

**A NATIONAL MEASUREMENT
GOOD PRACTICE GUIDE**

No. 106

Quantifying Microstructural
Heterogeneity in Hot
Compression Testpieces

Measurement Good Practice Guide No. 106

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Good Practice Guide for Quantifying Microstructural Heterogeneity in Hot Compression Testpieces

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ABSTRACT

This document describes good practice for measurements of material microstructure in hot compression testpieces such as AC (axisymmetric) and PSC (plane strain). It is applicable to hot (isothermal) compression tests at medium to high rates of strain (10^{-4} to 10^2 s⁻¹) at deformation temperatures below the solidus. Technical input to the document has been provided by a steering group containing academic researchers and representatives of industrial users and producers of a range of engineering materials. An experimental and modelling programme has been conducted during the preparation of the document to underpin the recommendations in the guide.

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Foreword

IN THIS CHAPTER

- Foreword
- Project Advisory Group (IAG)

Foreword

These guidelines were supported by the Materials Measurements and Processing Programmes, underpinning materials measurement research activities financed by the UK Department of Trade and Industry.

The Guide describes good measurement practice for quantifying heterogeneous microstructures in testpieces for obtaining the hot compression deformation response of metallic materials, at medium to high rates of strain (10^{-4} to 10^2 s $^{-1}$), at deformation temperatures below the solidus. The guidelines recommend procedures to minimise uncertainty in the measurement process. The procedure has been supported by FE modelling work at the IRC for Computer Aided Engineering, University of Wales, Swansea and experimental tests at NPL, the IRC and IMPETUS, the University of Sheffield on Al alloy 5052, Waspaloy (a nickel-base alloy), 316 stainless steel and Ti6Al4V at a range of temperatures and strain rates.

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Rationale

1

IN THIS CHAPTER

- Drivers
- Measurement Framework

1. RATIONALE

The state of internal microstructures is a key parameter that determines material properties. In hot metal working there is a requirement to obtain the required microstructure, first time, whatever the forming process, aided by appropriate models. Large cost and material savings result if expensive processes of trial and error can be avoided. Repeatable and reproducible hot compression tests are needed which can provide validated data for modelling, for process deformation strategies and for correlation with microstructural evolution studies. The required data are generally obtained from one or more of the three most commonly used types of hot deformation test: axisymmetric compression, plane strain compression and torsion. To obtain improvements in the quality of metal components produced by hot deformation processing, the need to optimise and control the microstructure of the component by control of, for example, the working temperature, strain rate and strain path is thus of ever increasing importance.

Currently, software modelling packages can be used to simulate metal working processes such as the manufacture of aeroengine discs and turbine components, and are vital for work scheduling such as defining rolling mill pass sequences to avoid endangering mill components. Their use can result in shorter times to market, increased yields and reduced costs, and ideally tailored properties through microstructural control with guaranteed property levels. Advanced models are available but there is uncertainty over microstructure state parameters and little or no data on the heterogeneity of the microstructure. A greater understanding is needed of the development of microstructure in processing and how this is controlled by deformation parameters.

At present, there are no standards in place for any of these tests. Documents for good practice in obtaining mechanical data in axisymmetric, plane strain compression tests, and torsion tests have already been written that complement this current Guide on Microstructural Measurements [1-3]. The current guidelines are concerned principally with axisymmetric (AC) and plane strain compression (PSC) tests. In due course it may be extended to cover hot torsion tests where the microstructural gradients are even more pronounced. In order to perform tests which provide accurate and reliable data the test conditions must be specified precisely, particularly concerning temperature distributions, temperature measurement, lubrication, testpiece microstructure and strain/displacement measurements. The development of these guidelines have been supported by experimental tests over appropriate strain rate and temperature ranges.

1.1 Background

The mechanical properties of engineering materials are especially influenced by their mean grain size and grain size distribution [4-10]. Standardised methods of measurement of structure are required to ensure that materials with repeatable properties are produced and a link between processing and structure reinforced. The lack of an agreed method to measure the extent of uniformity is clearly a hindrance to optimised use. Subjective assessments are often made from an evaluation by eye of structural images. There is a generic requirement for improved guidelines for the measurement of grain size distribution in a wide range of industrial materials. In both single and multiphase materials the measurement of an average value of the grain size is vital and there are existing and widely used standards for this purpose, such as ASTM E112, E1181 and E1382 (<http://www.metallography.com/grain.htm>).

However, although there are routines in ASTM E1181 for the measurement of bimodality in distribution there is a need to improve measurement practice for size distributions in general.

Some relevant microstructural standards are:

ASTM E112	-	Grain size Heyn - linear Jeffries - circle
ASTM E1181	-	Duplex Grain Sizes
ASTM E1382	-	Grain Size using Image Analysis
ASTM E562	-	Determining Volume Fraction by Systematic Manual Point Count
ASTM E930	-	Largest Grain Size
EN 623-3	-	Advanced Technical Ceramics – Monolithic Ceramics – General and Textural Properties. Part 3: Determination of Grain Size and Size Distribution (Characterised by the Linear Intercept Method).

ASTM E1181 addresses some of the issues associated with grain size distribution for example it discusses various typical examples, such as; isolated coarse grains in a finer grained matrix; extremely wide distributions; bimodal distributions. It also mentions other difficult issues, such as; banded structures, necklace structures and systematic variations across sections of products.

ASTM E1181 also contains comparison charts for estimating area fractions (possibly in multiphase materials). A number of procedures are defined, including a test grid on transparent overlay - superimposed image - estimate area fractions; largest grains; comparison charts, and an intercept method for grain size. The standard advises that the estimation of area fractions of different sizes is subjective and prone to error. The standards recommend that the most effective method for measuring intercept distributions is to use a semi-automatic image analysis system with digitising tablets and electronic pencils/cursors - using a test grid with 5 evenly spaced horizontal lines. The intercepts are classified by size and the data can be presented as a histogram or frequency plot.

The linear intercept technique is thus popular because it produces size distribution information. It is often found that the measured intercept number distribution is lognormal in form, which gives a straight line when plotted on lognormal probability paper [11,12]. Another option is measurement of the sectional grain area, as discussed by Vander Voort and Friel [13] and used effectively by Chakrabarti et al to quantify bimodality in high strength low alloy steels [14], but this depends on the production of suitable images for image analysis. The increasing use of electron backscattered diffraction techniques for imaging grain structures is likely to invigorate this option for grain size measurement since many grains can be potentially be measured more accurately thus reducing the uncertainties associated with area measurements and sampling (see Appendix on EBSD measurements).

The type of distribution measurement to be preferred depends on what effect a specific distribution of sizes has on properties. There is some evidence that volume fraction (size) determines hardness in Powder Metallurgy products [6], and that wider grain size distributions are accompanied by a lower strength [4]. Material structures are generally characterised, apart from their chemical composition, by a size parameter, S . In single phase materials the most commonly used size parameter is grain size, d , which has the dimensions of length. Because structures are usually measured on a two-dimensional polished surface the grain size can be obtained either by counting many intercepts measured on random lines and taking the number average to give an arithmetic mean linear intercept, d_{im} .

$$d_{im} = \frac{\sum d_i}{N} \quad (1)$$

where N is the total number of intercepts and d_i are individual intercept lengths, or counting the number of grain boundary intersects along a line of known length and dividing the length by the number to get an average intercept length. This value in many cases is taken to be the grain size without converting it to a true 3-D grain diameter. Property models are frequently related to the size parameter by power law equations. For example in many metallic materials the Hall-Petch equation can be used to predict the effect of grain size on strength or hardness. When models of these types are compared with microstructural measurements the grain size is frequently taken to be the arithmetic (number average) mean linear intercept, d_m , obtained from a two-dimensional image. But the structures of single-phased materials can be characterised in several ways other than by using a number distribution of d_i . For example, the grain area or volume distributions can be obtained on a size basis, by line, by area fraction or by volume fraction. All these different methods of structure characterisation can have different consequences for the predictive models described above since they lead to different integrals which have to be evaluated. The most common technique is to use a number distribution of size $f_n(S)$; this is frequently found to be lognormal for intercepts [9,15]. However, a different distribution can be developed based on the size fraction $f_s(S)$ rather than number [16]. Size fraction is obtained by dividing the size of interest by the total size and the size fraction distribution is derived by plotting the size fraction against size. Thus, there are six possible distributions, three of number and three of size, based on intercept, area or volume. These distribution functions are

Number	- $f_n(I)$, $f_n(A)$ and $f_n(V)$
Size	- $f_s(I)$, $f_s(A)$ and $f_s(V)$

Some of these distributions (i.e. $f_n(I)$ and $f_n(A)$) can be measured directly but some types of distribution, i.e. $f_n(V)$ and $f_s(V)$, must be calculated from directly measured distributions, usually by making assumptions about grain shape.

Using conventional microscopy to obtain measurements of the areas of grains is more time consuming than measurements of intercepts without fully automated image analysis systems, especially as several hundred grains need to be measured to obtain reasonable distribution data. It is likely that in measurements on real microstructures that short intercept values are under-counted due to difficulties in image resolution and grain rounding. However, if the data are plotted as size probabilities this difference disappears, because clearly the fraction of area calculated from the smaller intercept values is tiny. Thus, if distributions based on size are important then it may not be necessary to measure the small intercepts with any great accuracy.

1.2 Guide Structure

These current guidelines provide a structured approach to enable the potential sources of uncertainty in microstructural measurements to be better understood and controlled. The microstructural guidelines have been integrated with some of the recommendations from the mechanical testing GPGs [1-3] in order that this current GPG can be followed without undue reference to the former. Quantitative finite element (FE) analysis of strain and temperature guidelines in some of the tests were conducted at the IRC in Computer Aided Engineering at the University of Wales, Swansea to investigate the effects of friction, strain rate, temperature, testpiece volume and strain using a fully coupled finite element procedure. The results are presented in a separate section within the Guide and used, in particular, to interpret optical metallography studies reported in Appendix C.

Colleagues at the IRC in Computer Aided Engineering prepared a summary of methods for characterising metallographic parameters and this is included in Appendix A in a condensed form.

Several case studies were performed on AC and PSC testpieces to generate underpinning information on structural heterogeneity and are discussed in appropriate Appendices. Optical, SEM and EBSD studies were conducted to obtain suitable data. These results were compared with the FE studies of strain and temperature distributions in representative testpiece geometries. EBSD was used quite intensively within the project and detailed background to the technique is given in Appendix B. A new ISO standard on the EBSD method is being developed by ISO/TC 202 (Microbeam Analysis) and further information can be obtained from national representatives appropriate to the ISO/TC 202 committee (ISO/TC 202 N137 ISO/WD). Also ASTM committee E04 (Metallography) is developing a standard for the measurement of grain size by EBSD (ASTM WK3577).

Making detailed microstructural studies over large surface areas of metallographic samples is costly in time and effort. To provide some insight into likely variations in microstructure a new, rapid, method of scanning the surface was applied before undertaking extensive metallography. This method was based on the use of a scanning microhardness test system [17]. Typical results are discussed in Appendix D.

Scope

2

IN THIS CHAPTER

- Scope
- Conditions of Testing

2 Scope

- 1.1 These guidelines are intended for use with metallic materials.
- 1.2 The guidelines specifically apply to “isothermal” plane strain (PSC) and axisymmetric compression (AC) tests on testpieces of a specified geometry. Further research is needed before recommendations can be given for torsion tests.
- 1.3 These guidelines apply to deformation strain rates in the range 10^{-4} to 10^2 s^{-1} , relevant to industrial forging and rolling processes.
- 1.4 These guidelines apply to hot metal working deformation temperatures below the solidus of the material in question.
- 1.5 The guidelines make specific recommendations for the measurement of grain size and other microstructural parameters and their variation in suitable testpieces.

Notes

- (1) *The guidelines have been supported by finite element modelling studies at the University of Wales, Swansea (IRC in Computer Aided Engineering) where deformation conditions were systematically changed to examine their effect on strain and temperature distribution.*
- (2) *The guidelines were also supported by experimental measurements of microstructural gradients at the University of Wales, Swansea (IRC in Computer Aided Engineering), University of Sheffield (IMMPETUS) and NPL on an Al alloy, AA5052; on 316 stainless steel, a nickel alloy, Waspaloy and Ti6Al4V, over a range of temperatures and strain rates.*

These guidelines for conducting microstructure measurements in association with mechanical tests contain a number of basic steps:

- a. Manufacture testpiece to chosen geometry.
Decide on testpiece values for each parameter.
- b. Choice of test system for measurements of load, displacement and temperature.
- c. Installation of testpiece in system.
Choose heating profile, and lubrication type.
- d. Deform testpiece at specified temperature and strain rate.
Measure changes in load and displacement (speed) and temperature.
- e. Remove testpiece from system; quench or slow cool in an agreed predetermined manner.
- f. Prepare testpiece for microstructural measurements.
- g. Conduct quantitative measurements of microstructure.
- h. Analyse and report data, including estimates of uncertainties.

Symbols and Units

3

IN THIS CHAPTER

- Symbol Definitions

3 Symbols and Units

For the purpose of these guidelines the following nomenclature shall apply:

Symbol	Designation	Units
d_{im}	Mean linear intercept grain size	μm
d_i	Linear intercept	μm
d_{ceq}	Circle equivalent diameter grain size	μm
V_f	Phase volume fraction	
V_R	Volume fraction recrystallised	
d_i^1	Mean linear intercept (phase 1)	μm
d_i^2	Mean linear intercept (phase 2)	μm
d_i^n	Mean linear intercept (phase n)	μm
V_f^a	Volume fraction (phase a)	
V_f^b	Volume fraction (phase b)	
V_f^n	Volume fraction (phase n)	
V_R	Volume fraction recrystallised	
A_f^a	Area fraction (phase a)	
P_p	Point fraction	
A_R	Area fraction recrystallised	
L_T	Line length	mm
S	Standard deviation	
CV	Coefficient of variation	
CL	Confidence limit	
RA	Relative accuracy	
n	Number of fields	
G	ASTM Grain Size Number	
N	Number of intercepts	
N_A	Number of grains per unit area	
N_L	Number of intercepts per unit length	
M	Magnification	
E	Proportional variance	

Definitions

4

IN THIS CHAPTER

- Terminology and Definitions

4 Definitions

4.1 Stress and Strain

Determination of the state of stress and strain is necessary for the analysis of plastic deformation in metals and subsequent correlation with microstructural parameters. The state of stress at a point can be determined by calculating the stresses acting on three mutually perpendicular oriented planes passing through the point. When determining the complete state of stress at a point, O, it is found that on one plane the normal stress increases as the angle of the plane is increased until a maximum is reached, whilst the shear stress on the same plane decreases to zero. This plane of maximum normal stress and zero shear stress is called the Principal Plane and the normal stress acting on the plane, the Principal Stress. There are also Principal Strains associated with these stresses. Thus the stress system at a point can be represented by three principal stresses, σ_1 , σ_2 , and σ_3 , which control the yielding behaviour of the metal and which give rise to the associated principal strains ϵ_1 , ϵ_2 and ϵ_3 [1,2].

4.2 Testpiece Geometry and Orientation

Detailed recommendations on the testpiece characterisation and definitions for AC and PSC hot compression tests are given in the complementary GPGs on mechanical testing metals [1,2]. Height and diameter are critical for the AC test; specimen breadth, height and width and platen width and length are critical for the PSC test.

4.3 Microstructural Parameters

There are many microstructural parameters that can potentially be examined depending on the state of the metallic alloy under investigation. For example, phase and grain size and distribution, phase and grain shape, phase volume fraction, precipitate size, volume fraction recrystallised, dislocation density and texture variations are all of significant importance. For the development of this Good Practice Guide four representative engineering materials were studied; an aluminium alloy (A5052), a nickel alloy (Waspaloy), a steel (316 stainless steel) and a titanium alloy (Ti6Al4V). The results of specific studies are given in the Appendices. The primary microstructural measurements evaluated in these studies were grain size and shape, phase volume fraction and volume fraction recrystallised with the following definitions:

4.3.1 Grain Size

Two methods were used for grain size characterisation:

Linear Intercept and Circle Equivalent Diameter.

4.3.1.1 Linear intercept grain size (d_l)

Arithmetic number average of line lengths intersecting individual grains in 2D metallographic image.

4.3.1.2 Circle equivalent diameter (d_{ceq})

Arithmetic number average of diameters of circles of equivalent grain areas in 2D metallographic image of microstructure.

4.3.2 Grain Shape

Grain shape is defined by the number average of the ratio of maximum to minimum grain intercepts in orthogonal directions parallel and perpendicular to the primary deformation direction.

4.3.3 Phase Volume Fraction (V_f^a)

The volume fraction of a phase constituent (alpha) is the volume of the constituent divided by the total test volume. It is measured indirectly by measurements of the area fraction (A_f) of the phase constituent on a 2D image of the microstructure; It is assumed that

$$V_f^\alpha = A_f^\alpha$$

4.3.4 Volume Fraction Recrystallised (V_R)

The volume fraction recrystallised of a phase constituent is the volume of the constituent that has recrystallised divided by the total volume of the constituent. It is measured indirectly by measurements of the area fraction, A_R , recrystallised. It is assumed that

$$V_R = A_R$$

5

Testpieces

IN THIS CHAPTER

- Preparation of Testpieces for Hot Compression Tests
- Metallographic Preparation

5 Testpieces

The testpiece is usually obtained by cutting/machining a sample from a larger product form. The size of the testpiece is a compromise between being large enough to be representative of the microstructure of interest but not too large as to present problems in non-uniformity of temperature or in machine capacity during deformation. Machining should be such as to minimise residual stresses and changes in local microstructure close to the surface. Spark machining (EDM) can be used first of all to blank the testpiece shape from a larger block if necessary and even to form the final shape of the testpiece provided the surface quality is good and provided the metallurgical structure of the material is not changed. The identity and orientation of the testpiece with relation to the original material stock should be recorded. Reference to BS EN-ISO 3785 (Designation of testpiece axes) is recommended.

