c) If $mean_{\text{bias}}$ is positive, or equal to zero, then:

$$bias_{\text{max}} = mean_{\text{bias}} + 2.\sigma_{\text{bias}}.$$

- (d) If $mean_{\text{bias}}$ is negative, then:

$$bias_{\text{max}} = mean_{\text{bias}} - 2.\sigma_{\text{bias}}.$$

9. Practical limitations on system measurement capability (uncertainty)

9.1 Uncertainty statement

A measurement system shall have an up-to-date uncertainty statement³.

Recommended targets for the limits for uncertainty of measured orientation parameters should be available and appropriate for the specimen type (*i.e.* nickel turbine components during manufacturing processing).

Targets for measurement uncertainty can originate either from customer expectations within the industry or from the results from collaborative assessment programmes between measurement facilities. In both, the uncertainty is taken as either the 95% repeatability limit or the 95% precision limit: (maximum combined precision for a system or individual specimen precision levels may be used provided comparisons are consistent).

Such targets for all derived orientation parameters can be confirmed by theoretical sensitivity studies

9.2 Measurement uncertainty limitations: collaborative assessment

The SCOMS (Single-Crystal Orientation Measurement Standardisation) Project, 2007–09⁴, completed a round-robin comparison exercise using five different measurement facilities. Specimens were as detailed in Appendix 6.1, measurements as detailed in Section 8.3 and repeatability derived as in Section 8.4.

The measurement reproducibility expressed as a 95% confidence level (population standard deviation $\times$ coverage factor of 2) for the directly measured primary angles, γ , δ and α (European convention), alongside those for the calculated angles θ and κ , and the R-value are shown in Table 9.1. Consistency of these parameters are assessed by k , the within-system consistency statistic (see Section 8.4.2 and Appendix 6.2).

³ For a simple guide to uncertainty, see Bell [7]. For a downloadable guide developed by UKAS, see [8]. For the formal standard for citing uncertainty, see [9].

⁴ a *Joint Industry Project (formerly Materials Studio Project)*, part of the *Materials Measurement* programme sponsored by the National Measurement System unit of the UK's Department for Innovation Universities and Skills

Table 9.1: Measurement uncertainty limits (repeatability standard deviation) derived in SCOMS Studio Project Round Robin study (2007-09)

Parameter	Measurement uncertainty, 95% confidence ($k = 2$)
γ (gamma)	$\pm 1.2^\circ$
δ (delta)	$\pm 1.2^\circ$
α (alpha)	$\pm 2.0^\circ$
θ (theta)	$\pm 0.8^\circ$
κ (kappa)	$\pm 2.0^\circ$
R-value	$\pm 1.5^\circ$
Note: Number of facilities (p): 5 Number of specimens (m): 8 Number of repeat measurements (n): 5	

9.3 Measurement uncertainty: sensitivity studies.

The realistic viability of customer defined and empirically established orientation parameter uncertainty limits can be verified by error analysis studies. Only three of the primary orientation angles, γ , δ and α , are directly measured. In the round robin described above, standard deviations of repeated measurement were up to $\pm 0.5^\circ$, $\pm 0.5^\circ$ and $\pm 1.0^\circ$ respectively, depending on operator and system. Using the equations from Table 2.1 and these uncertainty levels, the effect on the values derived angles can be computed. In cases where the impacts of individual uncertainties act in the same direction, the maximum uncertainties are for theta θ : $\pm 0.7^\circ$, and for kappa κ : $\pm 1.5^\circ$.

Applying the same uncertainty levels for the directly measured angles to establish the impact on R-values, the calculated uncertainty is orientation angle difference dependent, but is typically $\pm 2.0^\circ$.

These error analysis sensitivity studies correlate reasonably well with the uncertainties displayed by the round-robin results.

9.4 Customer-defined measurement uncertainty limitations

A typical example of customer measurement uncertainty limitations (95% confidence) is displayed in Table 9.2 for both primary and R-value orientation parameters.

Table 9.2: Example of customer defined measurement uncertainty limits for orientation parameters (European convention for primary orientation)

Parameter	Measurement uncertainty limit (95% confidence),
γ (gamma)	$\pm 0.8^\circ$
δ (delta)	$\pm 0.8^\circ$
θ (theta)	$\pm 1.0^\circ$
κ (kappa)	$\pm 1.0^\circ$
α (alpha)	$\pm 1.0^\circ$
ρ (rho)	undefined if $\theta < 5^\circ$
	$\pm 10^\circ$ if $5^\circ \leq \theta < 7^\circ$
	$\pm 7^\circ$ if $7^\circ \leq \theta < 12^\circ$
	$\pm 4^\circ$ if $12^\circ \leq \theta < 16^\circ$
	$\pm 3^\circ$ if $16^\circ \leq \theta < 20^\circ$
	$\pm 2^\circ$ if $\theta \geq 20^\circ$.
R-value	± 1.5

The experimental results of the round-robin show that the above customer-defined limits are broadly realistic for all primary orientation parameters. The customer limit set for R-values typically contains a contingency for practical alignment difficulties of grain boundary measurement, *e.g.* proximity of measurement points to the boundary, but may underestimate the impact of uncertainty propagation.

10. Guidance on control of a measurement system

The information within this section is additional to the calibration advice contained within Section 7.

The selected items all have significant impact on the efficacy and precision of single-crystal orientation measurement. These items should all be covered by the good practice of a facility operating within ISO 9000:2000 (Quality systems) and / or ISO/IEC 17025:1999 (General requirements for competence of testing and calibration laboratories).

10.1 Methods

Documentation shall be subject to approval, issue and change control management (*i.e.* in accordance with local quality instructions), and shall exist for the following:

- (a) The technique(s) of specimen surface preparation.

Note: Multiple methods are included as different methods are and can be used for primary orientation and misorientation assessments.

- (b) A description of the measurement system and its key constituent parts. This shall include the alignment configuration and alignment conditions of the measurement system;
- (c) The operations to be performed which will lead to the final orientation data, for both primary orientation and R-values;
- (d) Formalised primary and secondary calibration and validation procedures (if used).

Additionally, the following shall also exist:

- (i) A list of parts on which the method is to be used;
- (ii) For each part number (or component type), descriptions of its approved fixture and reference secondary component (if used, see section 7.2);
- (iii) For each part number, for primary orientation, a description of its specified axes (*i.e.* reference direction, RD and reference plane, RP) and their alignment within its approved fixture / jig;
- (iv) For each part number, for primary orientation, a description of its measurement point (*i.e.* the defined location for orientation measurement);
- (v) Method used to establish formal measurement system uncertainty;
- (vi) Method(s) used for comparison of measurement systems.

10.2 Fixtures

10.2.1 Measurement system

All alignment features of the measurement system shall be subject to equipment calibration and control checks. These should be detailed in a local procedure and shall include the following (where applicable to the measurement system):

- (a) The silicon single-crystal standard shall be subjected to periodic degradation checks, and if independently mounted, the mounting checked.
- (b) Secondary calibration reference components shall be subjected to periodic degradation checks.
- (c) The primary orientation bed of the measurement system shall be subjected to periodic degradation checks.
- (d) The working distance pointer shall be checked to ensure that the correct working length is being used.

The frequency of these periodic checks will depend on system usage (measurement throughput) and organisation / customer requirement. The maximum period between checks should be specified in the local procedures.

10.2.2 Component fixtures

All part number / component specific alignment fixtures / jigs shall be subject to periodic independent metrology validation in accordance with metrology local procedures.

10.3 Operators

All operators shall be formally approved for single-crystal orientation measurement. A local procedure shall give approval details and associated documentation requirements.

Separate approvals shall exist for primary orientation and grain boundary R-value measurements (recognition of the higher level of operator proficiency required for set-up and measurements of grain boundaries).

As a minimum, initial operator approval shall include:

- (a) Theory training;
- (b) Practical training;
- (c) A specified minimum period of monitored experience;
- (d) Specified measurement competency demonstration;

For example: (1) generation of individual operator reproducibility statistics for the category of orientation parameters conforming to defined limits that should be equal to or better than declared measurement system uncertainty; or (2) comparative measurement of a specified significant number and range of orientations and components. Acceptance criteria for competency shall be defined.

A periodic re-approval of operators should be specified (measurement competency demonstration only).

The frequency of periodic re-approvals will depend on customer requirements and relevant local quality assurance guidelines.

11. Guidance on comparison of systems

A customer needs to be assured that measurements taken on any two different systems will generate equivalent orientation parameters within some defined limits.