Calculate the approximate maximum stress required to deform the chosen testpiece size, if necessary from a series of preliminary exploratory tests. Choose a testpiece geometry which will ensure that the load capacity of the test machine is adequate to maintain sufficient force at the required strain rate **to the maximum value of applied strain of the test**. Manufacture sufficient testpieces for the required study.

It is more important to protect the surface of the testpiece from oxidation than to specify a surface finish to a required standard. There are several options e.g. plating, inert atmosphere or vacuum testing, the use of glass etc. Plating, although sometimes costly, can be beneficial in minimising surface oxidation prior to the test. The plating must be crack free, hard chromium plating in accordance with BS 4641.

After machining the testpiece dimensions should be measured with a tolerance of ± 0.02 mm on each individual measurement of the geometrical features [1,2].

5.1 Metallographic Preparation

There are many documents giving precise instructions for the preparation of metallic surfaces for image quantification by metallography and some of the more recent and thorough examples are given in the bibliography in Section 10 and Appendix A. In developing this guide for measurements of heterogeneity in structure several representative engineering materials were studied, including Al 5052; Waspaloy, 316 stainless steel and Ti6Al4V. The detailed metallographic procedures that were followed for preparing suitable surface of these materials are given in Appendix C. The key elements for optical microscopy were:

- Al 5052: Coarse and fine diamond grind and polish; colloidal SiO₂ suspension polish; Electrolytic etch in Barker's reagent. Examined by polarised light.
- 316 Stainless steel: Coarse and fine diamond grind and polish; Etching with glyceric acid for 2 min, preceded by immersion for 5 min in hot water; Reflected light microscopy.
- Waspaloy: Coarse and fine diamond grind and polish; Etching by swabs with 1 ml nitric acid, 2 ml sulphuric acid, 97 ml hydrochloric acid for 3 mins; Reflected light microscopy.
- Ti6Al4V: Coarse and fine diamond grind and polish; Etch with a Kroll's reagent variant (1 ml hydrofluoric acid, 2 ml nitric acid, 97 ml water) for 2 mins; Reflected light microscopy.

The key elements for the EBSD studies were

- Al 5052: Polish as above, final electropolish with 30% nitric acid in methanol.
- 316 stainless steel: polish as above, final 60 min polish with colloidal silica.
- Waspaloy: Polish as above, final 60 min polish with colloidal silica.
- Ti6Al4V: Polish as above, final polish with colloidal silica/20% hydrogen peroxide solution.

Specific guidelines, including extensive references, for specimen preparation for EBSD have been outlined by Katrakova and Mücklich [18]. These recommendations were usually followed in our studies. In particular

- Mounting in conductive resin.
- Minimum extent of grinding to obtain a flat surface.
- Extensive final polishing, often with colloidal silica.

Other recommended practices, such as ion beam polishing or etching were not investigated in the current exercise.

6

Compression Apparatus

IN THIS CHAPTER

- Basic Equipment for Hot Compression Tests
- System Calibration
- Data Recording

6 Compression Apparatus

Machines are available that apply compressive loads at well defined deformation rates to the testpiece either using servohydraulic actuators, or using mechanical methods, e.g. cam plastometers. In addition the testpiece may be heated. For this reason these guidelines recommend only that the test machine be calibrated for load, displacement and temperature measurement and that the calibrations are traceable. There is no formally recommended type of test machine. The apparatus required for the test requires some form of heating to be applied to the testpiece, usually a furnace, but alternatives such as an induction coil or direct heating using an electrical current are allowed. Tests should be carried out on a machine which has good lateral rigidity and accurate alignment between the testpiece tools, (platens). The machine loading can be hydraulic or electromechanical. Departure from conformance, i.e. lack of parallelism between loading platens, will result in non-uniform deformation. A valid test is one in which deformation is applied uniformly to the testpiece. The shape of the testpiece must be measured after the test to confirm the validity or otherwise of the test [1,2].

6.1 Load Measurement and Calibration

Full details of load measurement and calibration are given in the associated GPGs for mechanical measurements [1,2].

6.2 Temperature Measurements, Control and Calibration

6.2.1 General

Detailed background to temperature measurement is given in the associated GPGs for mechanical data [1,2].

There is a need to recommend tolerances for acceptable temperature gradients within the test system and for the temperature of test as microstructures are generally very sensitive to temperature distributions. The variation in indicated temperature from point to point within the testpiece and the deviation from the specified test temperature should not exceed the values given in Table 1 for AC tests. FE modelling studies at the IRC in Computer Aided Engineering, University of Wales, Swansea [1,2] allowed Table 1 to be constructed based on an uncertainty of about $\pm 2\%$ in the flow stress. Table 1 provides values for the precision and spread of temperature within a testpiece required to achieve this level of uncertainty.

Table 1
Axisymmetric Compression: Initial Temperature Tolerances*, °C

Test temperature, T	Temperature variation ⁺ , °C	Precision ⁺⁺
T < 600	± 2	± 2
900 > T > 600	± 4	± 3
T > 900	± 6	± 4

⁺ Variation is defined as the difference between the measured temperature of the test (usually at the centre) and the mean temperature of the testpiece (obtained through calibration measurements of typical temperature variations).

⁺⁺ Precision refers to the nominal uncertainty of the temperature measurement on the testpiece associated with the indicating sensor (thermocouple, pyrometer, etc) including errors from all sources, nominally at the 95% confidence level.

* The term “initial tolerance” has been used since at very high strain rates inherent deformational heating may cause the temperature to deviate outside the specified tolerances during the test (see Appendix C).

For PSC testpieces the variation in indicated temperature within the testpiece and the deviation from the specified test temperature should not exceed the values given in Table 2.

Table 2
Plane Strain Compression: Initial Temperature Tolerances*, °C

Test temperature, T °C	Temperature variation ⁺	Precision, ⁺⁺ °C
<i>Across testpiece thickness</i>		
< 600	12	± 2
600 < T < 900	18	± 3
> 900	24	± 4
<i>Across testpiece breadth</i>		
< 600	16	± 2
600 < T < 900	24	± 3
> 900	32	± 4

⁺ Variation is defined as the difference between the measured temperature of the test (usually at the centre) and the mean temperature of the testpiece (obtained through calibration measurements of typical temperature variations).

⁺⁺ Precision refers to the nominal uncertainty of the temperature measurement on the testpiece associated with the indicating sensor (thermocouple, pyrometer, etc) including errors from all sources, nominally at the 95% confidence level.

* The term “initial tolerance” has been used since at very high strain rates inherent deformational heating may cause the temperature to deviate outside the specified tolerances during the test (see Appendix C).

6.2.2 Time Dependent Temperature

Rapid deformation of the testpiece can cause the testpiece temperature to rise. This can have significant effects on microstructural heterogeneity since the temperature changes are generally non-uniform. The magnitude of the temperature rise will depend upon the material being tested and its associated thermal properties, the thermal properties of the platens and the rate of

deformation of the testpiece. Preliminary measurements with fully instrumented testpieces may be necessary to determine the magnitude of this effect.

This can be done by using a number of thermocouples at different positions inserted or attached in the testpiece (along its length) and at the platens. Subsequent tests can then be controlled using the furnace temperature provided that regular checks are made to ensure the calibration persists. Alternatively a system can be installed which uses contact thermocouples to the testpiece or thermocouples permanently in place within or close to the deformation platens. Large errors can be introduced if thermocouples are not in intimate contact with the testpiece and platens [1,2].

6.3 Hot Compression Test Data Recording

A system capable of collecting and storing the mechanical data should be used. The frequency of the system should be such that finite changes in load, cross head velocity and displacement with time can be measured, with at least 10 data points for each interval of 0.1 true strain. For a typical test at a strain rate of 1 s^{-1} , this means a sampling rate of 100 Hz [1,2].

7

Testing Procedure

IN THIS CHAPTER

- Methodology Outlines
- Testpiece Installation and Removal

7 Testing Procedure

The procedure for conducting hot, isothermal compression tests for the purpose of studying the evolution of microstructure heterogeneity contains the following basic steps.

1. Manufacture testpiece to chosen geometry.
Decide on testpiece numbers for each test parameter.
2. Calibrate test system for load, displacement and temperature.
3. Install testpiece in system.
Heating profile, lubrication.
4. Deform testpiece at specified temperature and strain rate.
Measure changes in load and displacement.
5. Remove testpiece from system, quench or slow cool in an agreed predetermined manner.
6. Prepare testpiece for microstructural measurements.
7. Conduct quantitative measurements of microstructural features.
8. Analyse and report data.
Include estimates of uncertainties.

These guidelines provide a methodology to address these basic elements.

7.1 Testpiece Manufacture

Detailed instructions are given in Section 5.

7.2 System Calibration

The test system must be calibrated regularly for load, displacement and temperature, including documentation of the calibration procedure as described in the complementary hot compression GPGs [1,2]. If equipment is moved or damaged and repaired it must be recalibrated before re-use.

7.3 Testpiece Installation

Ensure machine anvils (platens) are clean and flat.

It is necessary to examine platens frequently to ensure no serious damage has developed on their surfaces. Re-machine at regular intervals if necessary. It is good practice to have a number of sets of interchangeable platens available so that clean platens can be used regularly.

Alignment of the platens should be checked at intervals not exceeding 100 tests. Alignment can be checked by using a suitable strain gauged compression cylinder (following the procedure described in prEN 3988:1995 - Test Methods for Metallic Materials. Constant Amplitude Strain-Controlled Low Cycle Fatigue Testing (ASTM E1012)), or by a dial gauge indicator. The platens should be parallel to within $\pm 0.2^\circ$.

Include details of the heating profile to the temperature of the test and the dwell at temperature prior to the test in test report.

Because the test requires the testpiece to be heated to the temperature of test, including a period at temperature before the deformation process, it is necessary to understand how the microstructure of the testpiece changes with heating profile so that significant changes are minimised in, for example, grain size during the heating profile of the test prior to deformation. For steels it is also necessary to define the heating profile to the temperature of test since the heating rate through the transformation temperature may have an effect on the size of microstructural features.

Heat the testpiece to the test temperature as quickly as possible, either by installation directly into a hot furnace or by applying a temperature ramp. Record details of the heating rate. Hold at test temperature for a specified time before applying deformation loads.

7.3.1 Lubrication

Position the testpiece on the platens using appropriate lubrication and position thermocouples. **Unlubricated tests are not recommended.**

Lubricant can be applied directly to the platens or by coating the testpiece prior to testing.

7.4 Deformation

Apply a deformation load at the test temperature and strain rate to ensure that the final value of the true strain required by the test is achieved.

Measure the load and store the data at true strain intervals of 0.01 or less.

Measure the displacements and store the data at true strain intervals of 0.01 or less.

Measure the testpiece or nominal temperature and record the value, including observed tolerances (see Tables, Section 6.2).

Record the velocity of the crosshead throughout the test.

7.5 Testpiece Removal

For assessments of microstructural heterogeneity it is necessary to measure changes in the test material microstructure after different amounts of deformation. It is thus generally necessary to quench-in the structure following the test.

For parallel studies of microstructural evolution.

- Remove the testpiece from the system and either quench or air cool if a furnace is used for the heating source. Record the method of cooling and a value for the cooling rate in the test report.
- If a resistance method is used, turn off the heating current. Record the method of cooling and a value for the cooling rate.
- If the cooling rate cannot be measured, specify the type of cooling used, for example, air, furnace or water quench.
- Report elapsed time between end of test and start of cooling process.

8

Microstructural Heterogeneity Characterisation

IN THIS CHAPTER

- Testpiece Sectioning
- Heterogeneity Characterisation
- Metrology Issues

8 Microstructural Heterogeneity Characterisation

To support the development of this Good Practice Guide both microstructure and microhardness maps were acquired from sections through test samples of uniaxially compressed cylinders or plane strain compression testpieces of a variety of engineering materials including an Al alloy, a stainless steel, an Ni superalloy, Waspaloy and Ti6Al4V titanium alloy. The samples were compressed under various lubricated conditions at strain rates of 1 s^{-1} and 0.01 s^{-1} at temperatures of $950 \text{ }^{\circ}\text{C}$ and $1040 \text{ }^{\circ}\text{C}$ for the 316L and Waspaloy respectively, to a total strain of 0.5. Following deformation the samples were immediately water quenched at an estimated cooling rate of $50 \text{ }^{\circ}\text{C/s}$. A high strain rate combined with low friction gives rise to temperature inhomogeneities whereas high strain rate and high friction leads to both temperature and strain pattern heterogeneity.

8.1 Microstructural Preparation

Section the AC or PSC sample along the central deformation axis (in the breadth direction for PSC) and prepare the cut surface for microstructure characterisation by optical or electropolishing methods (see Section 5 and Appendices). Schematic diagrams of the cut surfaces are shown in Figs 1 and 2 for the AC and PSC testpieces. Positions are marked within strips where most of the metallographic studies were conducted. Full strips can be measured using EBSD techniques whereas specific fields should be selected for optical metallography. The latter should be sufficient to monitor heterogeneity through the testpiece from top to centre.

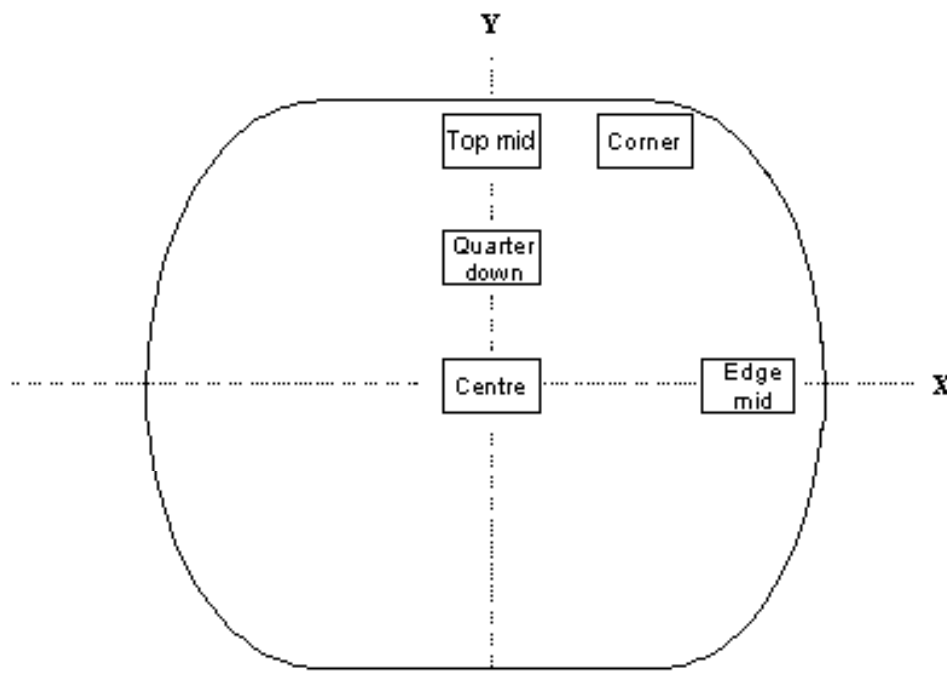


Figure 1 Typical schematic of AC testpiece showing positions for metallography.

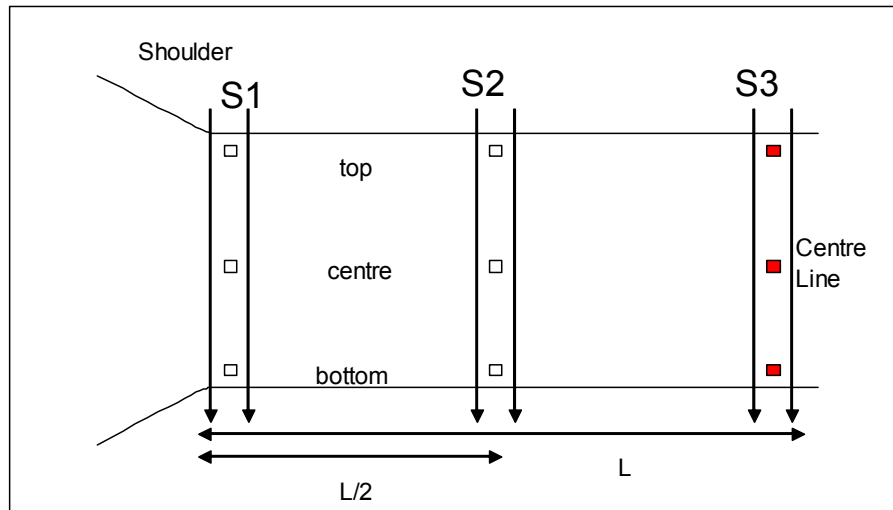


Figure 2 Schematic of PSC testpiece showing positions for metallography.

8.2 Microstructural Feature Quantification

Decide on the appropriate features that require quantification. Prepare polished surface to a degree requisite with accurate feature measurement (etch if required). Use optical, SEM or EBSD methods to image structure, appropriate to the material of interest. Measure size and other desired attributes of each targeted microstructural feature. An example is given in Fig 3 of an assessment of grain shape effects (see Appendix C for more detail). For both optical and SEM methods there are a range of metrological issues that need to be addressed and comments and good practice are summarised and discussed individually in the following sub-sections (8.2.1 and 8.2.2).

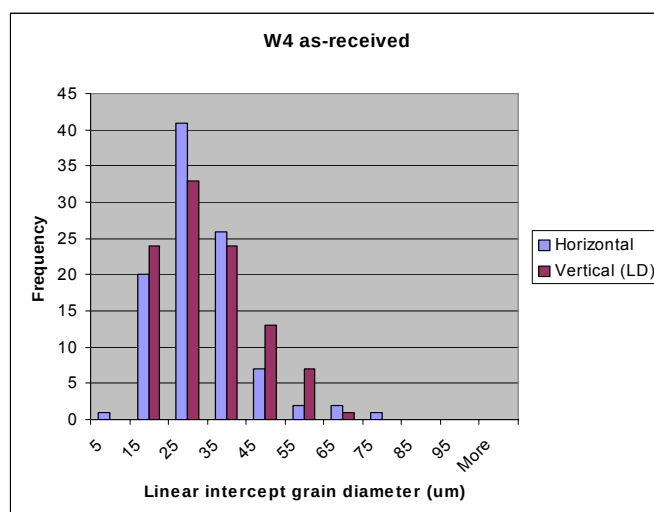
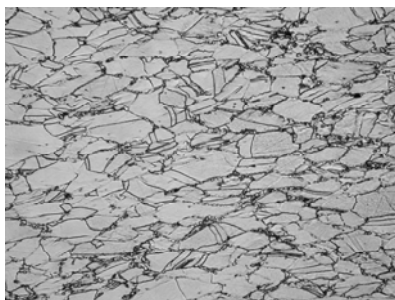


Figure 3 Optical grain size measurements from the PSC Waspaloy testpiece deformed dry at high strain rate.

8.2.1 EBSD Related Issues

Electron Back Scatter Diffraction, EBSD, is increasingly used as a tool for characterising the microstructure of a wide range of engineering materials by mapping the crystallographic orientation at many points across a polished section of a suitable testpiece [19-21]. The automated nature of EBSD means it can provide a large amount of information which is less susceptible to the skill and subjectivity of individual. However, there is still a need for care in specimen preparation and choice of operating and analysis conditions, in particular the step size:grain size ratio since this may vary with position. Indexing rates of >85% are generally required for minimal errors but with suitable noise reduction to 'infill' non-indexed points this requirement for indexing can fall to about 50% [19], but only for fully recrystallised unstrained samples. EBSD maps typically do not correctly identify 100% of the points examined, so the effects on measured grain size of post-acquisition methods to correct these points, or noise reduction, should also be considered. Thus, in formulating a guide for good practice in the metallographic characterisation of heterogeneity in structure from an EBSD perspective there are various issues that need to be considered. These are discussed in detail in Appendix B and summarised in the following sections 8.2.1.1 to 8.2.1.9. Some of these recommendations also apply to images generated by conventional SEM or optical methods.

8.2.1.1 Microscope stage drift

An acceptable level of drift needs to be defined. Knowledge of stage drift is vital as it can introduce errors in length measurements, which further vary with changes in step size and diffraction pattern acquisition times.

8.2.1.2 EBSD software

There are two major analytical software packages in use (Oxford Instruments/ HKL-Channel 5 and TSL) and questions arise as to the comparability of measurements using the different systems.

8.2.1.3 Noise removal; grain dilation/infilling of nearest neighbours

No EBSD map will achieve accurate indexing of every pixel examined. In addition to errors of orientation measurement for each pixel indexed, some pixels will not be indexed at all, and some may be incorrectly indexed. The relative proportions of these will depend on sample preparation, SEM operating conditions and EBSD indexing parameters. In simple recrystallised samples, accuracies of 95% of pixels correctly indexed can be achieved. This level of 95% should be the target for all maps, but in many cases this level is not reached and if the raw data is used to determine grain size severe errors in size will result. Equally, incorrect or excessive manipulation of the raw data can alter the final measured sizes significantly. The following steps for good practice should generally provide reliable data:

Remove all grains of 3 or fewer pixels (remove all single pixels as a minimum – 'wildspike' removal with HKL software does not remove all such pixels). Look at the histogram of sizes to determine if larger clusters should be removed, bearing in mind the increasing error in size measurement as the size reduces, and also whether the small grains have a significant impact on the structure/properties. Use a smaller step size to map a selected area to determine the reality of these clusters (nb. See section 8.2.1.6 on grain boundary definition).

Indexing of unindexed pixels is variously referred to as infilling, dilation or extrapolation. This should be kept to a minimum, especially for large step sizes. Recommended good practice is to index any unindexed pixels where they are surrounded by *X or more pixels* of the same orientation, where X is dependent on the grid used for mapping (square or hexagonal) but use the largest number possible (e.g. 5 or 6 neighbouring pixels for a square grid). Avoid increasing the percent indexed by more than 5%, and record the percent increase. It may be advisable to determine grain sizes before and after this operation to check on the effect.

Use an orientation filter such as the Kuwahara [22] filter to reduce errors in orientation measurement - especially for heavily deformed samples.

The choice of noise reduction methods can also lead to changes in calculated mean grain size greater in magnitude than that produced by the different step sizes. Careful consideration needs to be given to the size and location of pixel blocks removed as noise, since a theoretical examination suggests that a 2-D section should result in many very small grain cross sections. If comparison is to be made with measurements of conventional optical and SEM images then the resolution of the difference techniques should be compared.

8.2.1.4 Choice of step size and dealing with small grains

If not known already, it is preferable to obtain an approximate grain size and shape by a quick imaging technique. Light optical microscopy may work on a region with slight polishing relief or an etched region adjacent to that to be examined by EBSD. Forescatter imaging using diodes mounted on the EBSD detector can also produce images relatively quickly.

The step size should be chosen in relation to the average grain size, unless information on a particular minimum size is required. In either case it must be recognized that a judgement is being made on the minimum number of pixels that are used to define a grain either by a lineal or areal method, and thus on the minimum grain size that can be measured. A simple rule that can be applied for a preliminary scan is that the step size should be less than 10% of the approximate mean grain size [20]. To confirm the validity of a chosen step size, repeat mapping of a single area at several step sizes can determine the maximum size below which no significant difference in average grain size is seen. Accurate analysis of small grains requires a smaller step size, but if they represent a small fraction of the total area then it may be possible to increase to 0.3 or even 0.5× small grain size.

EBSD linear intercept measurements appear slightly less sensitive to step size and noise reduction than the use of circle equivalent diameters and results in a larger measure of mean grain size because fewer of the very small grains are sampled [23].

Duplex structures were observed in the high strain rate Waspaloy samples and this resulted in difficulties in resolving fine grains and a wide range of measured diameters. The optical grain size results in the AC testpieces were significantly larger than those obtained by the EBSD technique (Table 5); However there was better agreement in the PSC testpieces where the etching technique had improved (see Fig 4 and Table 3), but the mean optical grain size was still 10-15% larger than that obtained by EBSD. It is useful to plot distributions of sizes (Fig 5) to allow greater discrimination between the different techniques.

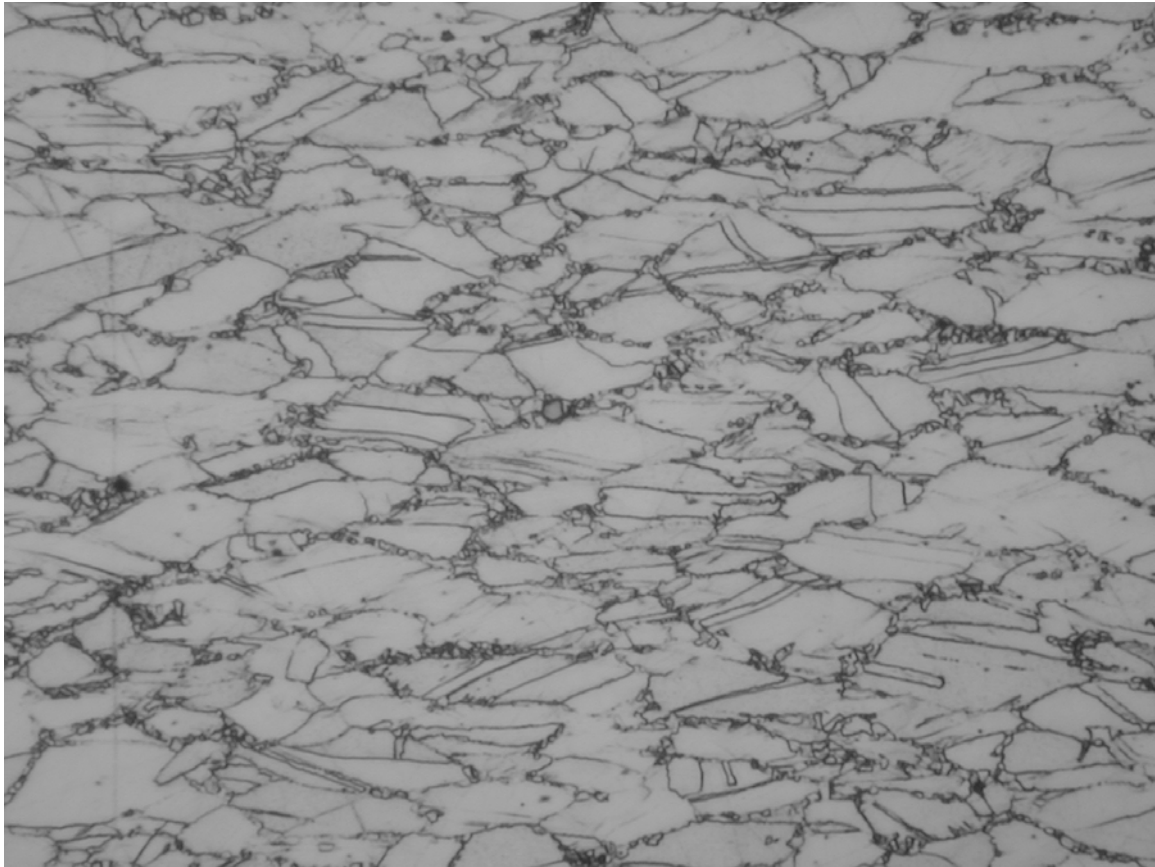


Figure 4 Optical micrograph of deformed Waspaloy PSC testpiece (W4) showing duplex grain size (nominal magnification x500).

Table 3 Waspaloy PSC testpiece grain size – number averages

Optical		EBSD	
	mli, μm	mli, μm	d_{ceq} , μm
all data	9.8	all	9.0
data > 4 μm	12.6	data > 4 μm	10.0
			8.5
			10.6

Table 4 316 stainless steel PSC testpiece grain size – number averages

Optical		EBSD	
	mli, μm	mli, μm	d_{ceq} , μm
all data	11.2	10°	9.0
data > 4 μm	12.2	15°	9.9
		10° > 4 μm	9.7
		15° > 4 μm	10.7
			11.3
			13.1
			11.8
			13.8

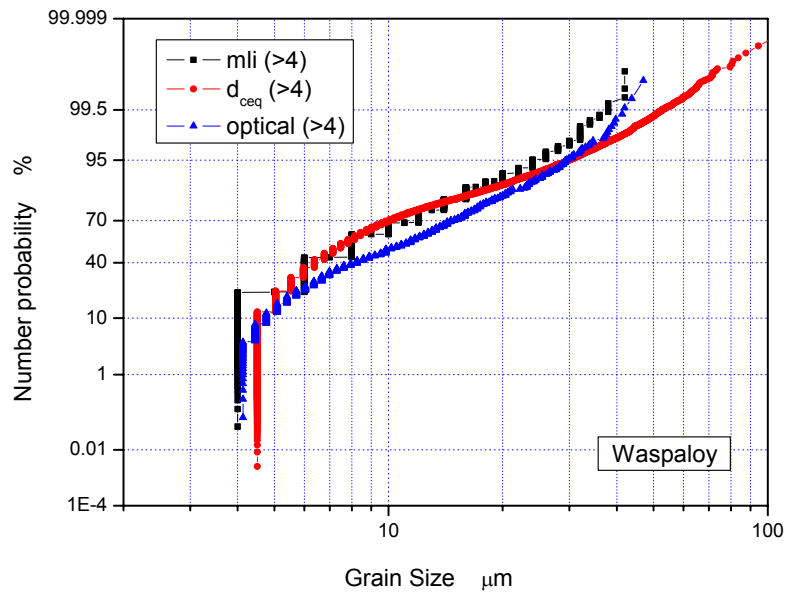


Figure 5 Distributions of grain size in deformed Waspaloy PSC testpiece (W4).

Table 5 Comparison of optical and EBSD grain sizes (μm) on Waspaloy AC testpieces.

			USEM	USEM	USEM	USEM	USEM	USEM	Swan
			d_{ceq}	d_{ceq}	Linear	d_{ceq}	d_{ceq}	Linear	Opt
GB Misorientation ($^{\circ}$)			10	15	15	10>4pix	15>10pix	15>10pix	
W3	S3	Top	10.4	11.8	11.6	14.1			38.3
		Centre	6.9	7.1	9.0	11.0			41.0
W4	S2	Top	9.7	10.1	11.9	14.0	14.7	15.0	19.5
		Centre	6.2	6.3	6.3	8.8	9.0	9.7	15.4
	S3	Top	9.7	10.1	10.1	14.5	15.2	14.0	19.8
		Centre	6.2	6.4	6.2	8.8	9.0	9.4	16.2

The assessment of grain size across a section of a hot deformed sample illustrates that the choice of step size and of the range over which averaging is applied are very important when changes in grain size are being assessed over large areas. For the recrystallised 316L stainless steel specimen, increasing the EBSD step size from 5% to 20% of the approximate mean grain size resulted in a change in calculated arithmetic mean grain size of >15%, based on a circle equivalent diameter value. The grain size distribution did not appear to be lognormal, having a large number of very small grains, and thus the geometric mean is no more appropriate than the arithmetic mean for quantifying the grain size of the microstructures examined here.

Optical linear intercept measurements in the 316L stainless steel produced measurements of grain size larger than EBSD measurements of the same sample because fewer small grains were resolved by etching and the use of the optical technique and some grain boundaries were simply missed by eye (Fig 6 and Table 4).

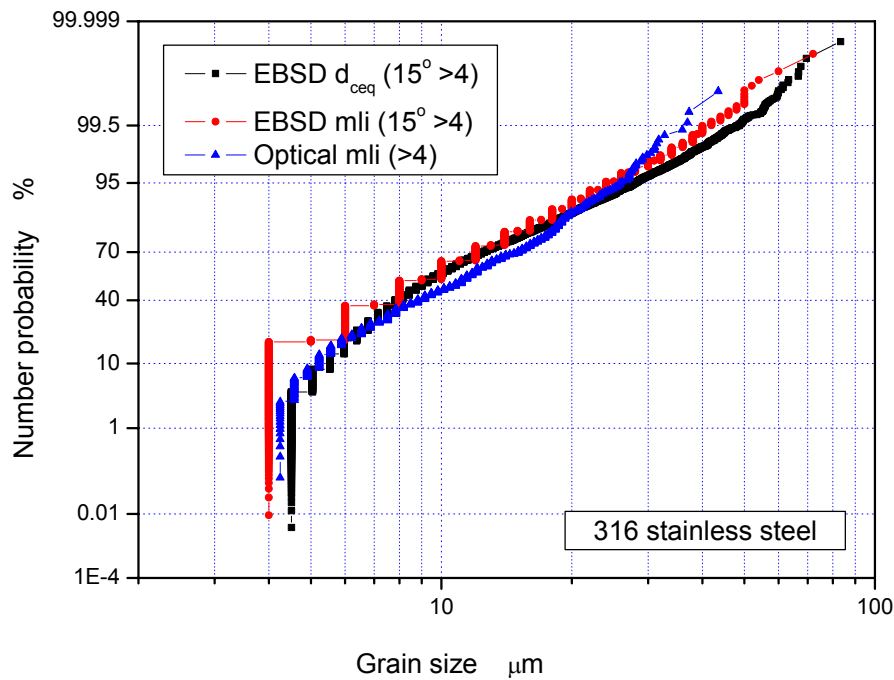


Figure 6 Distributions of grain size in deformed 316 stainless steel testpiece (W4).

Further examples of the effects of step size in EBSD maps from the Waspaloy testpieces and stainless steel are shown in Fig 7 (discussed in detail in Appendix B).

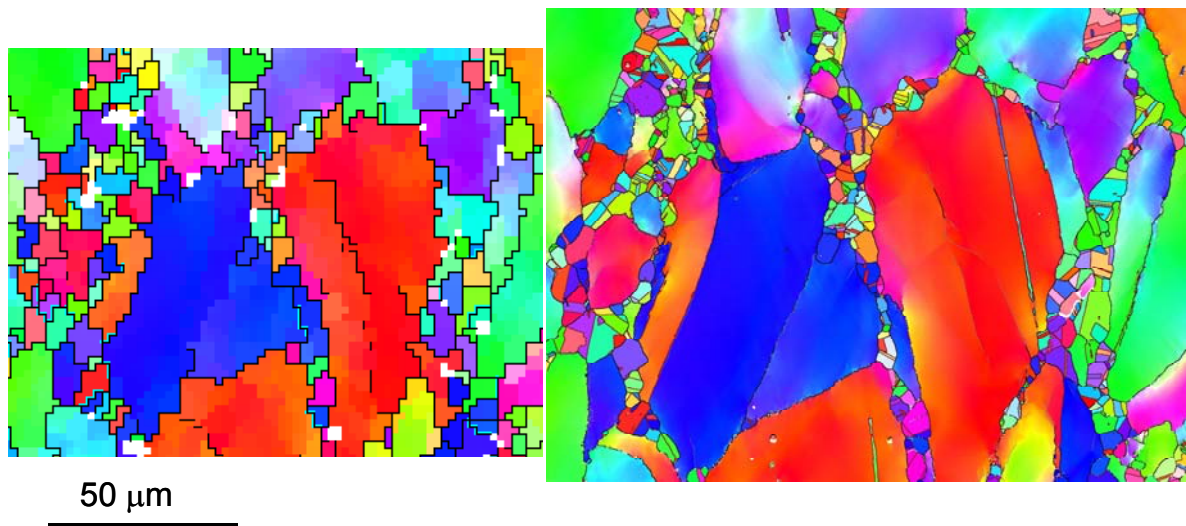


Figure 7 Effect of step size on image resolution in recrystallised Waspaloy testpiece.

8.2.1.5 Grain boundary definition (10° or 15°) and twins

After following the steps above and acquiring data to plot maps of, for example, orientation, grain boundaries can be drawn on the maps. This requires choice of the angular definitions

for the various possible boundaries. Guidelines are given below, but whether these or other values are used, the definitions used should be quoted with all results.