This can be evaluated by either:

- (i) The reproducibility statistics from pooled data / collaborative assessment programmes, namely the cell deviation, d , the reproducibility standard deviation for a measurement system, s_R , and its associated between laboratory consistency statistic, h , (see section 8.3.2 and Appendix 5.2). The h statistic should be used to establish trend patterns for different specimens and measurement systems and used to identify abnormalities for specimen / laboratory combinations.
- (ii) For individual comparison of a system with a nominated master system, by the maximum calculated bias (at 95% confidence level, see section 8.4.1 and Appendix 5.3).

As noted earlier, the pooled data evaluation approach is recommended, where viable, as a nominated master system is not required.

The acceptability limits for comparison of systems apply either to d and R or to the maximum calculated bias value. Recommended values are shown Table 11.1. The limits only apply to the directly measured primary angles, γ , δ , α (EUROPEAN convention) and the calculated primary calculated angles θ and κ , and the R -value.

Table 11.1: Measurement system recommended acceptance limits for orientation parameters

Parameter	Measurement uncertainty repeatability, r
γ (gamma)	$\pm 0.8^\circ$
δ (delta)	$\pm 0.8^\circ$
α (alpha)	$\pm 1.0^\circ$
θ (theta)	$\pm 1.0^\circ$
κ (kappa)	$\pm 1.0^\circ$
R-value	$\pm 1.5^\circ$

The key to comparison of systems is to look for trends (using either the h statistic or historical of trends respectively) so that drift between systems over time (error creep) is noted and investigated.

The selected approach and method detail or formal comparison of systems shall be included in the formal methods records for a system (see section 10.1).

12. Completing measurements

Measurement systems differ with respect to their geometry and operating systems and also with their associated processing and inspection requirements to support specimen / component preparation. Therefore recommended generic operational flow-charts only are included for primary orientation and R-value assessments (Figures 12.1 and 12.2).

These should be used as a guide for good measurement practice and be reflected within the method's documentation (see 10.1).

Primary Orientation Measurement Flow Chart (Real Time Systems)

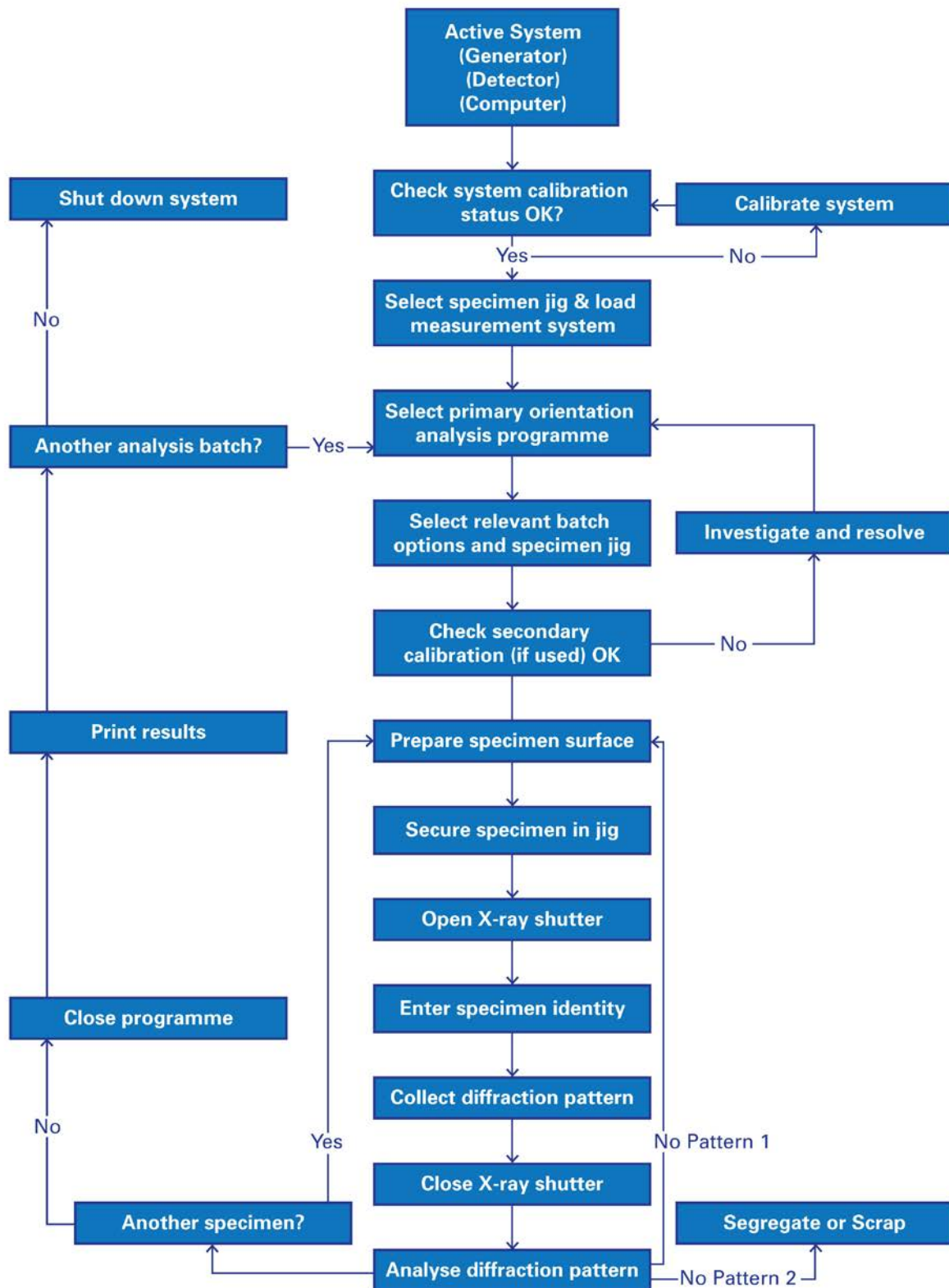


Figure 12.1: Flow chart, primary orientation measurement. Note: this chart only covers measurement steps, not component quality assurance decisions.