For recrystallised or cast undeformed metals the misorientation that is used to define the grain boundary may be taken to be as little as 5° . For other materials larger angles, typically 10° or 15° are used [24-26]. Measurement of grain size as a function of angle may be useful in revealing information about the structure. Repeat analysis with different angles may indicate a degree of substructure. In fully recrystallised metals choice of angle will have a small effect. In some materials, particularly after deformation, some boundaries may not extend completely between two regions to terminate at a triple point with another boundary because the measured misorientation changes along the length of the boundary fall below the defined grain boundary angle. In such cases it may be possible to extrapolate the boundary by reducing the minimum angle generally used elsewhere in the map to a new, lower, defined value. If this is done it should be recorded with the final result (preferably reporting effect on mean size with and without extrapolation).

With conventional techniques special boundaries such as twins in cubic materials, which can be identified by their morphology, are ignored for the purposes of grain size measurement. But definition must be as tight as possible to avoid false classification of small boundary sections as twins. Since EBSD measures orientation these boundaries can be easily determined by software and excluded from EBSD measurements of grain size. However, since there will be some variation in measured orientation about the idealised value, the tolerances used to define the boundaries should be recorded (e.g. $60 \pm 2^\circ$ about $\langle 111 \rangle \pm 2^\circ$). It should also be noted that:

- a) EBSD will define some boundaries as twins because they meet the defined tolerances where conventional light microscopy would not identify them as such because the typical morphology of twinning is not obvious.
- b) some boundaries in a) will lead to much larger grain sizes than expected if the software is told to exclude twins from grain size measurement.

8.2.1.6 Recrystallisation

Although many techniques have been used to characterise recrystallisation kinetics, metallography is the most definitive [27-31]. Point counting methods were typically used to estimate volume fraction recrystallised, V_R , prior to the use of EBSD, in homogeneously deformed materials; but this can give quite large uncertainties (sometimes as large as a factor of $\times 2$ due to the difficulties in etching all relevant boundaries [32]. The potential for higher accuracy definition of boundaries, and their nature, using EBSD (especially when line scan methods, even if lengthy, are used rather than point counting) has encouraged various detailed studies of recrystallisation kinetics, particularly in Al alloys [29-31]. Various approaches to determining the recrystallised fraction from EBSD data are possible. What constitutes a recrystallised grain (as opposed to a subgrain or deformed grain) must be clearly defined, usually in terms of misorientation either between neighbouring pixels or averaged over all pixels within a grain. The exact approach depends on whether the material forms a cell substructure e.g. Al [29-31] or does not e.g. stainless steel [33]. Examples of this latter case are shown in Appendix B, B3.1.3. Recrystallised grain volume fractions are generally fitted to Avrami-type plots and in an homogeneous material the numbers of grains that need to be measured to give acceptable accuracy at $t_{0.50}$ (time for 50% recrystallisation) is about 500 [28]. However, in materials with gradients of recrystallisation this may not be possible. It is

advisable, therefore, to use the running average methodology based on fields of specific size and to study the effect of field size, and therefore statistics, on the outcome.

8.2.1.7 Mapping large areas

Stitching mapped areas together is difficult because of distortions towards the edges and corners of the mapped area, but can be more accurate than analysing low magnification single maps. Using multiple smaller areas can enable different step sizes to be used for different areas but the software may not stitch different step sizes. At low magnifications the distortions at the edges become worse still and away from the centre the beam sample geometry deviates a long way from the geometry used to calibrate the EBSD system so indexing rates also drop.

8.2.1.8 Quantifying gradients in structure

Running averages, defining strips for quantification, selection of specific size classes, and algorithms for initiating and defining EBSD mapping parameters are all issues that need to be addressed for quantifying gradients in structure. To illustrate the issue of mapping heterogeneity Figure 8 shows an orientation map obtained using an electron backscattered diffraction (EBSD) technique for a Ni superalloy (Waspaloy) with inverse pole figure colouring. The map was acquired with a 1 μm step size from the centre line of an axial cross section of a cylindrical axisymmetric compression sample. The sample was produced with a relatively high friction coefficient between sample and platens, with a strain rate of 0.01 s^{-1} at a temperature of 1040 $^{\circ}\text{C}$. The high friction results in a region of large unrecrystallised grains, evidenced by EBSD measurements of strain, close to the surface while the inner region consists of many smaller recrystallised grains. It is important to be able to quantify this heterogeneity in structure and define where in the testpiece is the optimum position for making measurements. In the current research the microstructure studies were largely being conducted by optical and EBSD measurements and these were complemented by the use of a scanning depth sensing microhardness test system recently developed at NPL [34]. Scanning microhardness maps provide useful insight on microstructural heterogeneity, enabling more focused studies to be performed of microstructural variations (see Appendix D for more detail).

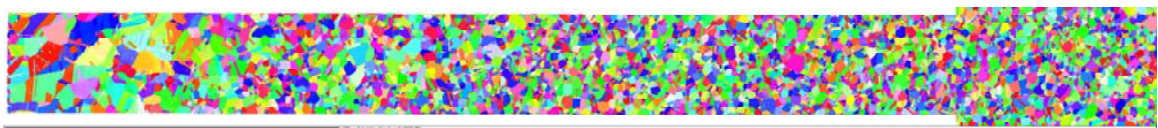


Figure 8 EBSD map of grain orientation in a section taken from a uniaxially compressed sample of Waspaloy. Top surface on left of map, centre of sample on right

In studies of the various factors that might be used to quantify heterogeneity through the Waspaloy PSC testpieces it was found that the mean misorientation parameter showed a greater variation than grain aspect ratio, grain size or feret diameter (Fig 9).

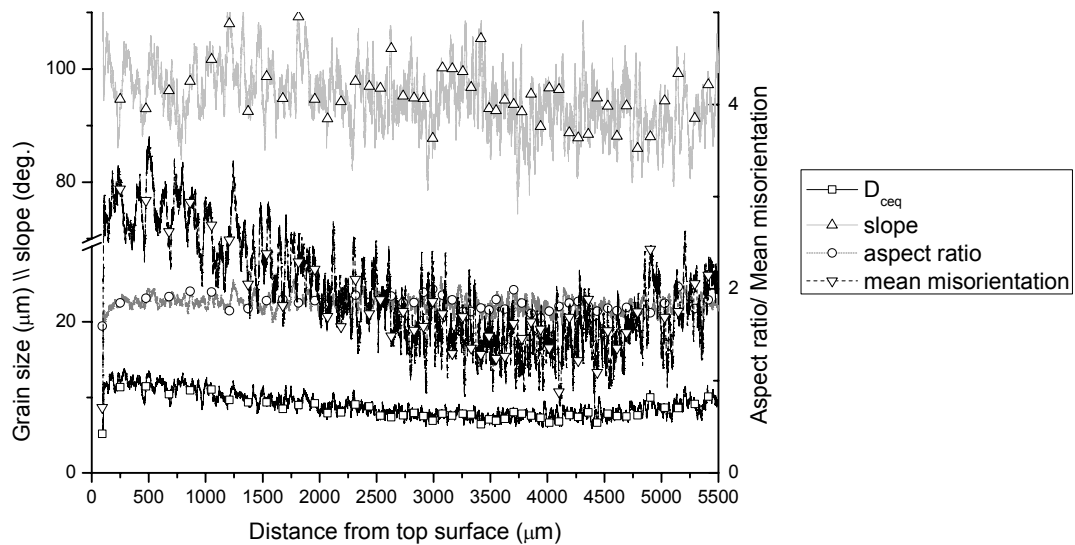


Figure 9 Variation in microstructure parameters through vertical strip in PSC Waspaloy testpieces.

An example of the effects of running averages in EBSD maps from the Waspaloy testpiece is shown in Fig 10 (discussed in detail in Appendix B). It is important to differentiate between moving averages and cumulative moving averages. The latter are occasionally used when investigating whether a particular mean value has settled down to a stable value. If it drifts with number then it could indicate a variation with position. Moving averages (where the number average is taken from a set that moves along the line of data) are used in the examples shown in this guide.

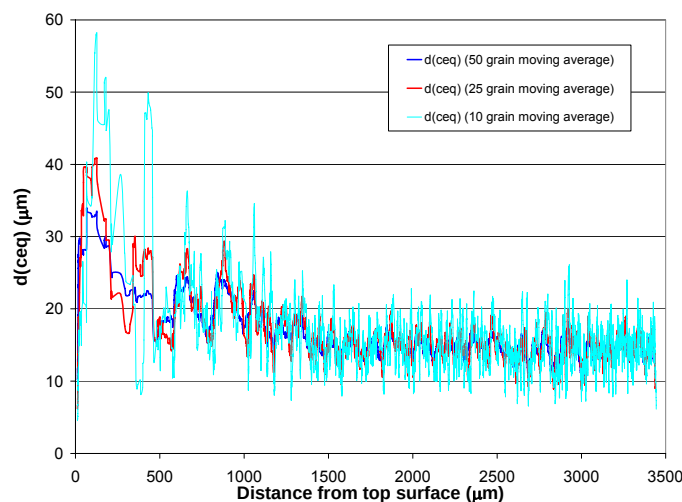


Figure 10 Effect of data analysis on running average grain size through central strip of AC Waspaloy testpiece.

Varying the number of grains averaged indicates the degree of homogeneity. It may be necessary to exclude the smallest grains to show trends, especially if data is noisy.

8.2.1.9 Grain shape

Figure 11 shows an EBSD map from a strip through the PSC testpiece of the 5052 Al alloy illustrating the necessity for measurements in the X and Y direction if intercepts are used for grain size measurement (due to the flattened shapes of the grains). Whether grains are really present or are an artefact of the measurement process is key to obtaining good data. It is recommended that plots of size distribution might help in making critical decisions as to whether to include a small sized grain or not. Also, from regions yielding more than 500 grains using d_{ceq} can be described by the ratio of max:min diameters (feret diameters), and the orientation of the principle axis can be used to plot changes in direction of elongation.

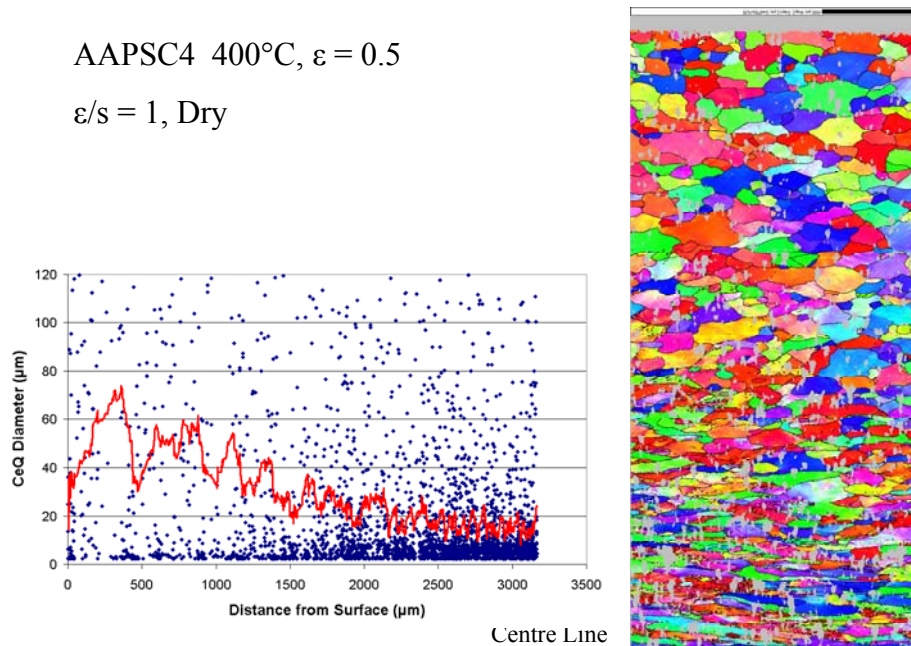


Figure 11 EBSD and grain size plots from AA 5052 PSC testpiece.

Figure 11 also illustrates a significant problem with EBSD of Al alloys, that of artefacts (misindexed or not indexed) caused by the presence of second phase (dispersoid) particles.

8.2.2 General Metrological Issues

8.2.2.1 Grain size representation (mean linear intercept or equivalent circle diameter)

There are two popular methods in use to estimate the grain size; one method is based on the measurement of an equivalent circle diameter (ECD) and the other method on the use of linear intercepts (LI). The LI method is based on the number average lengths of intercepts through each crystal/grain along a line drawn across the material surface. The ECD method is based on the number average area of the grain/crystals. The average area is converted to an equivalent circle diameter (ECD) as a measure of size. De Hoff and Rhines, in “Quantitative Microscopy”, [35] stated that an empirical relation had been found in measurements in aluminium and ferrous alloys where

$$LI = \sqrt{A} \quad (2)$$

where $\bar{A}$ is the average grain area. Thus since $\bar{A} = \frac{\pi}{4}(\text{ECD})^2$

$$\text{ECD} = \sqrt{\frac{4}{\pi}} \sqrt{\bar{A}} = 1.13 \sqrt{\bar{A}} = 1.13 \text{ LI} \quad (3)$$

There is a reasonable probability that a proportion of small grains may not be visible in the nominally planar surface, either as a result of a loss or as a result of resolution limits. This is very difficult to quantify, but it is likely to affect LI and ECD measurements equally and should not bias the measured correlation unduly.

EBSD will give information on the size of all the grains examined and thus the distribution of sizes can be relatively simply reported as well as a mean value. Linear intercept, d_{lin} , is directly comparable with conventional measurements and if measured in 2 directions indicates anisotropy. d_{ceq} needs shape data to complement mean size but gives data on all grains present and with x-y coordinates allows size versus position to be plotted.

8.2.2.2 Size distribution

ASTM E1181 addresses some of the issues associated with grain size distribution for example it discusses various typical examples, such as; isolated coarse grains in a finer grained matrix, extremely wide distributions and bimodal distributions. Other difficult issues are mentioned too, such as; banded structures, necklace structures and systematic variations across sections of products.

ASTM E1181 also contains comparison charts for estimating area fractions (possibly in multiphase materials). A number of procedures are defined, including:

- test grid on transparent overlay - superimposed image - estimate area fractions
- largest grains
- comparison charts
- intercept method for grain size.

The standard advises that the estimation of area fractions of different sizes is subjective and prone to error. The standards recommend that the most effective method for measuring intercept distributions is to use a semi-automatic image analysis system with digitising tablets and electronic pencils/cursors - using a test grid with 5 evenly spaced horizontal lines. The intercepts are classified by size and the data is presented as a histogram or frequency plot.

The linear intercept technique is frequently used for measurements. This is popular because it also produces size distribution information. It is often found that the measured intercept number distribution is lognormal in form, which gives a straight line when plotted on lognormal probability paper [6]. Another option is measurement of the sectional grain area, as discussed by Vander Voort and Friel [5], but this depends on the production of suitable images for image analysis. The use of EBSD potentially provides a considerably more efficient and accurate determination of this parameter [14] and is thus particularly suited to distribution characterisation.

8.2.2.3 V_f second phase

The comments in 8.2.2.2 on assessing area fractions apply equally to assessment of volume fractions of second phases. EBSD will however clearly identify different phases from the crystallographic identification of each pixel and can thus estimate second phase fraction by area (within the limits of noise reduction in 8.2.1.3) or by linear intercept techniques.

8.2.2.4 Number of fields and features

Standard ASTM E112 suggests a minimum of 3 fields to be examined, with a minimum of 50 grains in each field. Because EBSD maps enable the size of all grains to be measured from a given field, this minimum number of grains will be easily exceeded. However the need for 3 fields, randomly located within the region of interest, remains if the uniformity of the microstructure over a larger area needs to be assessed. The use of running average plots can be useful in showing that a stable repeatable value has been obtained. Confidence limits may be calculated as with conventional metallography [36]

8.2.2.5 Size classification, by number or by area

Classification by number is most common, but particularly if many small grains are present and/or noise (false small grains) is a problem calculating mean values by area (size) can prevent the noise dominating the results.

8.3 Summary Recommendations

To obtain reliable and repeatable microstructural measurements, a consistent approach to acquiring and analysing the data must be taken, whether by optical, SEM or EBSD methods. To compare two regions by EBSD then maps should:

- have the same step size, chosen relative to the mean grain size ($< 10\%$) or for complex distributions of sizes, to give data on the smallest grains of interest with a minimum of 10 pixels/grain
- have been prepared with the same noise reduction routines, e.g. removal of grains < 3 pixels in size, minimal grain dilation into non-indexed areas and use of a Kuwahara filter
- use a consistent definition of grain boundaries (e.g. 10° misorientation), and sub grain boundaries if necessary.

Ideally the effects of changing any of these parameters should be explored to check that no artefacts are introduced or trends obscured. To represent the data clearly and to show trends in microstructure with position in the sample, it is recommended that moving averages are employed, and that all the data available is analysed. This should include the use of diameter, area, aspect ratio and particularly mean misorientation if EBSD is used. This latter parameter is useful to distinguish between recrystallised and highly strained non-crystallised grains. Also, it is helpful to present information on grain sizes within specific sets of fields as size distributions for enhanced interpretation of the effects of large numbers of small grains or a non-uniform population of larger grains.

9

Finite Element Modelling

IN THIS CHAPTER

- Framework for Modelling Studies
- Results of FE Analyses on AC Testpieces
- Preliminary Results of FE analyses on PSC Testpieces

FE modelling studies were conducted of the AC and PSC geometries to define test conditions in the four materials of interest (Al 5052, 316 stainless steel, Waspaloy and Ti6Al4V) to ascertain what conditions would generate sufficient heterogeneity in structure. For example, a high strain rate combined with low friction gives rise to temperature inhomogeneities whereas high strain rate and high friction leads to both temperature and strain pattern heterogeneity. Details of the final systematic testing conditions are given in Table 6:

Table 6 AC and PSC Testing Conditions

Test number	Material	Initial Temp (°C)	Friction	Strain rate (s ⁻¹)
W1	Waspaloy	1040	lubricated	0.01
W2	Waspaloy	1040	dry	0.01
W3	Waspaloy	1040	lubricated	1
W4	Waspaloy	1040	dry	1
T1	Ti 6:4	950	lubricated	0.01
T2	Ti 6:4	950	dry	0.01
T3	Ti 6:4	950	lubricated	1
T4	Ti 6:4	950	dry	1
A1	Al 5052	400	lubricated	0.01
A2	Al 5052	400	dry	0.01
A3	Al 5052	400	lubricated	1
A4	Al 5052	400	dry	1
S1	316L	950	lubricated	0.01
S2	316L	950	dry	0.01
S3	316L	950	lubricated	1
S4	316L	950	dry	1

The FE model outputs of effective strain were colour coded in zones to show where in the deformed testpieces the mean value of target strain had been achieved ($0.5 \pm 10\%$; colour coded green). It can be seen from the diagrams shown in sections 9.1 to 9.4 for the AC testpieces that this zone is generally below 50-60% of the distance from the centre of the testpiece to either the top or bottom surfaces within the central strip. As can be seen from the accompanying microstructural studies this is a reasonable portion within which to measure average features in the structure if resource is only available for limited studies. ***It is recommended, however, that in general a full strip from top to centre is fully characterised due to the heterogeneous nature of deformation within most testing conditions. This recommendation equally applies to the PSC testpieces described in section 9.5.***

9.1 Al 5052 – AC Model Studies

The finite element model of the axisymmetric compression test allows the calculation of strains throughout the deformed specimen. It is possible that the grain shape in the specimens also reflects these strains. This will only be so if no change in grain shape occurs as a result of structural changes such as grain boundary migration, grain spheroidization, grain growth or recrystallisation. Although specimens were rapidly quenched, it is not clear if these conditions were met. It was nevertheless useful to attempt a comparison. The strain in the y direction can be calculated from

$$\varepsilon_{yy} = \ln \left(\frac{h}{h_o} \right)$$

where h and h_0 are the final and initial tangent grain diameters in the y direction. The model strains are available from the finite element results and typical examples are shown in Figs 12 and 13. For comparison purposes, the average strains over the different sampling areas were calculated from the finite element outputs. The strains from grain measurement, together with their confidence intervals, and the strains from the model are shown in Table C7 (Appendix C).

9.2 316 Stainless Steel – AC Model Studies

Computer models of the four specimens generated a set of results similar to those described for the Al 5052 in the previous section. Examples are shown in Figs 14 to 19. Figures 14 and 15 show the effective strains for the specimens S1 and S4 and Figs 16 and 17 show the temperature rises. The computer model allows the calculation of grain aspect ratio for the deformed grains on the assumption of no recrystallisation during test. The complementary contour plots of grain aspect ratio are shown in Figs 18 and 19.

The aspect ratios calculated on this basis are shown for the four specimens and four sampling areas in Table C9 (Appendix C). Where boxes are shaded, the calculated value is not significantly different from the measured values of Table C8 (Appendix C). Thus, for all areas in sample S1 the measured ratios correspond with the model estimates indicating that no recrystallisation had occurred. Elsewhere the measured values are less than the calculated values indicating recrystallisation. For these areas, the grain sizes are also smaller than those for un-recrystallised zones. For the recrystallised areas the measured aspect ratios are greater than zero indicating that some deformation had occurred after the recrystallisation process. In general, the propensity for recrystallisation increased with increasing strain rate (i.e. increasing deformation temperature) and with increasing friction (i.e. with increasing inhomogeneity of strain).

9.3 Waspaloy – AC Model Studies

It is likely that the grain size distributions observed are a function of the thermal and strain history of the specimens. Finite element calculations of the distributions of strain and temperature have been made. Figures 20 and 21 show the effective strains for specimens W1 and W4. Those specimens deformed at low strain rates showed uniform final temperatures with temperature rises of 2.3 °C and 2.6 °C for low friction and high friction (W1 and W2) respectively. The temperature distributions for the high strain rate tests were not uniform and are shown in Fig 22 for the high friction sample (W4). A major factor affecting recrystallisation is local strain. For lubricated specimens (W1 and W3) strains are sufficiently uniform and large to have produced nearly full recrystallisation. For the unlubricated specimens, the low strains associated with the dead zones beneath the dies have failed to complete recrystallisation.

9.4 Ti6Al4V – AC Model Studies

The specimens T1 to T4 were modelled by a thermally coupled finite element programme. Typical effective strain patterns for deformed specimens are shown in Figs 23 and 24. The inhomogeneity of strain increases with increasing friction. The specimens deformed at 0.01 s^{-1} , T1 and T2, showed uniform temperature rises of 0.9 °C and 1.1 °C respectively. The final temperature distribution in specimen T4 is shown in Fig 25. Deformation without

temperature rise has a small effect in changing alpha volume fraction towards equilibrium values but the major effect is produced by temperature rise. Comparison of Table C11 (Appendix C) with Fig 25 shows that the greater the temperature rise in parts of the deformed specimens, the greater the tendency towards equilibrium structure.

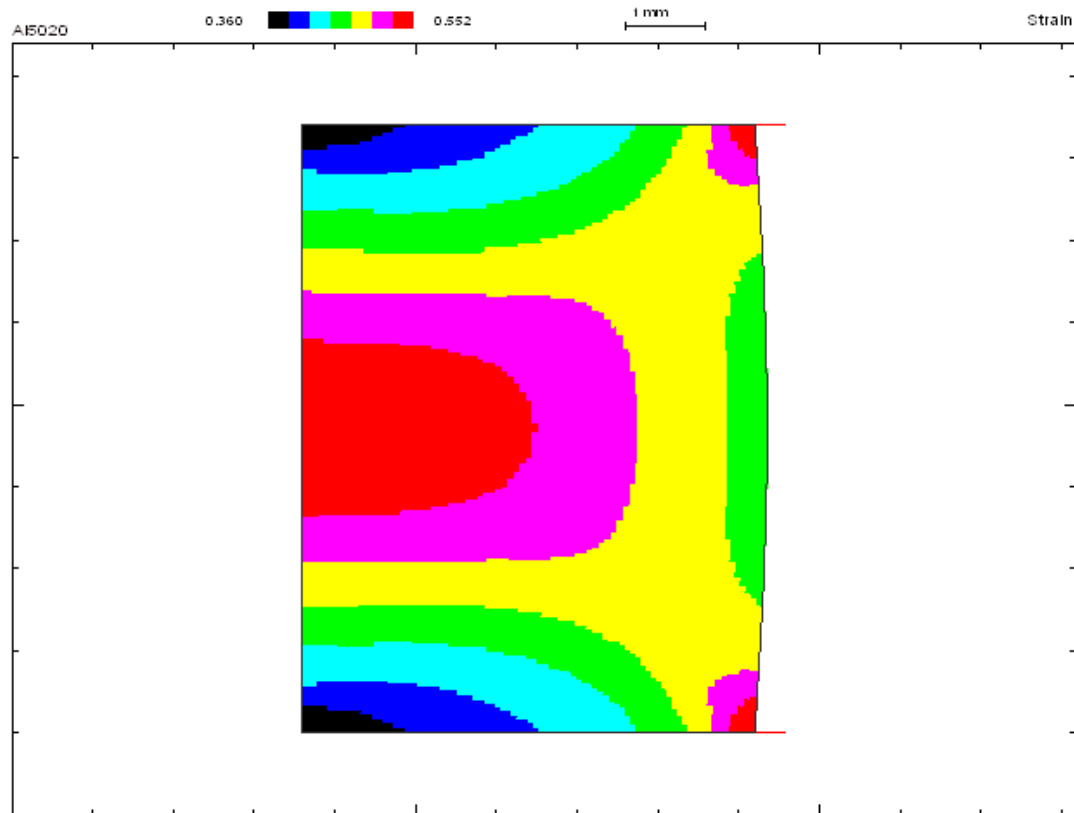


Figure 12 Finite element model strain distribution in specimen A1.

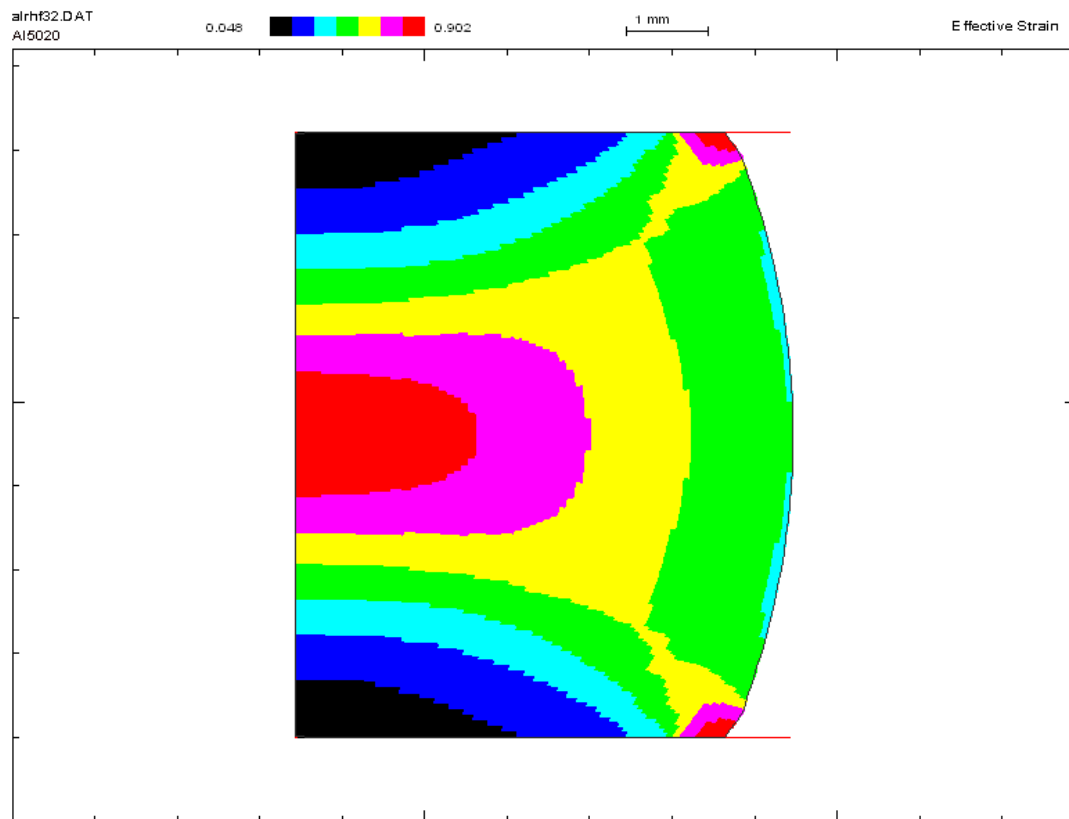


Figure 13 Finite element model strain distribution for specimen A4.

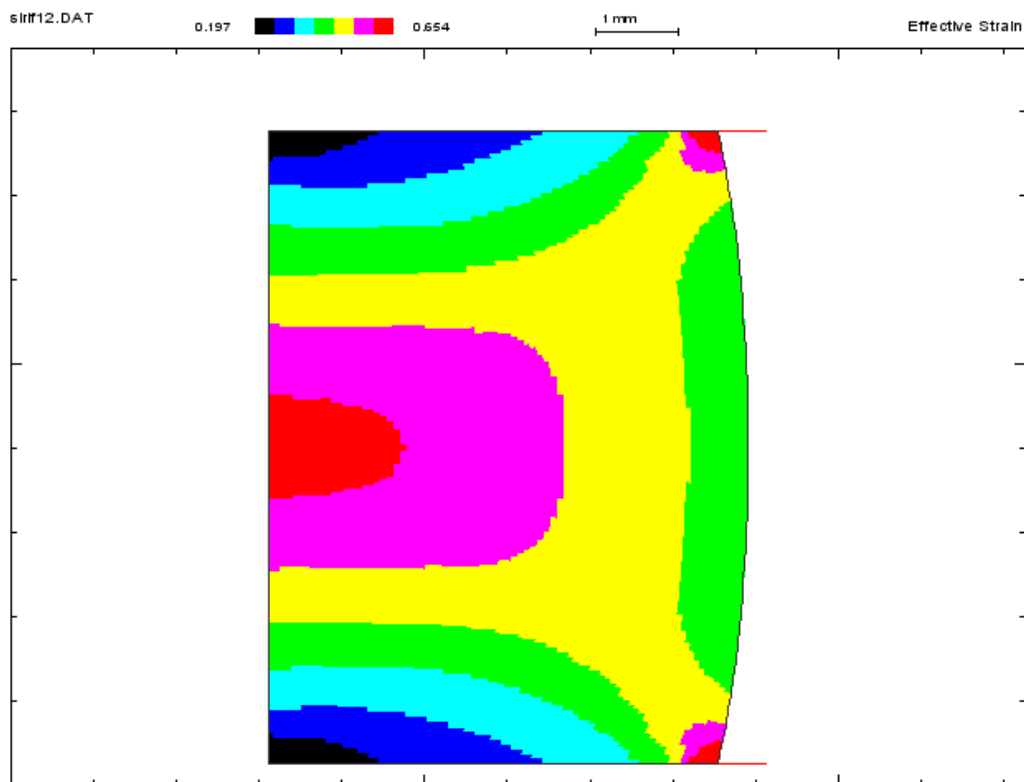


Figure 14 Contour plot for effective strain distribution for specimen S1.

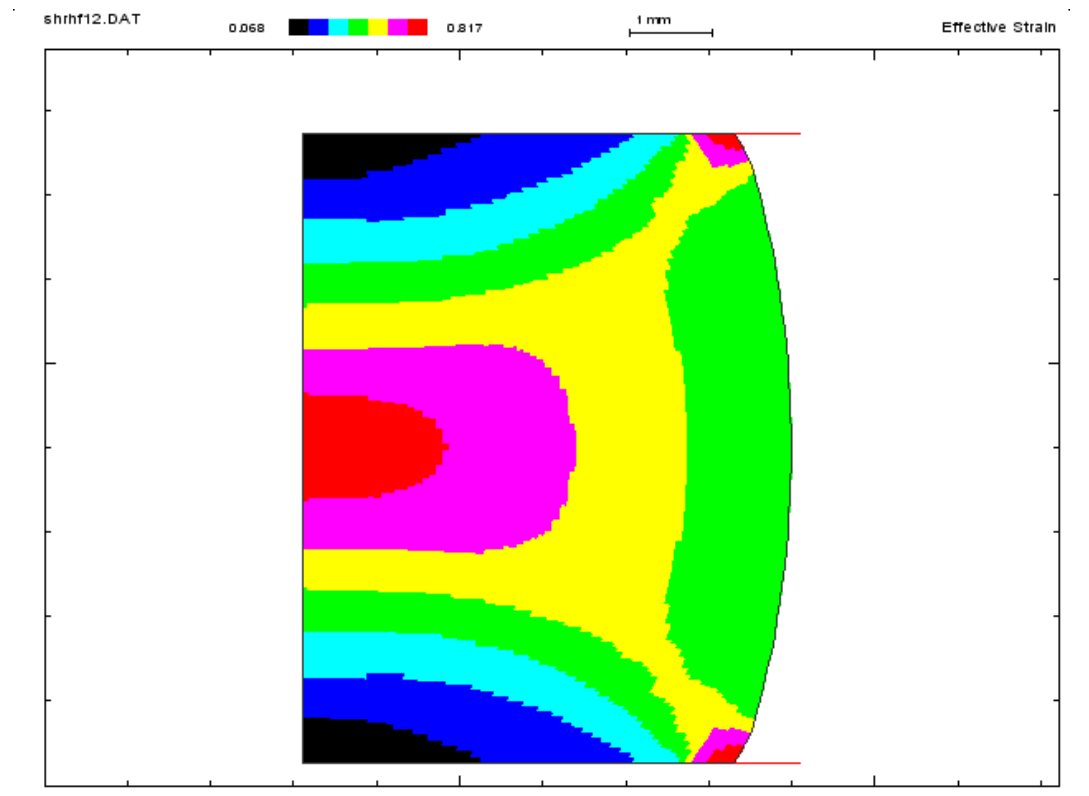


Figure 15 Contour plot for effective strain distribution for specimen S4.

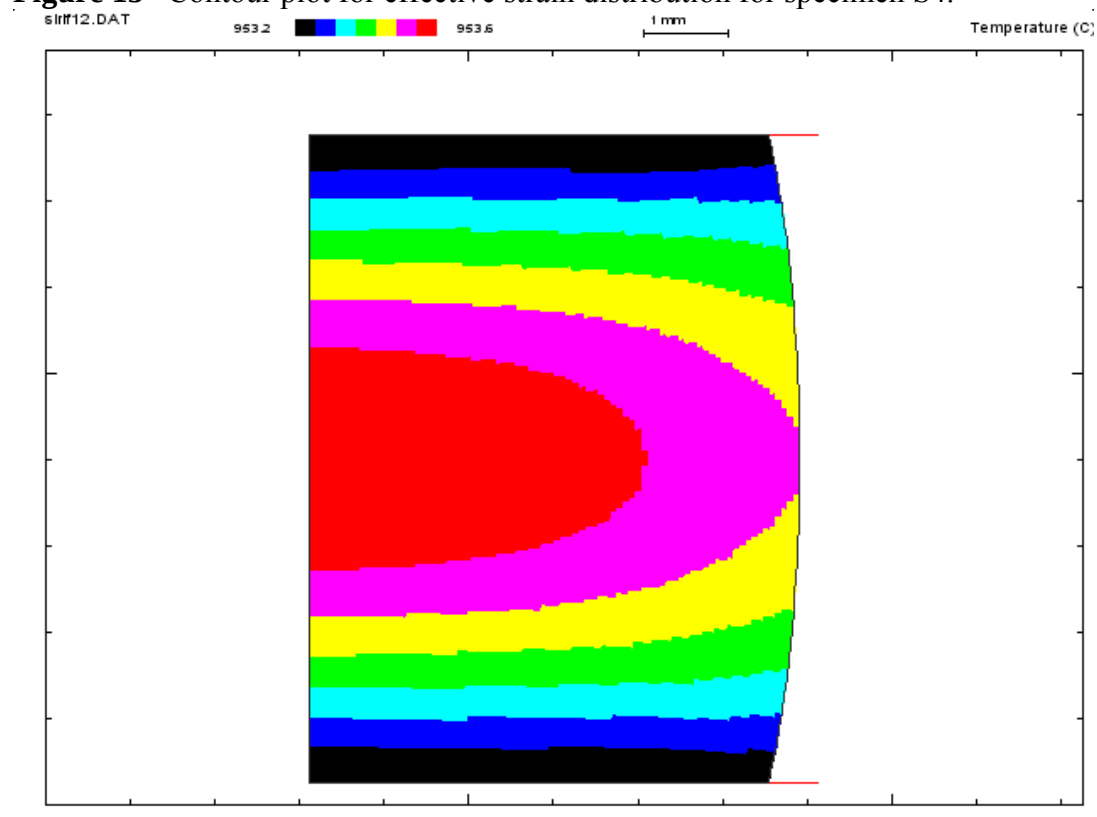


Figure 16 Contour plot of temperature at the end of deformation for specimen S1.