R-Value Measurement Flow Chart

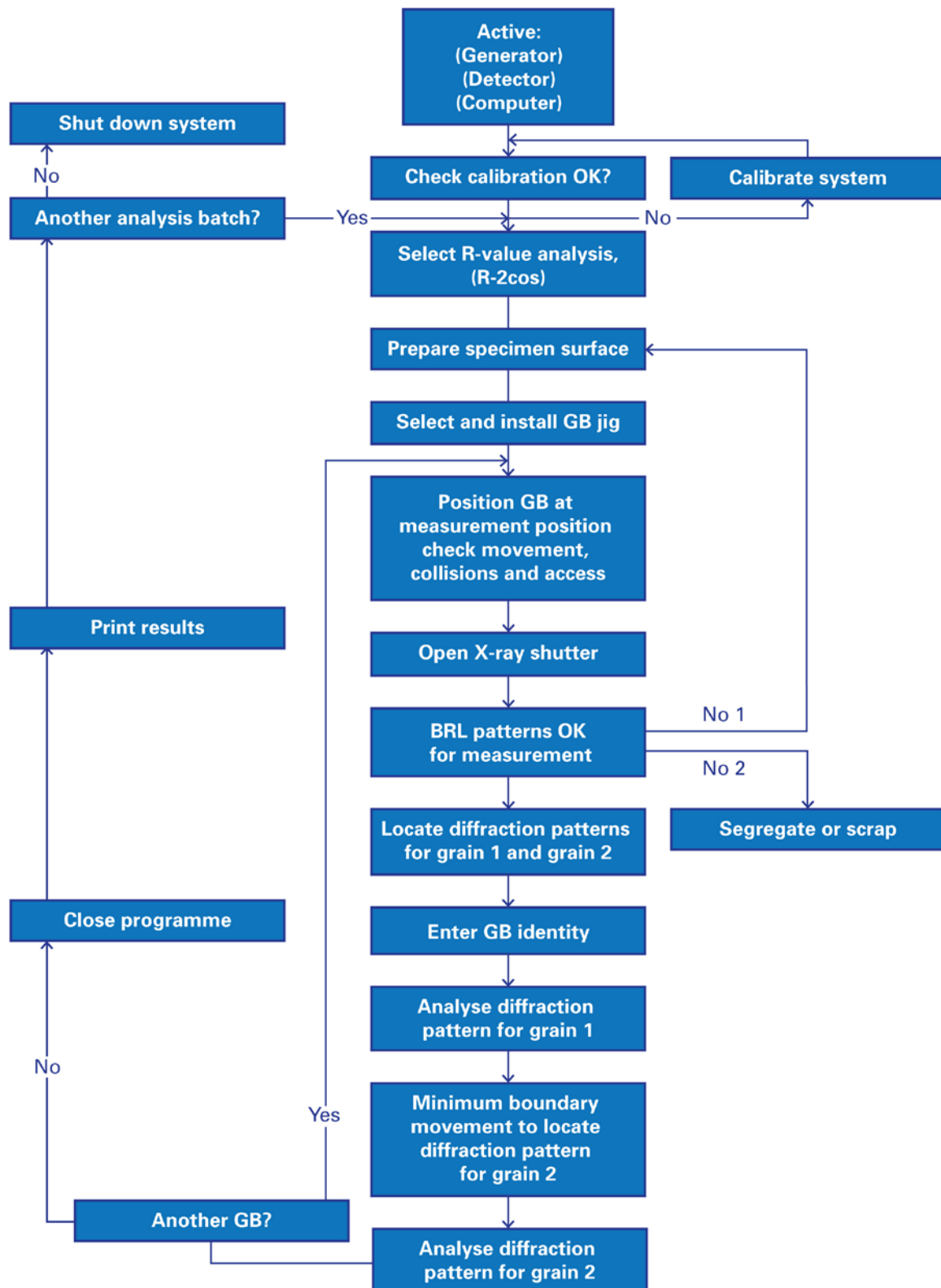


Figure 12.2: R-value measurement flow chart, real-time systems. Note: Flow chart only covers measurement steps, not component quality assurance decisions.

Appendix 1: Definition of primary angles (European convention)

Starting from a position with the crystal lying with a $\langle 001 \rangle$ parallel to the reference direction, RD, and a crystal $\{100\}$ aligned parallel with the reference plane, RP.

Note that gamma, delta and alpha are directly measured from the Laue pattern. See table 2 for the relationship of theta, kappa and rho to the three measured angles.

When viewing towards the measurement face, along the component reference direction:

GAMMA (γ): is a rotation of the crystal about an $\langle 001 \rangle$ axis lying within the reference plane and perpendicular to the reference direction. Rotation is measured from the reference plane, positive being defined as an upward tilt when viewing the examined face.

Gamma range: *from 0° to $\pm 45^\circ$, or from 0° to $\pm 54.7^\circ$, depending on alpha value.*

DELTA (δ): is the rotation about the normal to the plane containing both the $\langle 001 \rangle$ nearest to the reference direction and the $\langle 001 \rangle$ direction lying within the reference plane perpendicular to the reference direction. Rotation is measured from the plane which contains the reference direction, to the $\langle 001 \rangle$ direction closest to the reference direction, positive being defined as a tilt to the left when viewing the examined face.

Delta range: *from 0° to $\pm 45^\circ$, or from 0° to $\pm 54.7^\circ$, depending on alpha value.*

THETA (θ): is the deviation, regardless of direction, from the reference direction to the $\langle 001 \rangle$ direction.

Theta range: *0° to $\pm 54.7^\circ$.*

ALPHA (α): is the clockwise rotation about the reference direction, measured from the reference plane to the nearest $\{001\}$ plane passing through the $\langle 001 \rangle$ direction nearest to the reference direction, when viewing the examined face.

Alpha range: *from 0° to 60° , or from 0° to 120° , depending on gamma and delta values.*

KAPPA (κ): is the clockwise rotation about the $\langle 001 \rangle$ direction nearest to the reference direction, measured from the reference plane to the nearest $\langle 001 \rangle$ direction when viewing the examined face.

Kappa range: *0° to 90° .*

RHO (ρ): is the rotation about the $\langle 001 \rangle$ direction nearest to the reference direction, from a plane containing both these directions to the nearest $\langle 001 \rangle$ direction. (The rotation can be either clockwise or anti-clockwise when viewing the examined face, but it is always in the direction that makes RHO less than 45°).

Rho range: *0° to 45° .*

Appendix 2: Definition of primary angles (US convention)

Starting from a position with the crystal lying with a $\langle 001 \rangle$ parallel to the reference direction, RD, and a crystal $\{100\}$ aligned parallel with the reference plane, RP.

Note that gamma, delta and beta are directly measured from the Laue pattern. Alpha may be derived from these three rotations.

When viewing away from the measurement face, towards the X-ray source and along the component reference direction:

GAMMA (γ): is a rotation of the crystal about an $\langle 001 \rangle$ axis lying within the reference plane and perpendicular to the reference direction. Rotation is measured from the reference plane, positive being defined as an upward tilt when viewing away from the examined face (towards the X-ray source).

Gamma range: *from 0° to $\pm 45^\circ$, or from 0° to $\pm 54.7^\circ$, depending on beta value.*

DELTA (δ): is the rotation about the normal to the plane containing both the $\langle 001 \rangle$ nearest to the reference direction and the $\langle 001 \rangle$ direction lying within the reference plane perpendicular to the reference direction. Rotation is measured from the plane which contains the reference direction, to the $\langle 001 \rangle$ direction closest to the reference direction, positive being defined as a tilt to the right when viewing away from the examined face (towards the X-ray source), *i.e.* a tilt to the left when viewing the examined face.

Delta range: *from 0° to $\pm 45^\circ$, or from 0° to $\pm 54.7^\circ$, depending on beta value.*

ALPHA (α): is the deviation, regardless of direction, from the reference direction to the $\langle 001 \rangle$ direction.

Alpha range: *0° to $\pm 54.7^\circ$.*

BETA (β): is the clockwise rotation about the reference direction, measured from the reference plane to the nearest $\{001\}$ plane passing through the $\langle 001 \rangle$ direction nearest to the reference direction, when viewing away from the examined face (towards the X-ray source), *i.e.* an anticlockwise rotation when viewing the examined face.

Beta range: *from 0° to 60° , or from 0° to 120° , depending on gamma and delta values.*

Appendix 3: Definition of angle omega (additional derived primary orientation angle, European convention)

Omega, ω , is a combination of two of the primary angles, gamma and delta. It is used for specific types of orientation control for some components because it gives a clear description of where the nearest <001> crystal direction is situated within a non-symmetric component cross-section profile.

As shown in Figure A3.1, when a turbine component:

- (a) has its datum plane A assigned as its reference plane, RP, and the intersection of planes A and B assigned as the reference direction and;
- (b) is viewed from the root end and;
- (c) is positioned with its aerofoil *concave* surface *uppermost* (and is swept for clockwise engine rotation) then;

omega, ω , is the angle between the RP and the plane containing both RD and the nearest <001> crystal direction. Omega is *positive* in the *clockwise* sense and is *zero* at the 3 o'clock position (coincident with the trace of RP).

On a stereographic projection, omega is measured either on the small circle (cone) of constant theta or at the circumference (Figure A3.2).

$$\tan \omega = \sin \gamma / \tan \delta$$

The range of omega is 0 – 359.9°.

The above equation must be appropriately restricted.

Table A3.1: Range restrictions for omega, ω

ω range, °	gamma γ	delta δ
0 to 90	-	-
90 to 180	-	+
180 to 270	+	+
270 to 359.9	+	-

In practice, omega, ω , only has any significance when theta, θ , is greater than 5°. It is recommended that for gamma and delta combinations that result in theta values below 5°, a statement such as ‘omega not defined’ should be substituted in place of the omega value.