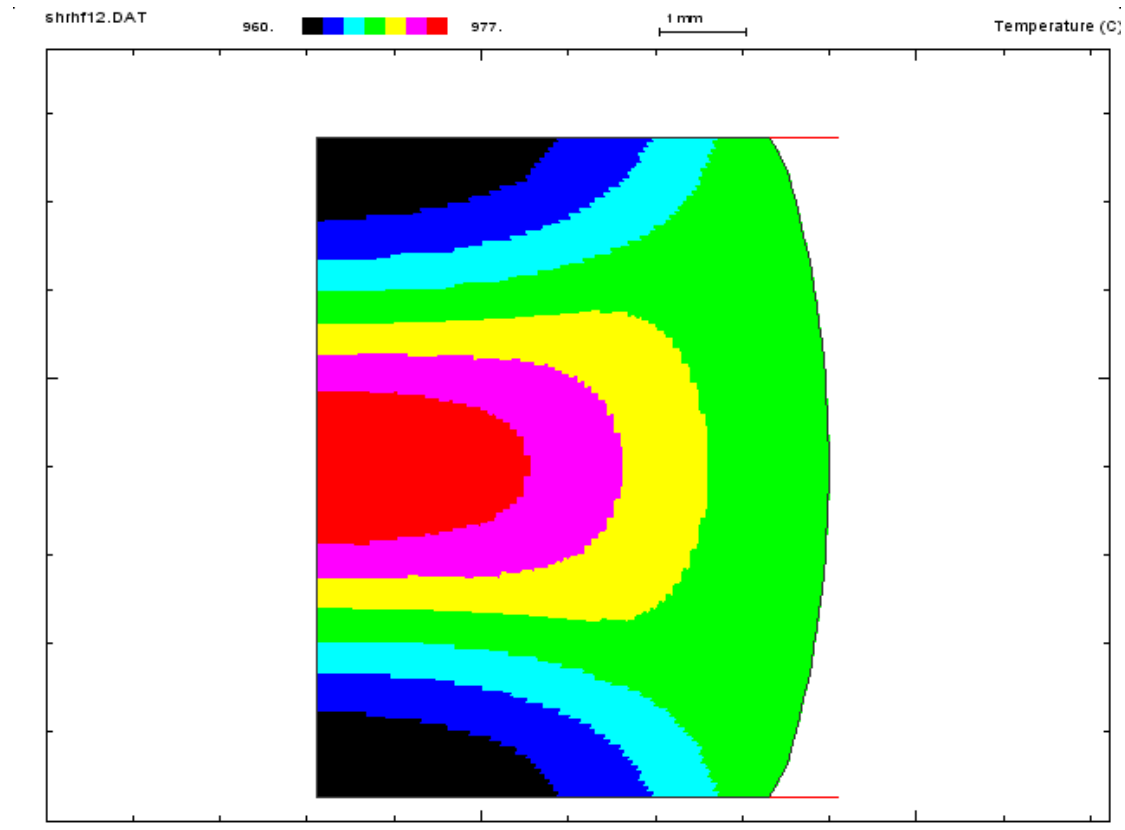


Figure 17 Contour plot of temperature at the end of deformation for specimen S4.

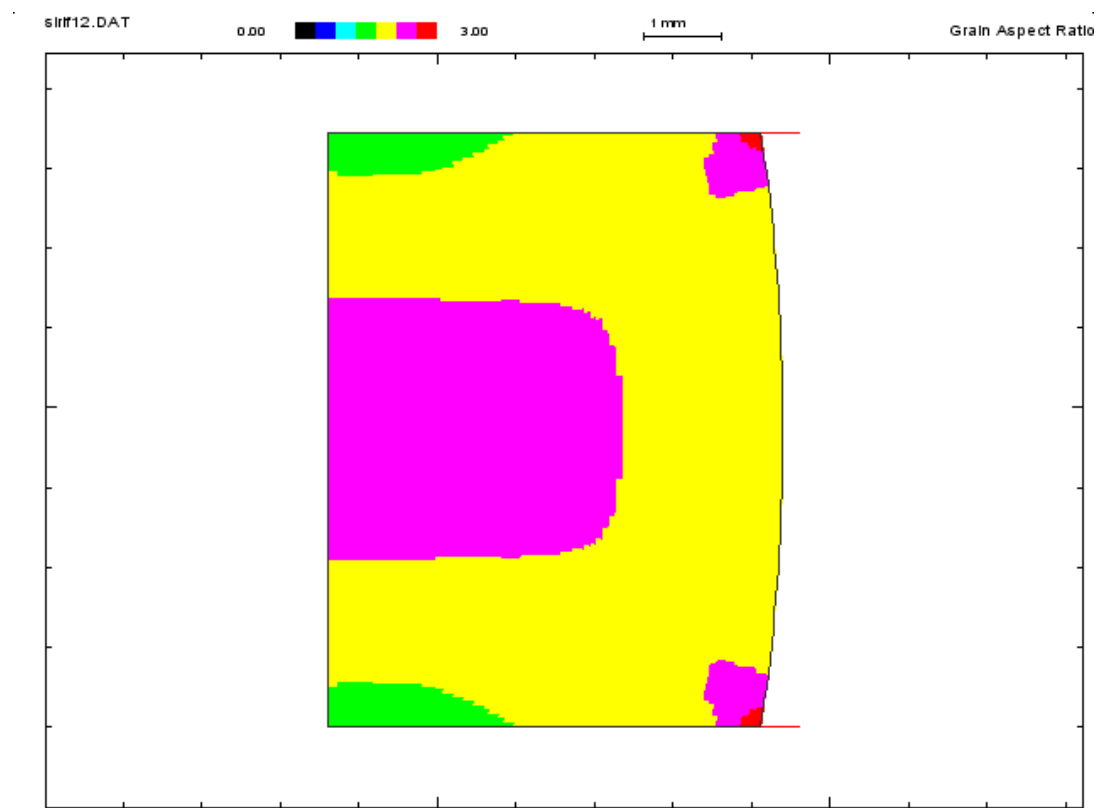


Figure 18 Contour plot of grain aspect ratio for specimen S1.

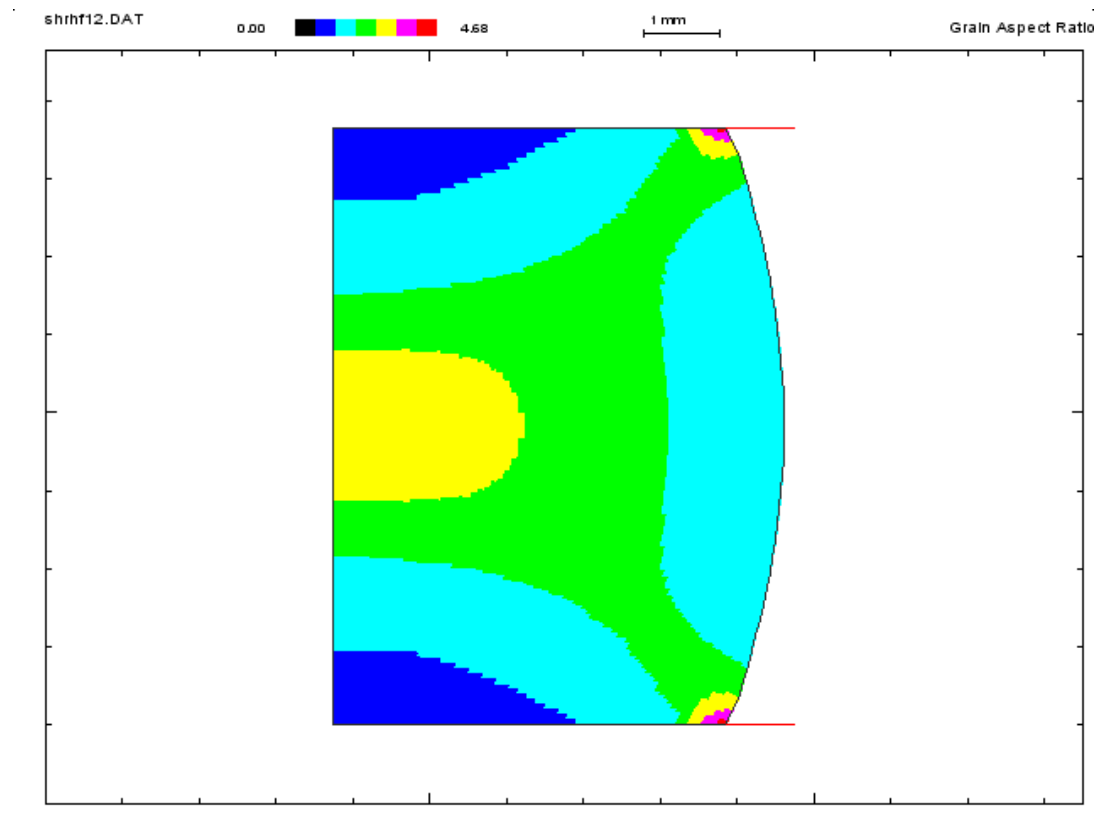


Figure 19 Contour plot of grain aspect ratio for specimen S4.

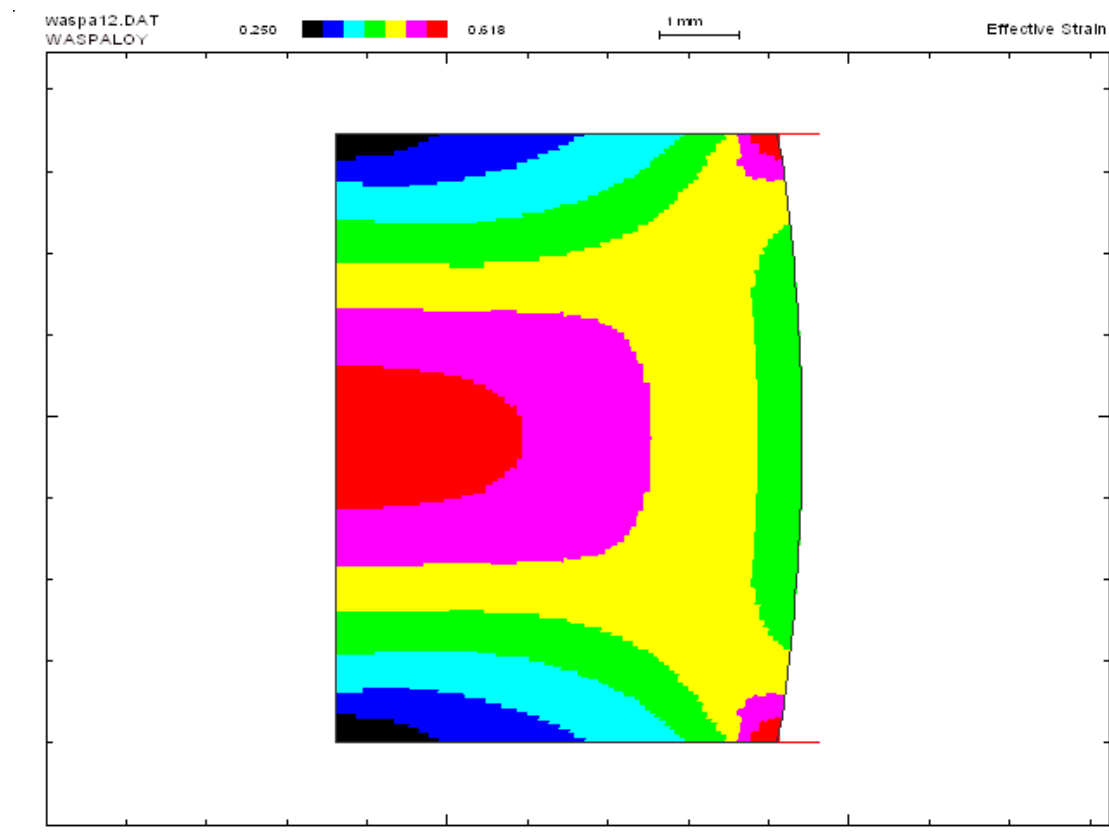


Figure 20 Calculated strain distribution for deformed sample W1.

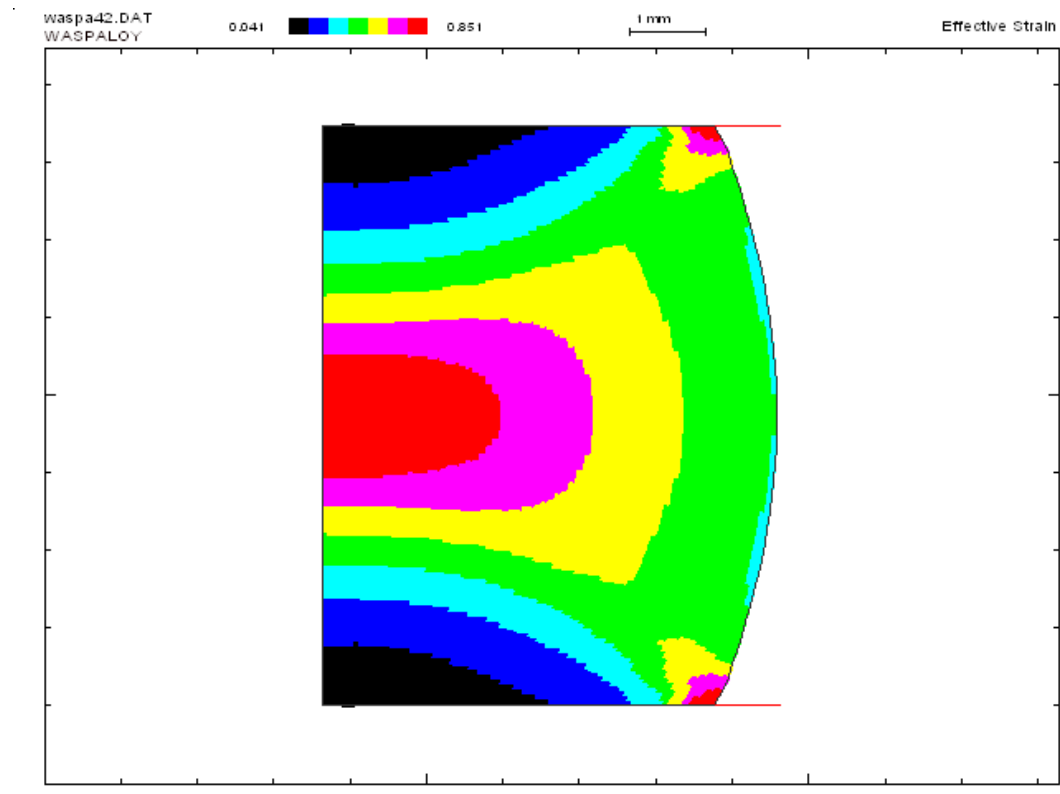


Figure 21 Calculated strain distribution for deformed sample W4.

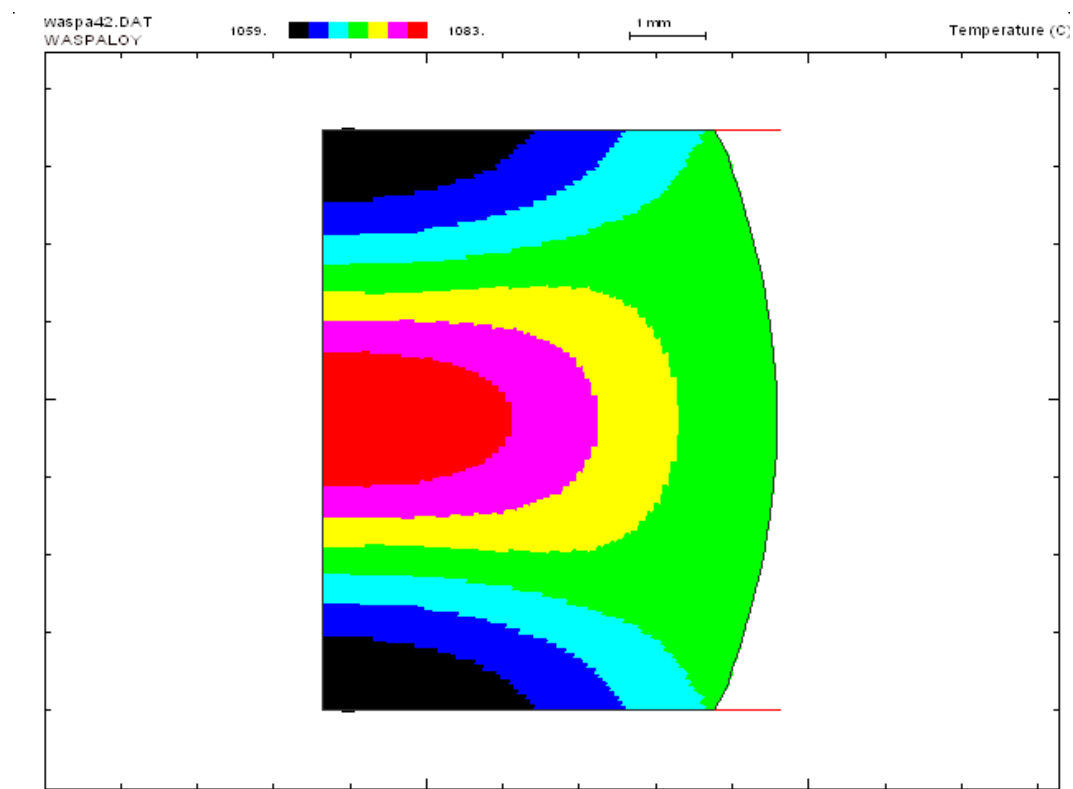


Figure 22 Calculated temperature distribution for deformed sample W4.