Note: $\cos \theta = \cos \gamma \cdot \cos \delta$

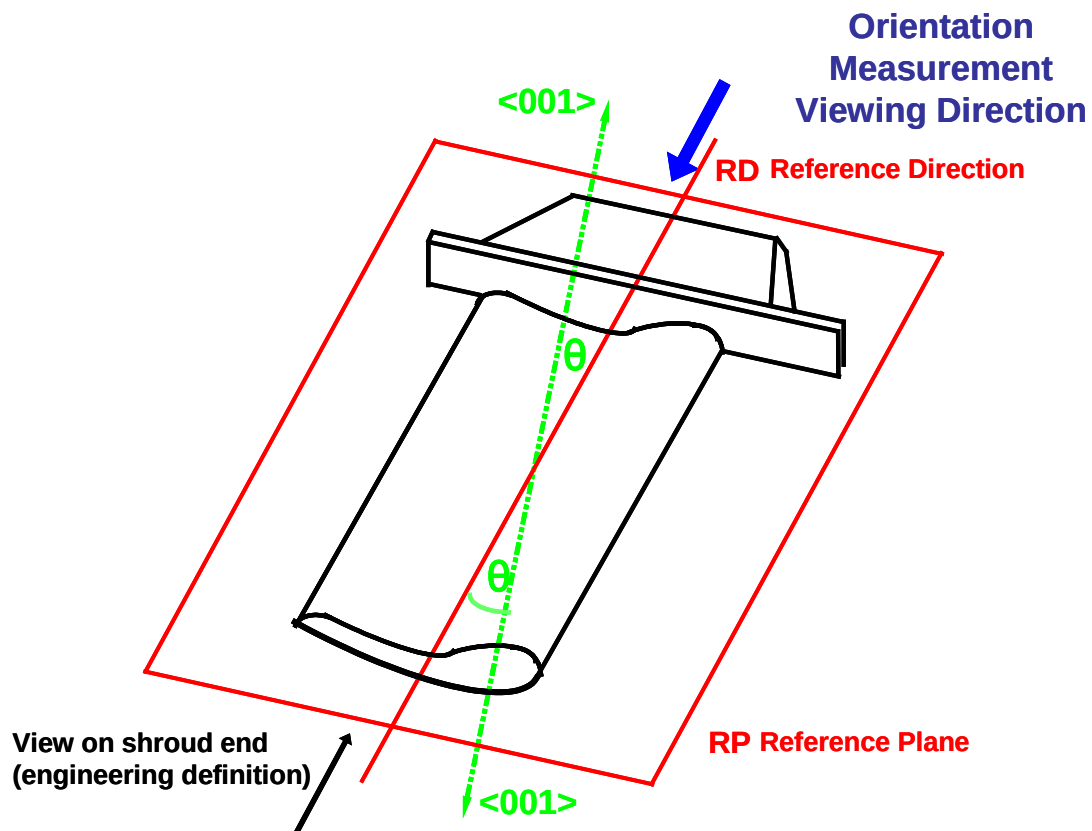


Figure A3.1: Viewing references for angle omega.

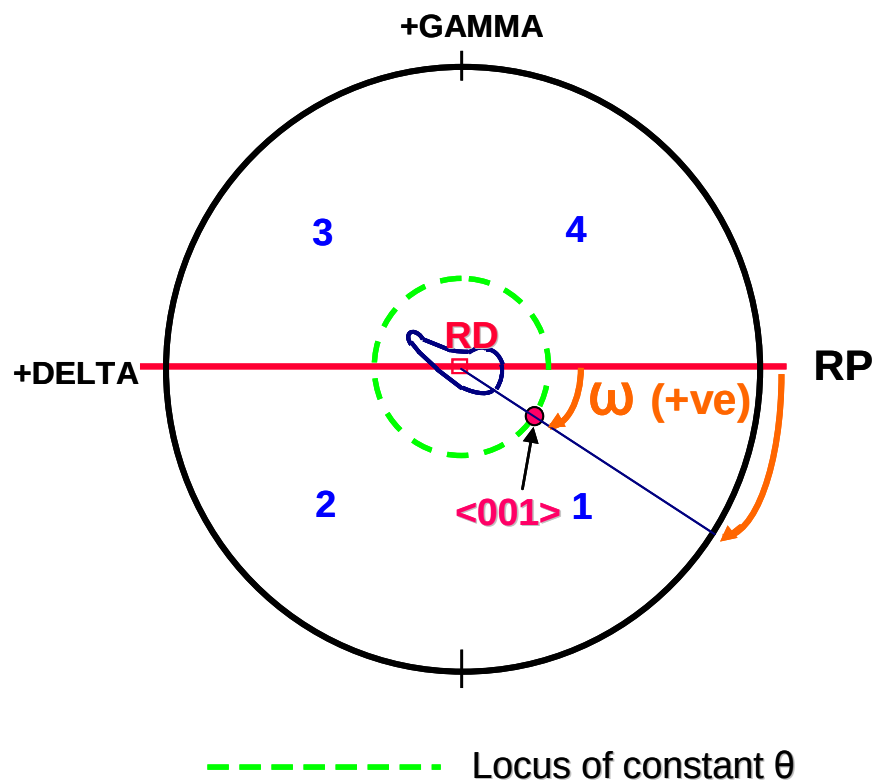


Figure A3.2: Stereographic projection and definition of angle omega. View and measurement on root end (aerofoil concave surface upwards).

Appendix 4: Details of R-value conventions

A4.1 Different R-value definitions

There is no one universally agreed approach to the description of R-value. The mathematically rigorous approach is complex, so often a simplified approach has been taken to allow hand calculation. Such simplified approaches are often sufficiently accurate for small angle differences, but become increasingly inaccurate for larger angles, particularly above about 20°. Table A4.1 lists some of the simple formulae.

Table A4.1 R-value definitions used over the past 30 years (European convention)

Name	Formula
Single-angle cos, (R-1cos)	$R = \text{Minimum of 24 single rotations}$
Two-angle cos, (REL or R-2cos)	$R = \cos^{-1}(\cos \phi \cos \tau)$
Two-angle RMS, (R-2RMS)	$R = \sqrt{(\phi^2 + \tau^2)}$
Three-angle cos, (R-3cos)	$R = \cos^{-1}(\cos(\gamma_A - \gamma_B) \cos(\delta_A - \delta_B) \cos(\alpha_A - \alpha_B))$
Three-angle RMS, (R-3RMS)	$R = \sqrt{(\gamma_A - \gamma_B)^2 + (\delta_A - \delta_B)^2 + (\alpha_A - \alpha_B)^2}$

A4.2 Two-angle, REL, (R-2cos) R-value definition

A4.2.1 Formal definition

PHI (ϕ) and TAU (τ) are the two minimum rotations which, when applied consecutively, transform grain A's orientation to the alignment of grain B.

PHI (ϕ) is the minimum rotation of the primary grain required to align one <001> axes of grain A with an <001> axis of grain B. The axis of the rotation is perpendicular to both of these <001> axes and is therefore the intersection of the two planes defined by these axes.

TAU (τ) is the minimum rotation of grain A, about the aligned pair of <001> axes (one from both A and B grains), to achieve alignment of the remaining two pairs of <001> axes.

Phi and tau are combined to generate the R-value, R:

$$R = \arccos(\cos \phi \cdot \cos \tau)$$

A visual representation of the rotations is shown in Figure A4.1 their illustration on a stereographic projection is shown in Figure A4.2.

A fuller description of the calculations of phi, tau and the R-Value from directly measured orientation angles is given in A4.1.2.

A4.1.2 Calculation method

Assumptions:

- (i) The directly measured orientation angles, gamma, γ , delta, δ and alpha, α , are available for both grain A and grain B.
- (ii) The value κ_{TRUE} is calculated for each grain, where

$$\tan \kappa_{\text{TRUE}} = \cos \gamma \cdot \tan \alpha / (\cos \delta - \sin \gamma \cdot \sin \delta \cdot \tan \alpha)$$

Calculation Stages:

(a) Setting up direction cosine vectors

For each grain, define the direction cosine vector for all (six) $\langle 001 \rangle$ crystal directions in terms of gamma, delta and κ_{TRUE} . (See note 1 below).

(b) Finding the minimum value of phi (ϕ)

Use the direction cosine vector property for finding the angle between two vectors (see note 2 below) to calculate all angles between every $\langle 001 \rangle$ direction of grain A and each $\langle 001 \rangle$ direction within grain B (with 6 vectors for each grain there are 36 combinations but consideration of symmetry results in 18 possible permutations).

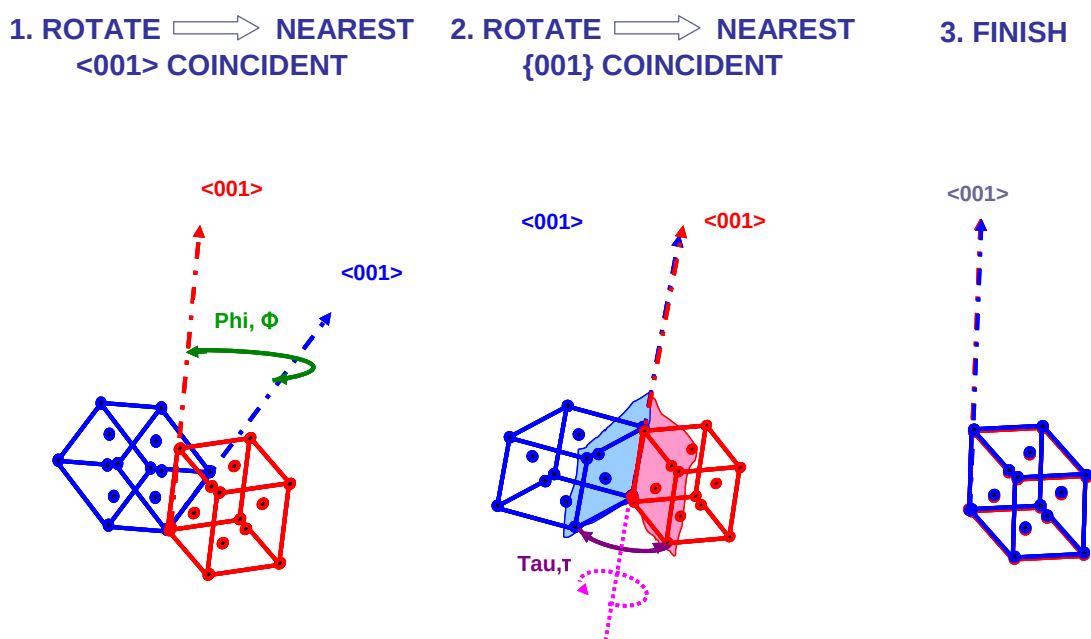


Figure A4.1: Visual representation, phi and tau ($R=2\cos$).