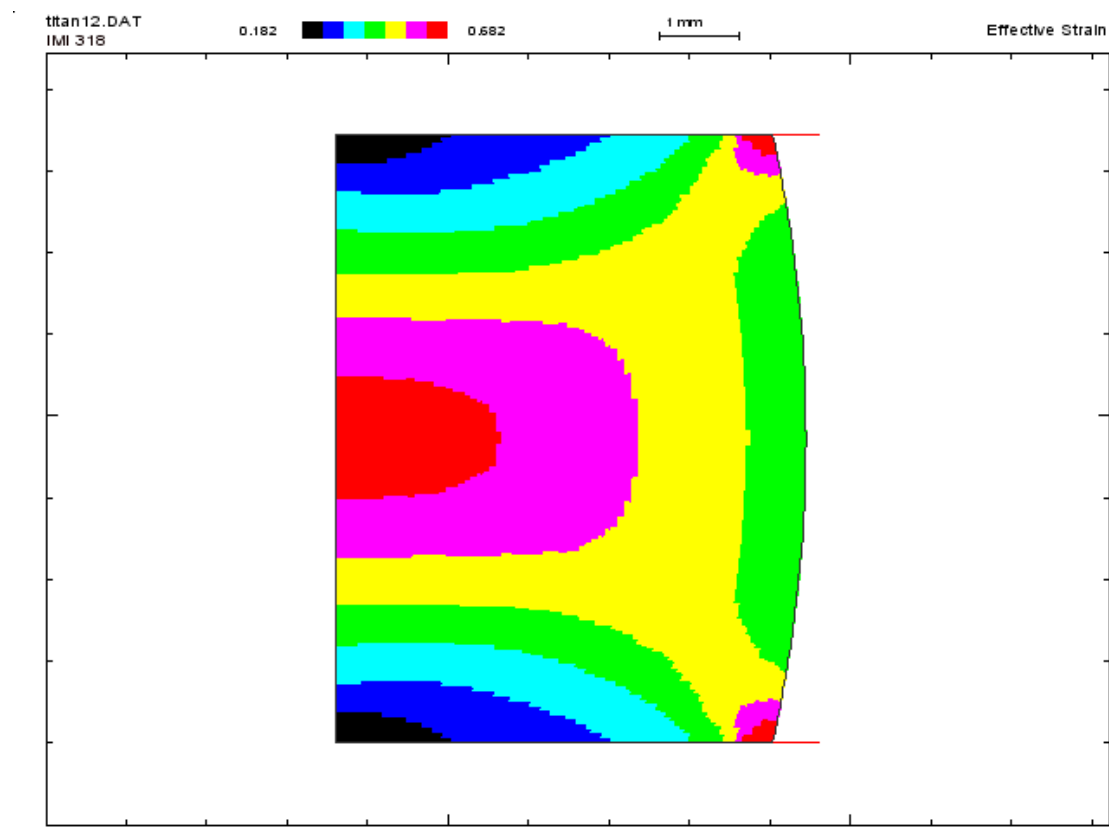


Figure 23 Calculated strain distribution for deformed sample T1.

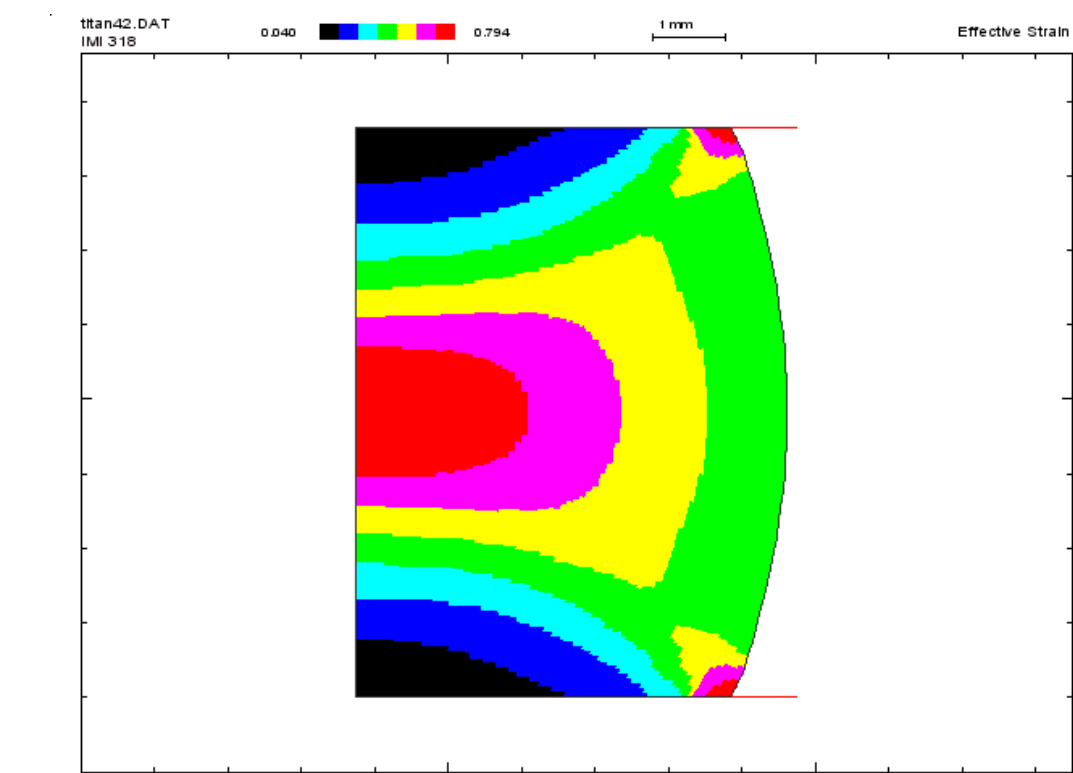


Figure 24 Calculated strain distribution for deformed sample T4.

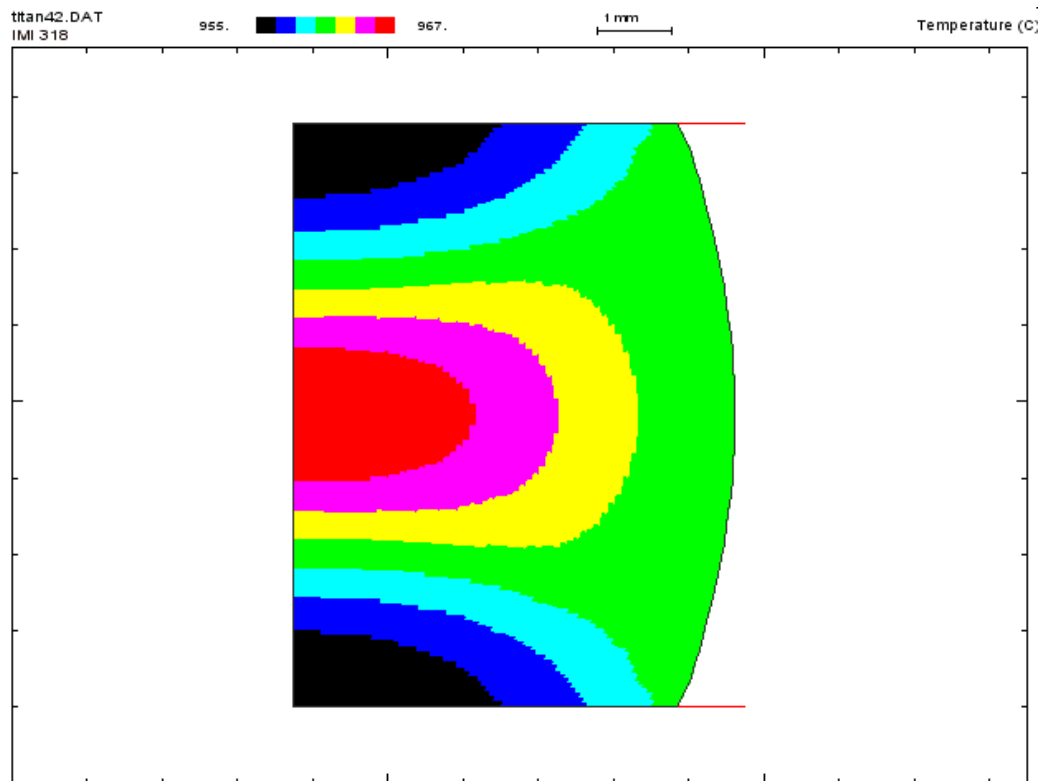


Figure 25 Calculated temperature distribution for sample T4.

9.5 PSC Model Studies

Although a large matrix of PSC testing conditions was modelled the full set could not be completed due to the untimely death of Prof. Russell Evans. However, before this setback a total of 2160 tests were completed as follows.

3 gauge lengths (mm)	20, 10, 5
8 w:h ratios	2.0, 1.681, 1.414, 1.189, 1.0, 0.693, 0.481, 0.333
5 friction factors	0.0, 0.05, 0.1, 0.2, 0.3
3 temperatures (°C)	989, 1050, 1125
6 strain rates (/s)	0.01, 0.1, 1.0, 10, 100, 1000

All simulations were taken to 70% strain where possible. Some tests failed to reach the larger strains without requiring a remesh and in these cases the simulation was not continued. For all the tests where 60% strain was achieved, contour plots of effective strain were drawn, along with a plot showing only those parts of the specimen with strains in the range $\pm 10\%$ of 0.6 compressive strain – i.e. strains of 0.53847 to 0.65813. In all cases, the strain in the samples is extremely inhomogeneous, and initial inspection of these plots would suggest that the areas within which metallographic measurements could be made which would be representative of 60% strain are very limited. A typical output showing strain variation is shown in Fig 26.

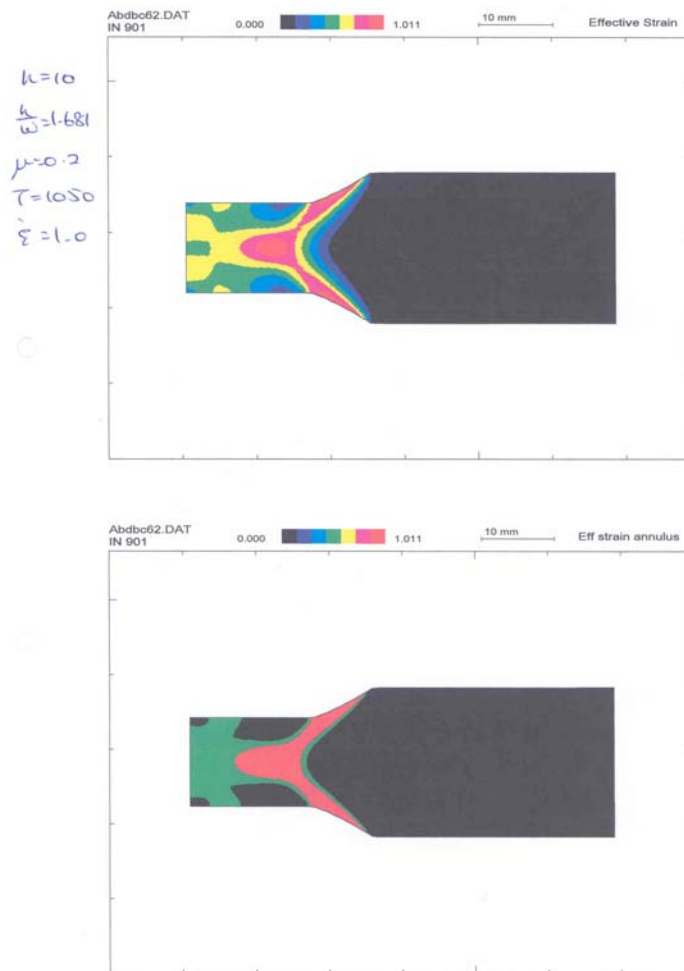


Figure 26 Modelled strain variation in a typical PSC test.

Before metallographic measurements could be correlated with internal strain conditions, it was considered necessary to know how individual points in the sample move during the plane strain test. To investigate this, data was obtained for a limited set of conditions in the first instance – one geometry only and one strain level only (60%). This was a matrix of 270 tests. Simulations were conducted which produced data files containing values of the strain rate at the centre of 48 elements for every time increment in the simulation. The elements chosen were along the centre of the sample, along the top edge, and mid-way between the two. At the time of Prof Evans' death work was in progress to examine the variation of strain rate with time; a polynomial fit was being investigated and the patterns of variation of the coefficients was being analysed. Clearly all the PSC model runs could not be completed.

However, the limited number evaluated do support the recommendation in Section 8 and Section 9 (AC tests) to fully examine strips of material at the positions indicated in Fig 2 and that good practice ideally should combine FE with large scale maps of structural changes.

Documentation of Results

10

IN THIS CHAPTER

- Report Structure and Content
- Uncertainty Assessments

10 Documentation of Results

10.1 General

Document the results in a suitable test report.

10.2 Assessment of Uncertainties

In 1994 an important new document was issued: 'Guide to the expression of uncertainty in measurement', published jointly by several authoritative standards bodies, namely BIPM, IEC, IFCC, ISO, IUPAC, IUPAP and OIML which is referred to here as the TAG4 Guide, after the ISO Technical Advisory Group 4 which drafted the document. It is a comprehensive document of over 90 pages based upon rigorous statistical methods for the summation of uncertainties from various sources. Its complexity has provided the driving force for a number of organisations to produce simplified versions of the TAG4 Guide, e.g. the National Institute of Science and Technology (NIST) in the USA, the National Measurement Accreditation Service (NAMAS) in the UK and the British Measurement and Testing Association, also in the UK. These various documents all give guidance of how to estimate uncertainty of measurement based upon an 'uncertainty budget' concept.

The total uncertainty of a measurement is determined by summing all the contributing components in an appropriate manner. All the contributions must be quantified. At a preliminary evaluation stage a decision must be made as to whether some contributions are negligible and can be excluded from subsequent calculations. For most practical measurements in the materials field the definition of negligible may be taken as a component smaller than one-fifth of the largest component.

The TAG4 Guide categorises two types of method for evaluating the uncertainties, A and B. Method A determines uncertainties from repeat observations and provided sufficient readings are available, conventional statistical analysis can be used to determine the standard deviation S . Method B uses alternative means, e.g. tolerances specified in standards, measured data, manufacturers specifications, calibration certificates, and from some basic knowledge of a simple model of the relationship between the various components. A combined uncertainty is then derived from estimates of A and B.

10.3 Test Report Content

It is recommended that the test report shall contain at least the following information:

- Reference to this procedure
- Material type and condition
- Identification of the testpiece
- Location and direction of sampling of testpieces
- Type of testpiece

- Preheating temperature profile
- Lubricant and platen materials
- Temperature and strain rate history of test
- Details of cooling procedures to retain high temperature microstructures through quenching.
- Platen temperatures
- Metallographic preparation methods
- Reference to procedures used to measure microstructural features; grain size and distribution, grain size heterogeneity, phase content, grain or phase aspect ratio.
- Results in pictorial, tabular or computer file format, and graphs when required.

Acknowledgements

11

IN THIS CHAPTER

- IAG
- Prof Russell Evans and colleagues
- Lin Sun, IMPPETUS
- Dr P Quested and Dr A Day
- DIUS (formerly DTI)

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A special thanks must also go to Prof Russell Evans (now deceased) and Penny Scharning from the IRC in Computer Aided Engineering in the University of Wales, Swansea. The initial drafts of Appendix A (Condensed Notes on Microstructural Metrology) and the optical studies for Appendix C were prepared by Penny and Russell. Furthermore, many of the supporting FE analyses were provided by these two academic colleagues. Thanks are also due to Lin Sun of IMPPETUS for contributions to the EBSD case studies when the previous student, Mark Thomas, left IMPPETUS to start work at TIMET UK, Ltd.

A great deal of the text in the Appendix concerned with uncertainties associated with EBSD measurements was taken directly, with a little modification, from the contributions of Drs Peter Quested (NPL) and Austin Day (formerly NPL and HKL Ltd, now Aunt Daisy Scientific Ltd) to a new ISO standard being developed for EBSD measurements.

The Good Practice Guide was produced as part of the DIUS (formerly DTI) Materials Measurement Programme, a programme of underpinning materials metrology research.

References

12

IN THIS CHAPTER

- List of Relevant Papers
- List of Relevant Standards

12 References and Other Related Procedural Documents

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12.2 Standards

The following referenced documents are *helpful in the use of this document*. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/DIS 23833, Microbeam analysis – Electron probe microanalysis (EPMA) – Vocabulary.

ISO/IEC 17025:1999 General requirements for the competence of testing and calibration laboratories.

MISC UNCERT (1995-01) Guide to the expression of uncertainty in measurement.

ISO/TS 21748:2004 Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation.

ASTM E3-2001 Standard Practice for Preparation of Metallographic Specimens.

ASTM E1558-1993 Standard Guide for Electrolytic Polishing of Metallographic Specimens.

ASTM E112-96 (2004) Standard Test Method for Determining Average Grain Size.

ASTM E1181-02 Standard Test Method for Characterising Duplex Grain Sizes.

ASTM E1382 – Test Methods for Determining Average Grain Size Using Semi-Automatic or Automatic Image Analysers.

ASTM E390 – Methods of Estimating the Largest Grain Observed in a Metallographic Section.

ASTM E562 – Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count.

EN 623-3 – Advanced Technical Ceramics – Monolithic Ceramics – General and Textural Properties. Part 3: Determination of Grain Size and Size Distribution (Characterised by the Linear Intercept Method).