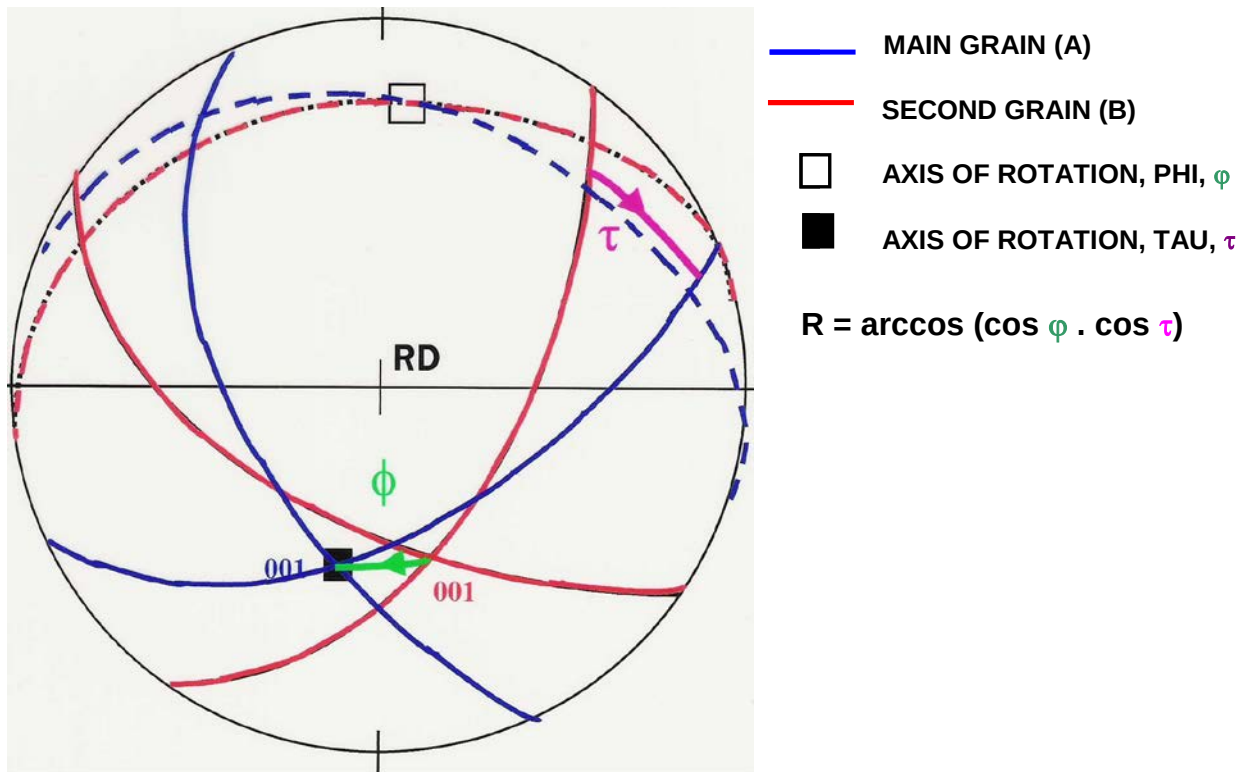
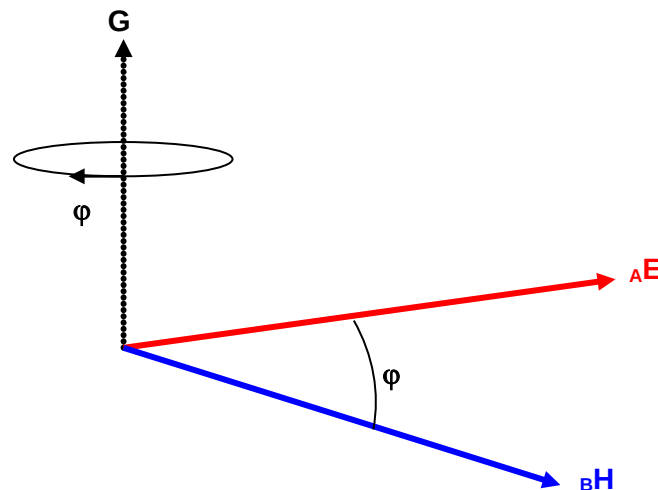


Figure A4.2: Stereographic representation R -value $R=2\cos$.

The lowest value of ϕ is selected, between vectors that shall be denoted ${}_A\mathbf{E}$ and ${}_B\mathbf{H}$, where ${}_A\mathbf{E}$ is an $\langle 001 \rangle$ crystal axis from grain A and ${}_B\mathbf{H}$ is an $\langle 001 \rangle$ crystal axis from grain B.

(c) Finding the axis of rotation for ϕ



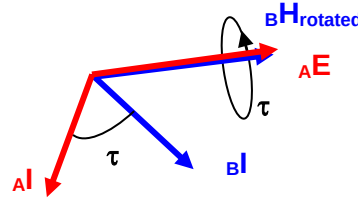
Let $\mathbf{G}$ be the normal to a plane defined by ${}_A\mathbf{E}$ and ${}_B\mathbf{H}$: $\mathbf{G}$ can be calculated by another direction cosine vector property (see note 3 below).

(d) Applying phi to grain 2

Grain A remains static and a rotation of ϕ around axis **G** is applied to grain B only.

The transformed (rotated) vector is only required for one other $\langle 001 \rangle$ axis in grain B that is orthogonal to **BH**. This transformed vector is denoted **BI**.

Stages (c) and (d) entail solutions of simultaneous vector equations and associated consistency checks for minimum values.

**(e) Calculating a value for tau (τ_{nominal})**

The axis for the second rotation, tau (τ_{nominal}) is the common axis of both grains **AE** and **BH_{rotated}**. τ_{nominal} is the angle between two $\langle 001 \rangle$ axes, one from each grain, in a plane normal to the common axis, *i.e.* **BI** from grain B and any $\langle 001 \rangle$ axis from grain A that is orthogonal to **AE**: it is randomly selected from the four possible options and is denoted **AI**.

A possible angle for τ is the angle between these two axes **AI** and **BI**: the vector cosine property for calculating the angle between 2 axes is used (see note 2 below).

(f) Finding the minimum value of τ

The following rules are applied, in the order of precedence shown, to τ_{nominal} to derive the minimum value for τ .

- (i) If $(\tau_{\text{nominal}}) < 0$, then $\tau = \tau_{\text{nominal}} + 90$
- (ii) If $(\tau_{\text{nominal}}) > 90$, then $\tau = \tau_{\text{nominal}} - 90$
- (iii) If $(\tau_{\text{nominal}}) > 0$, then $\tau = 90 - \tau_{\text{nominal}}$

(g) Calculating the R-value (REL, R-2cos)

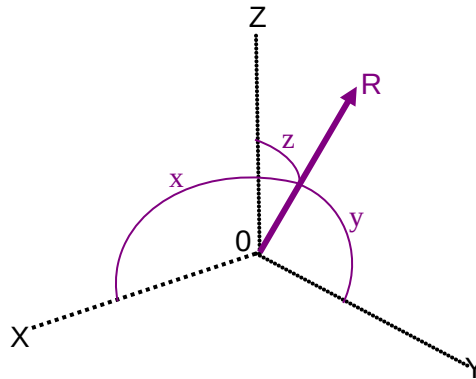
Using the minimum values derived above for phi and tau,

$$R = \arccos (\cos\phi \cdot \cos\tau)$$

Notes for R-Value calculation stages

1. Defining direction cosines

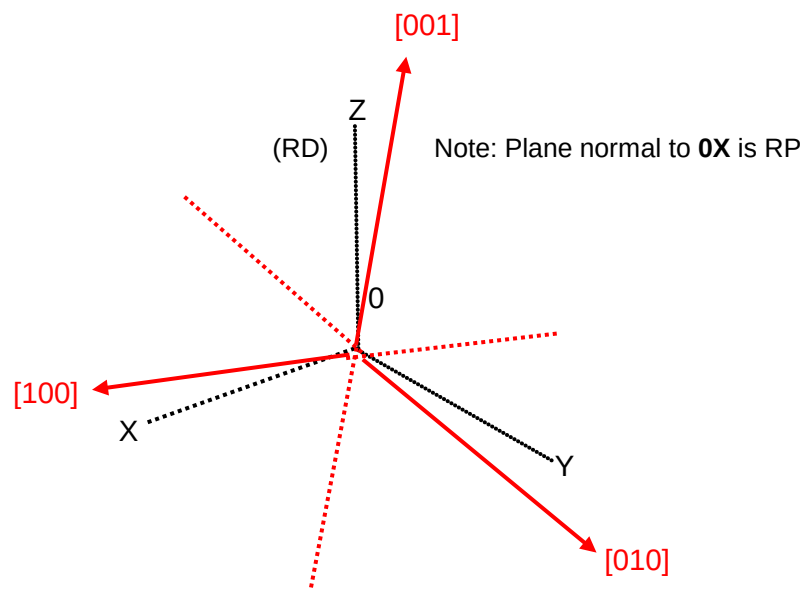
For any XYZ set of axes with an origin 0 and a vector **OR** then the directional cosines for **OR**, then its directional cosines are defined as the cosines of the three angles (x, y, z) made by **OX**, **OY** and **OZ** with vector **OR**, as shown below.



If the direction cosines are a_1 , a_2 and a_3 , then $a_1^2 + a_2^2 + a_3^2 = 1$.

Within the following definition of direction cosines for crystal $\langle 001 \rangle$ axes, the symbol K is used through to denote the parameter K_{TRUE} .

A face-centre cubic crystal is defined by its $\langle 001 \rangle$ axes, $[100]$, $[010]$ and $[001]$. When not aligned parallel with the XYZ reference system each of these $\langle 001 \rangle$ axes (vectors) will have a set of direction cosines.



If the alignment (orientation) is defined by primary orientation angles gamma, γ and delta, δ and the calculated parameter K, then these direction cosines are:

[100]:

$$\cos \delta \cdot \cos \kappa \quad \cos \gamma \cdot \sin \kappa - \sin \gamma \cdot \sin \delta \cdot \cos \kappa \quad -\sin \gamma \cdot \sin \kappa - \cos \gamma \cdot \sin \delta \cdot \cos \kappa$$

[010]:

$$-\cos \delta \cdot \sin \kappa \quad \cos \gamma \cdot \cos \kappa + \sin \gamma \cdot \sin \delta \cdot \sin \kappa \quad -\sin \gamma \cdot \cos \kappa + \cos \gamma \cdot \sin \delta \cdot \sin \kappa$$

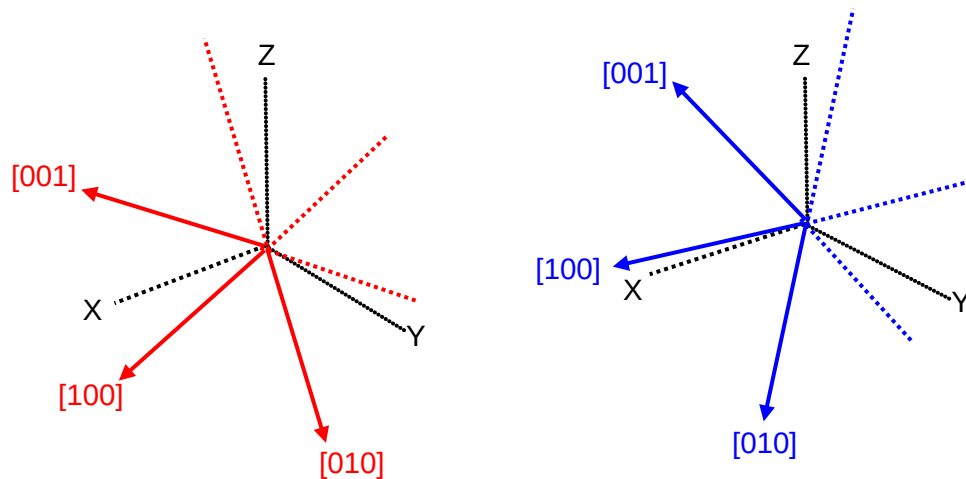
[001]:

$$\sin \delta \quad \sin \gamma \cdot \cos \delta \cdot \quad \cos \gamma \cdot \cos \delta$$

Further, an analogous set of direction cosines exist for the opposite crystal directions, *i.e.*

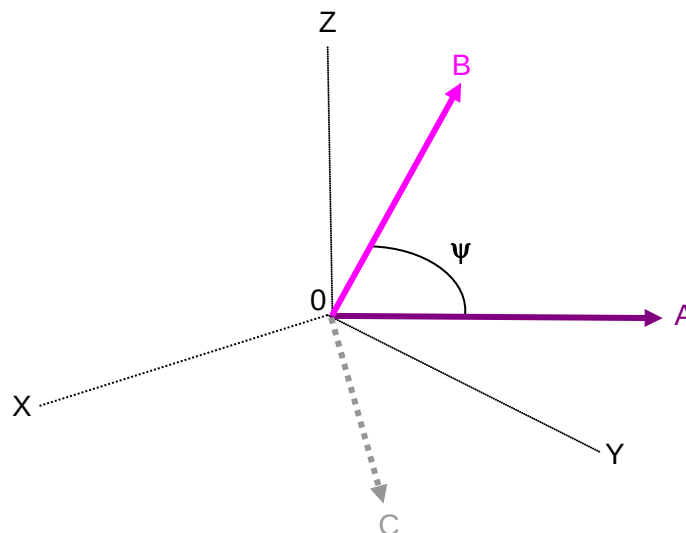
[-100], [0-10] and [00-1].

With two grains adjacent to a boundary, grains A (red) and B (blue), each grain will have six sets of direction cosines.



2. Vector property: finding the angle between two vectors.

If two vectors **0A** and **0B** with direction cosines of (**a₁ a₂ a₃**) and (**b₁ b₂ b₃**) respectively:



The angle between the two vectors, ψ , can be calculated directly from the direction cosines of **0A** and **0B**.

$$\cos \psi = a_1 b_1 + a_2 b_2 + a_3 b_3$$

3. Vector property: finding the normal to a plane containing two vectors.

The two vectors **0A** and **0B** with direction cosines of $(a_1 \ a_2 \ a_3)$ and $(b_1 \ b_2 \ b_3)$ respectively define a plane. If the normal to this plane is vector **0C** with direction cosines $(c_1 \ c_2 \ c_3)$, then these direction cosines can be calculated from the direction cosines of **0A** and **0B**:

$$c_1 = (a_2 b_3 - a_3 b_2) / J$$

$$c_2 = (a_3 b_1 - a_1 b_3) / J$$

$$c_3 = (a_1 b_2 - a_2 b_1) / J$$

where

$$J = \sqrt{((a_2 b_3 - a_3 b_2)^2 + (a_3 b_1 - a_1 b_3)^2 + (a_1 b_2 - a_2 b_1)^2)}$$

A4.2 Single-angle (R-1cos) R-value definition

A4.2.1 Formal definition

A vector (axis) and angle are both defined such that a rotation by the angle about the vector, bring the lattices of the two grains, A and B, into coincidence. The angle axis pair selected must be the minimum of the 24 different possibilities. The R-value is the magnitude of this minimum angle.

A4.2.2 Calculation method summary

The method for misorientation angle axis pair determination is detailed in [4] (pp. 31 – 36): the minimum value of the angle, the disorientation is defined in [4].

Appendix 5: Assessment of SSCS diffraction pattern symmetry

The overall symmetry of the reference pattern from the silicon single-crystal standard (SSCS) can be used to gauge the co-planar alignment of the critical system axes, XA, SA and DPN. Different types of pattern asymmetry are typical of systematic non-coplanar conditions (as described in Section 6.4). The reference pattern must be obtained with the SSCS securely positioned in its appropriate fixture at the system working distance, S .

A5.1 Horizontal axis asymmetry

The following descriptions are illustrated in Figure A5.1:

- (i) On the vertical (001) zone, identify a pair of equivalent ($hk0$) diffraction poles, one of the pair on each side of the central 001 pole. Check, via measurement, the distance of each from the 001 pole. Record which pole in the pair (top or bottom) is at the greater distance from the 001 pole.
- (ii) Select other pairs of equivalent ($hk0$) diffraction poles on the same vertical (001) zone and repeat the assessment (i).
- (iii) For other (non-vertical) zones, select a pair of equivalent (hkl) diffraction poles, one of the pair on each side of the central 001 pole. Check, via measurement, the distance along a vertical construction line, the distance of each from the line of the horizontal 001 zone. Record which pole in the pair (top or bottom) is at the greater distance from the 001 zone: this can be repeated for other (hkl) pole pairs on the selected and other zones.

If the data from the above checks consistently show that the top poles are either closer to or further from the 001 pole (or zone), the pattern does not have horizontal axis symmetry.

Note: Step (iii) may not be necessary to establish asymmetry or otherwise.

A5.2 Vertical axis asymmetry

The following descriptions are illustrated in Figure A5.2:

- (i) On the horizontal (001) zone, identify a pair of equivalent ($hk0$) diffraction poles, one of the pair on each side of the central 001 pole. Check, via measurement, the distance of each from the 001 pole. Record which pole in the pair (left or right) is at the greater distance from the 001 pole.
- (ii) Select other pairs of equivalent ($hk0$) diffraction poles on the same horizontal (001) zone and repeat the assessment (i).
- (iii) This step ONLY applies to true BRL geometry systems. For other (non-horizontal) zones, select a pair of equivalent (hkl) diffraction poles, one of the

pair on each side of the central 001 pole. Check, via measurement, the distance along

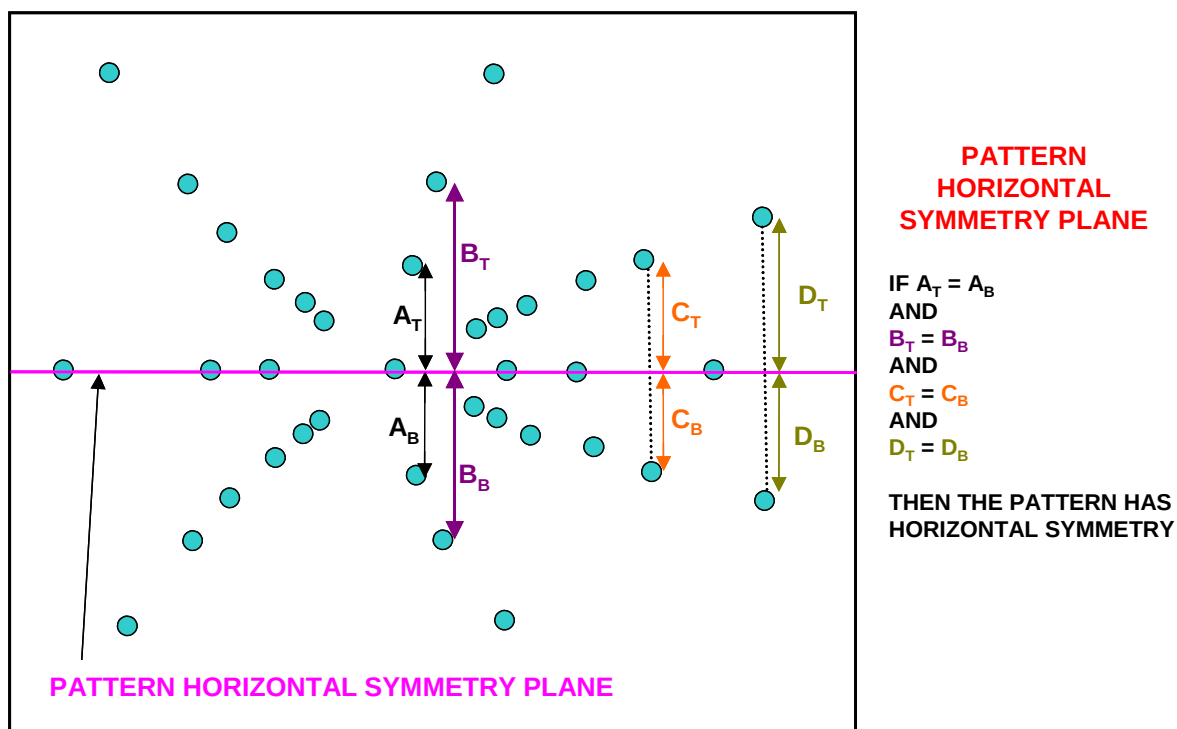


Figure A5.1: Assessment of the horizontal symmetry of a silicon single-crystal standard diffraction pattern.

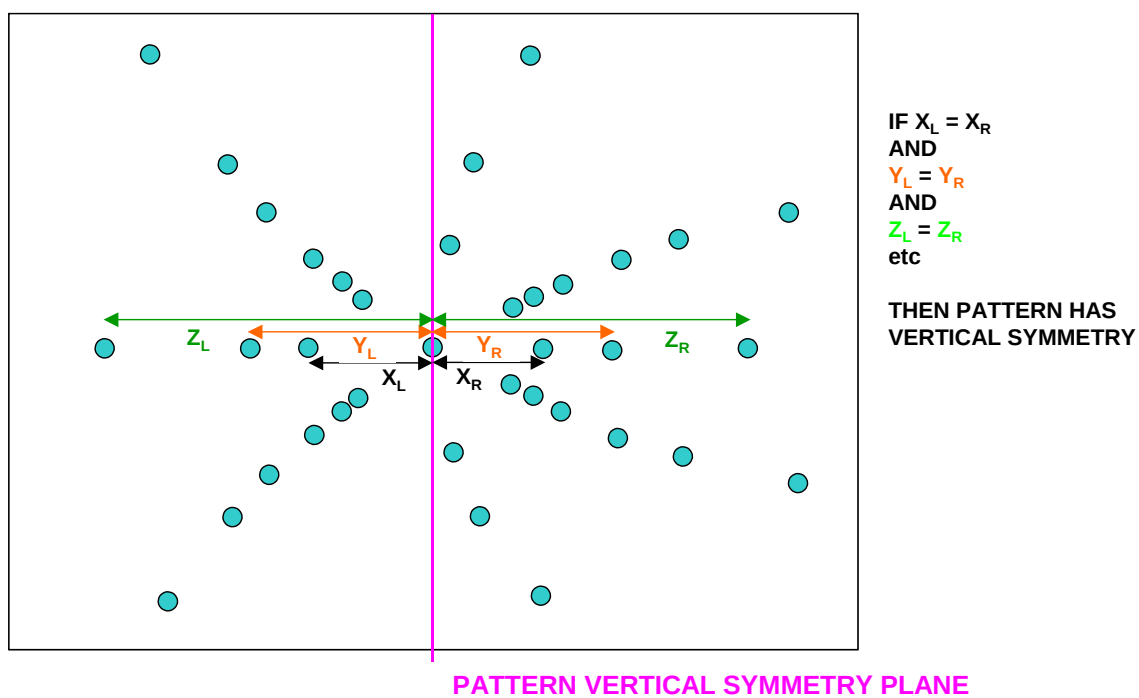


Figure A5.2: Assessment of the vertical symmetry of a silicon single-crystal standard diffraction pattern.

a horizontal construction line, the distance of each from the line of the vertical 001 zone. Record which pole in the pair (left or right) is at the greater distance from the 001 zone. This can be repeated for other (*hkl*) pole pairs on the selected and other zones.

If the data from the above checks consistently show that the left poles are either closer to or further from the 001 pole (or zone), the pattern does not have vertical axis symmetry.

A5.2 Practical considerations

Practical methods for completing the asymmetry checks depend on the image collection and pattern analysis capabilities of a system.

For systems without a pattern overlay, this will need to be completed by ‘manual’ measurements on the film / image, as directly described above. For systems that use a calculated pattern overlay, the asymmetry assessment can be deduced from the fit trends in either half of the pattern (top / bottom and left / right, for horizontal and vertical symmetry respectively) when the overlay is positioned on the 001 observed pattern centre and its horizontal 001 zone is aligned with that of the SSCS diffraction pattern.

Appendix 6: Details of system validation parameters

A6.1 Examples of validation specimen data sets

The examples given are the results of evaluation of specimen sets used in a round robin conducted in the Studio SCOMS Project, NPL, 2007-2009.

Note: The total number of specimens in each set, m , is 8.

The number of repeat measurements, n , is 5.

The number of different facilities included in comparisons is p .

A6.1.1 Primary orientation values

The following tables show the global average result for each test-piece, together with the typical within-laboratory standard deviation s_r and the between-laboratory standard deviation s_R . It can be seen that the s_R is typically twice s_r or larger.

Specimen	Gamma			Delta		
	Average	s_r	s_R	Average	s_r	s_R
1	1.2	0.13	0.19	1.4	0.29	0.37
2	-7.9	0.18	0.41	-2.1	0.21	0.37
3	11.4	0.10	0.23	9.9	0.13	0.27
4	-7.4	0.13	0.60	-18.7	0.13	0.59
5	25.5	0.07	0.50	-9.4	0.07	0.50
6	-34.2	0.14	0.36	-16.2	0.14	0.35
7	-20.4	0.19	0.85	-36.5	0.61	0.85
8	46.9	-	-	15.4	-	-

Specimen	Theta			Alpha		
	Average	s_r	s_R	Average	s_r	s_R
1	1.9	0.28	0.34	8.6	0.58	0.79
2	8.1	0.19	0.39	7.1	0.48	1.03
3	14.4	0.11	0.27	79.6	0.33	1.04
4	20.0	0.17	0.37	52.9	0.40	0.99
5	27.1	0.09	0.40	56.9	0.34	0.42
6	37.3	0.20	0.31	31.1	0.29	0.85
7	41.3	0.21	0.23	57.2	0.30	0.95
8	49.1	-	-	48.5	-	-

Note: The European angle convention has been used.

A6.1.2 R-values.

The following tables show the global average result using the R-2cos convention for each test-piece, together with the typical within-laboratory standard deviation s_r and the between-laboratory standard deviation s_R . It can be seen that the s_R ranges from a value similar to s_r to rather larger values.

Specimen	R-value		
	Average	s_r	s_R
1	3.3	1.16	1.35
2	7.1	0.38	0.38
3	10.8	0.52	0.53
4	12.7	0.69	0.74
5	22.9	0.33	0.37
6	33.9	0.44*	0.70
7	43.9	0.42*	1.79
8	49.9	0.46*	2.40

* Some variation with measurement positions on the specimens

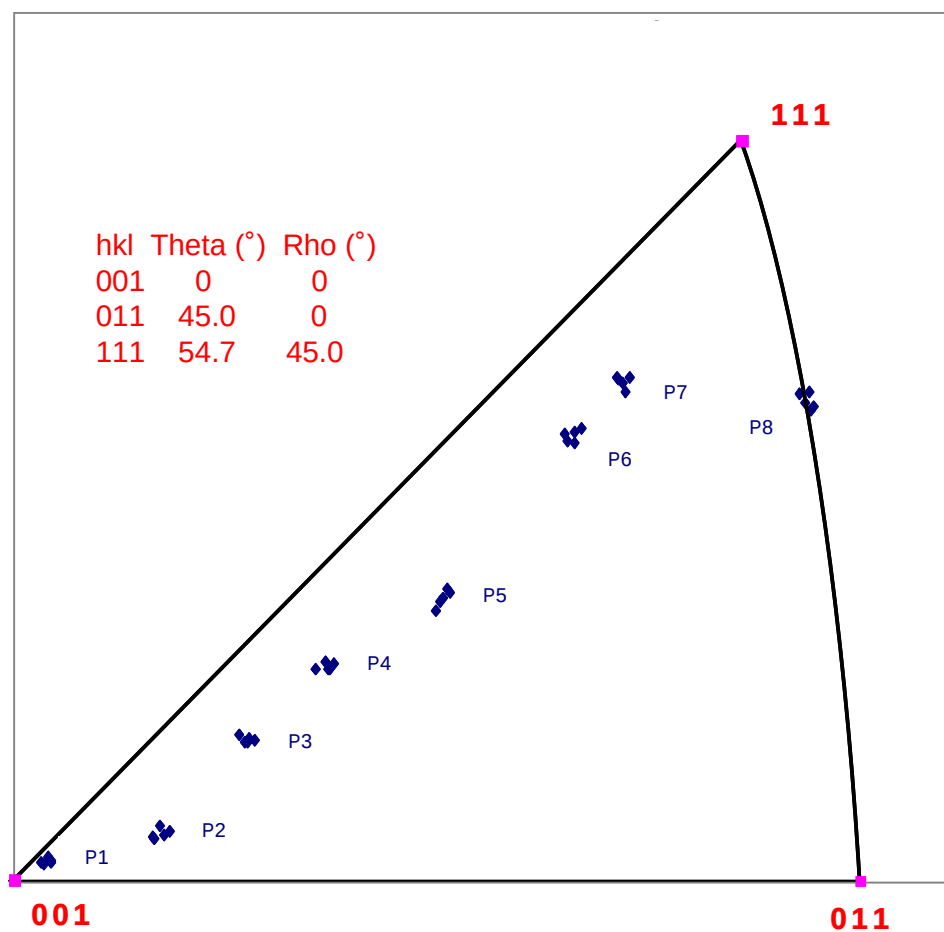


Figure A6.1: Range of values – sample primary orientation measurement set.

A6.2 Multiple facility comparison (data pooling)

The following is a summary of the statistics used in ASTM E691-05 ‘Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method’. ASTM International, 2005, Vol. 5.

The total number of specimens in each set, m , is 8.

The number of repeat measurements, n , is 5.

The number of different facilities included in comparisons is p .

Average of the test results, $\bar{x} = \sum_{i=1}^n x_i / n$

Standard deviation, (σ_n) or $s = \sqrt{\sum_{i=1}^n (x_i - \bar{x})^2 / (n-1)}$

The ‘mean of the means’, $x^* = \sum_{i=1}^p \bar{x}_i / p$

The ‘cell deviation’, $d = \bar{x} - x^*$

Standard deviation if the cell averages, $s_{x^*} = \sqrt{\sum_{i=1}^p d_i^2 / (p-1)}$

Repeatability standard deviation, $s_r = \sqrt{\sum_{i=1}^p s_i^2 / p}$

Reproducibility standard deviation, $s_R = \text{the larger of } s_r \text{ and } \sqrt{((s_{x^*})^2 + (s_r)^2 (n-1) / n)}$

Between system consistency statistic, $h = d / s_{x^*}$

Within system consistency statistic, $k = s / s_r$

Tables within [6] are used to find the 0.5% significance levels for both h and k .

95% repeatability limit, $r = 2.8s_r$.

95% reproducibility limit, $R = 2.8s_R$.

A6.3 Nominated master facility (datum system)

A6.3.1 Measurement system precision (or uncertainty)

The system bias for a measurement system shall be defined by the maximum observed precision. This parameter is required for each orientation parameter (primary and R-value).

For each specimen the standard deviation (σ_n) for the orientation parameter shall be calculated:

- (i) For each specimen, the precision shall be derived for each angle (95% confidence level) as $\pm 2 \sigma_n$.
- (ii) The maximum observed precision for the system for the orientation parameter shall be calculated (based on the rules of combined variance) from the standard deviation (σ_n) values of all m specimens:

$$\sigma_{\text{combined}} = \frac{1}{n} \sum \sigma_n^2$$

$$\text{Maximum observed precision} = \pm 2 \sigma_{\text{combined}}$$

For a fuller description of combined uncertainty statistics, the reader is referred to [7].

A6.3.2 Measurement system bias

The measurement system bias shall be defined by the maximum calculated bias (bias_{max}) for the subject system relative to the datum orientation measurement system. This parameter is required for each orientation parameter (primary and R-value):

- (i) For each specimen within each data set the mean value shall be calculated for each orientation parameter;
- (ii) For each specimen and each orientation parameter the bias between the systems shall be derived as follows:

$$\text{Mean (subject system)} - \text{Mean (datum orientation measurement system)}.$$

- (ii) The maximum calculated bias (to a 95% confidence level) for the subject system shall be derived for each orientation parameter as follows:
 - (a) Derive the mean bias value of all the m specimens, $\text{mean}_{\text{bias}}$;
 - (b) Derive the standard deviation within the bias values for the m specimens, σ_{bias} ;
 - (c) If $\text{mean}_{\text{bias}}$ is positive, or equal to zero, then:

$$\text{bias}_{\text{max}} = \text{mean}_{\text{bias}} + 2 \cdot \sigma_{\text{bias}}.$$

- (d) If $\text{mean}_{\text{bias}}$ is negative, then:

$$\text{bias}_{\text{max}} = \text{mean}_{\text{bias}} - 2 \cdot \sigma_{\text{bias}}.$$

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