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Appendix A

13

IN THIS CHAPTER

- Condensed Notes on Microstructure Metrology

Appendix A Microstructure Metrology

A1 Introduction

This condensed review was prepared as a background note for the experimental measurements conducted in the underpinning work on microstructural heterogeneity. It is included as an Appendix to the main GPG as it contains useful references and practical guidelines.

Quantitative metallography is concerned with the measurement of microstructural features such as grain size, as well as the size and spatial distribution of second phase particles from observations made on sections by optical or scanning electron microscopy. Knowledge of the accuracy of the experimental values of microstructural parameters is essential for their valid interpretation and application. Accuracy improves with the square root of the number of measurements taken, but the effort of making measurements increases linearly with their number. A compromise is therefore usually necessary [A1-A5]. The precision, reproducibility and repeatability of microstructural measurements are influenced by the variability of the material, the quality of specimen preparation and the test method. The sectioning plane cuts through grains anywhere between their major diameter and the apex of a corner. This effect makes the structure appear variable in size and shape regardless of the uniformity of the three-dimensional size.

Quantitative metallography requires considerable experience and skill, and is subject to inherent statistical variation. Manual methods are sometimes difficult and time-consuming, especially for complicated microstructures. The tedium of making measurements by hand is being increasingly removed by sophisticated image processing and image analysis software, but it is usually necessary to calibrate the systems for the particular alloy, the thermomechanically processed condition, and the metallographic preparation procedure to ensure that only the appropriate microstructural features are being examined [A1].

A2 Uncertainties in Measurement

It is important to establish the magnitude of the uncertainty associated with a particular type of measurement. An appreciation of the errors involved may be useful in deciding the most suitable technique for a particular task, and it also guards against metallurgical significance being attached to results which merely show acceptable experimental scatter. The estimation of grain size, for example, is complicated by a number of factors:

- The procedure used to reveal the grain boundaries for optical or SEM examination can be very selective in what it reveals. Some boundaries may be completely invisible, some may be incomplete or indistinct, and in other cases subgrain boundaries may also become visible. Proper detection of grain edges is of the utmost importance.
- Grain structures may be duplex, grain shapes may be distorted by processing procedures, and twin boundaries may be present.
- Even in a simple single phase material, the plane of measurement will contain a distribution of grain areas, varying from zero to a maximum value, depending upon where the plane cuts each individual grain.

Grain shape also varies, particularly as a function of grain size. The tetrakaidecahedron, whilst being an optimum space-filling shape, does not satisfy the required 120° dihedral angles between grains where three adjacent grains meet at an edge. The pentagonal dodecahedron agrees well with observations of grains, but is not a space filling shape [A3]. In addition to statistical uncertainties and measurement errors, there are a number of potential sources of systematic error that can arise from preparation of the sample or observation area, or from the metallographic technique [A1,A2].

Systematic uncertainties can arise from experimental measurement errors. Some common ones are:

- errors in calibrating the magnification.
- counting features that should not be counted, e.g. twin boundaries,
- omitting to count features that should be counted, e.g. unclear grain boundaries,
- misidentification of phases in a duplex structure.

These types of uncertainty also occur in automatic image analysis [A1] but can be avoided by experimental care and accurate image interpretation, which comes with experience and understanding.

All stereological measurements are estimates rather than absolute values. In a real material the grains have a range of sizes and shapes, and if the measurement is repeated several times, the same value may not be obtained every time. To assess the precision of a measurement, the mean and standard deviation should be calculated. The standard deviation of the data is expressed as

$$s = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n-1}} \quad (A1)$$

where $\bar{X}$ is the mean value of the individual measurements X_i and n is the number of measurements. Although the standard deviation is an absolute measure of data dispersion, it is difficult to compare standard deviations when the mean values differ substantially. It is then useful to normalise the standard deviations by dividing s by the mean and expressing this value as a percentage. This ratio is called the coefficient of variation, CV.

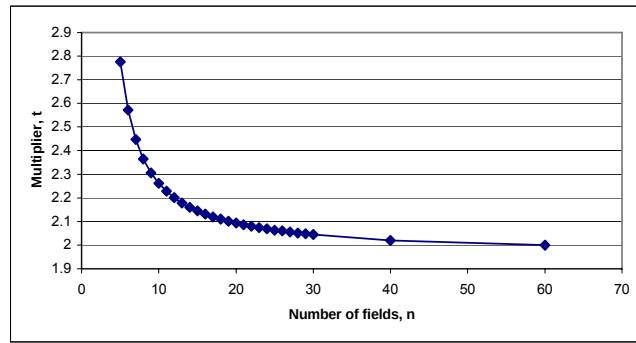
$$CV = \frac{s}{\bar{X}} \times 100 \quad (A2)$$

The 95% confidence limit, CL, is calculated using the relationship

$$95\%CL = \frac{ts}{\sqrt{(n-1)}} \quad (A3)$$

where t is known as a confidence interval multiplier. The value of t varies with the number of fields measured, but is often taken to be 2.0, which corresponds to 60 or more fields measured. Values of t for smaller values of n are given in the ASTM Standard E-562. Some values of t are:

Number of fields, n	Multiplier, t
5	2.776
10	2.262
20	2.093
30	2.045
40	2.020
60	2.000
∞	1.960



The percentage relative accuracy, RA, (also referred to as the relative confidence limit) is determined by dividing the 95% confidence limit by the mean value

$$\%RA = \frac{95\%CL}{\bar{X}} \times 100 \quad (A4)$$

For most measurements, a 10% relative accuracy is satisfactory. If a more accurate value is required (i.e. less than 10%), there is a simple formula to determine how many fields, n , must be measured to obtain a specific desired degree of relative accuracy at the 95% confidence limit:

$$n = \left(\frac{200}{\%RA} \times \frac{s}{\bar{P}_p} \right)^2 \quad (A5)$$

The most efficient procedure should be used for any measurement, i.e. the method that gives the greatest accuracy for the least effort. To determine the most efficient analysis method, the magnitude of the sampling errors must be determined. The standard deviation of the field measurements is used to quantify sampling errors. It is impossible to determine the population standard deviation, σ , but the standard deviation of the observations, s , approaches that of the population as the number of measurements N becomes very large. As N increases, s decreases. Thus, both the sample homogeneity and the number of measurements alter the standard deviation. For a given level of homogeneity, the number of fields required to obtain a reasonable accuracy must be determined. Each type of measurement requires a certain amount of effort to obtain a desired level of accuracy [A2]. All grain size determinations should indicate the precision to which the measurement has been made. “Normal confidence” is usually taken to represent the expectation that the actual error will be within the stated limits 95% of the time. This is known as the confidence limit when expressed in the same units of measurement as the grain size itself. Many specimens vary measurably in grain size from one field of view to another, and when the natural variability is higher than normal it is desirable to indicate the range of sizes present as well as the precision of the average size determination [A4].

A3 Specimen Preparation

The sample should be representative of the bulk, but in cases such as castings and forgings where the structures are heterogeneous, this may not be easy. Careful consideration needs to be given to the purpose of making the measurements in order to choose the best sampling

position. The size of the sample must be sufficiently large to ensure that no spurious effects are introduced from the surfaces [A1]. Polishing and etching must reveal the true microstructure without distortion, smearing, gouging, etc. Automatic polishing equipment is recommended to obtain maximum flatness, control of relief and reproducibility of polishing quality [A2].

A4 Magnification

The magnification used and the number of fields or features assessed affects the end results. As the magnification increases and the field size decreases, the field-to-field variability increases due to the inherent heterogeneity of the microstructure. Also, each field samples less of the structure. Choosing a magnification is always a compromise between resolution and variability, but the lowest possible magnification is to be preferred.

A5 Grain Size Measurement

A5.1 Manual Methods

Conventional methods of measuring grain size are based on point counting and linear intercept measurements. The average grain size of a specimen with a single distribution of grain diameters (unimodal) can be estimated by the chart comparison method or measured by planimetric or intercept methods. ASTM standard E-112 covers this unimodal distribution, whereas E-1181 covers measurement of specimens with bimodal or ‘duplex’ grain size distributions. Newer methods of measurement currently under assessment include ultrasonics, image analysis, electron backscatter diffraction and colour orientation contrast imaging. The ASTM standard E-1382 addresses image analysis measurement methods.

The ASTM grain size scale, adopted in 1951 (ASTM E91), defined the ASTM grain size number, G , as

$$n = 2^{G-1} \quad (\text{A6})$$

$$\text{or,} \quad G = \frac{\log n}{\log 2} + 1 \quad (\text{A7})$$

where n is the number of grains per square inch at $\times 100$. Other countries have also prepared standards for rating grain size, based on the metric system, using the number of grains per square millimetre at $\times 1$. The Swedish, Italian, Russian, French and ISO standards defined a grain size number, G_m , according to

$$G_m = \frac{\log m}{\log 2} - 3 \quad (\text{A8})$$

where m is the number of grains per square millimeter at $\times 1$. Grain size numbers produced by this method are about 4.5% higher than those calculated by the ASTM standard. Both the German and Japanese specifications produce grain size numbers equal to the ASTM number [A2].

A5.2 Planimetric Method

The planimetric method, developed by Jeffries in 1916, is based on the determination of the number of grains per unit area, N_A , which is directly convertible to the ASTM grain size number, G . In this method, the number of grains that are completely within a test circle, and the number of grains intersecting the circle, are counted. The test circle is typically 79.8 mm in diameter (5000 mm²), and the magnification should be adjusted to provide at least 50 grains within the measurement area [A4]. Accurate counting requires physical marking of the grains. This makes it rather slow when done manually [A5]. If the number of grains completely within the area is n_1 and the number which intersect the perimeter of the test area is n_2 , there are a total of $n_1 + n_2/2$ grains. This total is multiplied by the Jeffries factor f for the magnification employed, to obtain an estimate of the number of grains per square millimeter at $\times 1$, N_A , as

$$N_A = f \left(n_1 + \frac{n_2}{2} \right) \quad (\text{A9})$$

The value of f for any magnification M can be found using

$$f = \frac{M^2}{5000} \quad (\text{A10})$$

The ASTM grain size number, G , can be calculated from N_A using

$$G = \frac{\log N_A}{\log 2} - 2.95 \quad (\text{A11})$$

A5.2.1 Circle Equivalent Diameter

More recent methods of measuring microstructural changes relying on the use of image analysis software or electron backscattered diffraction (EBSD) can generate data on the 2-D grain areas intersected by the plane of polish. These grain areas can be converted to a circle equivalent diameter, d_{ceq} , i.e. a linear parameter equivalent to the diameter of a circle of equal area to that measured for each grain. These measurements are increasing in popularity because they can provide useful information on the distribution of grain diameters as well as a mean value.

A5.3 Intercept Method

The intercept method, developed by Heyn in 1903, is based upon the average length of line segments within each grain using a line of known length placed over the structure. Straight lines, curved lines or test circles may be employed. One counts either the number of grains intercepted by the line, N , or the number of grain boundary intersections with the line, P . For a single phase structure, N will differ from P by one at the most, and either counting method can be used. The mean lineal intercept length, $\bar{L}$, is the reciprocal of the number of intercepts or intersections per unit length of test line, N_L or P_L . An accurate count does not require marking of the test template so the method is faster than the planimetric method when done manually [A5].

The method is ideally suited for measuring both equiaxed and non-equiaxed grains. The line length and magnification should be chosen to produce from 50 to 150 intersections per field to obtain an accurate estimate. If grain boundary intersections with the line are counted (rather than the number of grains), then grain-boundary intersections are counted as one point, triple-point intersections are counted as 1½ points and tangent hits as 1 point. The end points of the lines are not intersections and are not counted except when the end appears to exactly touch a grain boundary, when a ½ point should be counted. These counting rules apply to single-phase structures only [A4].

If L_T is the total line length and M is the magnification, then

$$N_L = \frac{NM}{L_T} \quad \text{and} \quad P_L = \frac{PM}{L_T} \quad (\text{A12})$$

For single phase structures, $N_L = P_L$, and the mean intercept length, $\bar{L}$, is then given by $1/N_L$ or $1/P_L$. The mean lineal intercept, $\bar{L}$, is less than the average diameter of the grains, which in turn is less than the maximum grain diameter. The average grain diameter measured from the Jeffries (planimetric) method is about 12.5% greater than that measured by the linear intercept method.

For $\bar{L}$ in mm, the ASTM grain size, G , is calculated by

$$G = [-6.6457 \log \bar{L}] - 3.298 \quad (\text{A13})$$

If enough fields are measured, the grain size obtained by the intercept method is accurate to one tenth of a unit of G [A2].

Full details of the experimental methods for measuring grain size by the intercept method are given in ASTM Standard E-112. This standard gives the required line lengths and circle circumferences which should be used to achieve specific accuracies [A4].

A5.4 Comparison Method

In the comparison method, the microscope image is compared with a set of standard micrographs of known grain size. The standards are available as wall charts, clear plastic overlays or as reticles, and each chart specifies the magnification to be used. The operator scans the specimen while viewing the standard pictures and picks the standard image most similar in grain size to that of the unknown. The method is simple and fast but not as accurate as the direct measurement methods. Grain size ratings by this method are generally accurate to within one ASTM grain size number if the structure is uniform and equiaxed [A2,A5]. Grain size distributions should not be attempted using this method. Individual grain measurements should not be made based on the standard comparison charts. The charts were constructed to reflect the typical log-normal distribution of grain sizes that result when a plane is passed through a three-dimensional array of grains, and they thus show a distribution of dimensions [A4].

It is best to view the sample at the same magnification as the standard, but other magnifications can be used. If the magnification M is greater than the chart magnification M_b ,

the true grain size number will be greater by a factor Q than the apparent grain size number. Suppose that a magnification of $\times 200$ is used with an ASTM chart designed for $\times 100$ and the apparent grain size number for the sample is ASTM 8. The value for Q is given by

$$Q = 6.64 \log \frac{M}{M_b} \quad (\text{A14})$$

In this example, M is 200 and M_b is 100, hence Q is 2. Thus the ASTM grain size is $8+2 = 10$. Using higher magnifications than M_b can be very useful for rating fine-grained samples and can also be used to obtain grain size ratings outside the range of the chart pictures [A2].

A6 Image Analysis

The essential problem with digital image analysis is that the features of interest must be well defined, either by edges, or unique brightness, or colour. The ability to record patterns using digital image analysis requires a special understanding of sample preparation and grey level image processing. Modern digital image analysers use a microcomputer to process digitised images using a range of software for enhancement, measurement and analysis. The process is interactive, and the operator usually uses his own experience and knowledge about the image to relate objects to one another and to the background [A6].

The main operations of an image analysis system are

- Image acquisition.
- Image processing
- Image segmentation
- Data analysis.

A6.1 Image Acquisition

The steps involved in acquiring and processing the data are mutually dependent [A7]. The process of image acquisition consists of recording the signal intensity for each pixel (picture element) within a digital image. The source of the signal is typically a digital camera mounted on a light microscope or a detector mounted in an SEM. The information within an image is carried by the intensities of the individual pixels. In a typical system, each pixel can have one of $2^8 = 256$ discrete values or grey levels, from 0 to 255, white being 255 and black being 0. However, 1024, 2048 and 4096 pixel systems are now being produced. The greater the number of pixels, the more accurate the detail that can be analysed. This is especially important for quantitative determination of edges, where a large number of pixels is required to exactly determine the edge location. However, the greater the number of pixels and the finer the grey level digitisation, the larger the amount of data that is obtained, and memory and file sizes for storage can become prohibitively large [A6].

A6.2 Sample Preparation

Sample preparation is critical. Images should be acquired in as consistent a manner as possible, and techniques for preparing specimens need to be selected with this in mind [A7]. The human eye can accommodate subtle differences in sample appearance (associated with poor sample etching, scratches, dust etc.) which a video camera cannot. Under these

conditions quantitative definition becomes impossible. In addition, sample preparation which has removed inclusions, rounded edges or smeared structure can lead to large quantitative differences in microstructural size, area and other attributes that are the major reasons for using image analysis. 'Perfect' metallographic sample preparation is essential when video microscopy and image analysis are used for quantitative feature analysis. Further, since the human eye can distinguish less than one quarter of the grey levels seen by a video camera, the image analysis system has much more information visible to it than the operator, and conventional sample preparation may then not be acceptable [A6].

A6.3 Reference Standards

Reference standards play an important role in establishing reproducible intensities for both scanning electron and light microscopy. If intensities are set by the operator without the use of reference standards, another level of uncertainty is added to the process. A reference standard for use with the SEM consists of polished magnesium, a portion of which is overlaid with a thin layer of silicon. The contrast and brightness settings are adjusted to obtain reproducible intensities for the two materials on the standard. For light microscopy, different coloured resins may be used [A7].

A6.4 Sources of Distortion

It is important to ensure that both the illumination and the signal collection parameters are consistent within and between images. Each time an image is obtained it is necessary to ensure that the microscope is perfectly aligned having uniform optics, and is free from dust and contamination. Electronic noise can always be present in an image and often requires that several fields be averaged so that the final signal is well above the noise threshold of the system. The lighting in a microscope is rarely uniform, and while the eye can accommodate non-uniformities, an image analyser cannot. Software is available that allows the operator to subtract the non-uniformities from the lighting to approach a more uniformly lit image. This is important for analysis of subtle grey level changes [A6].

A6.5 Image Processing

Once an image has been acquired, features and their distinguishing characteristics must be identified. The term image processing covers a variety of techniques that are used to enhance or modify contrast, find and enhance edges, clean images, etc. Many image processing techniques are used because of a lack of clarity in the signal, thus it is important to obtain the clearest possible signal in the first place.

A6.6 Contrast and brightness

Contrast and brightness are, respectively, the relative and absolute values of the pixel intensities [A7]. The range of intensities within an image can be represented using a grey-level histogram. The grey level of an image is rarely optimal – regions of interest may be too dark or too bright, or contrast may be too low. Software exists which allows the operator to alter the normal histogram of grey levels to form a new distribution. The grey level distribution in an image is optimum when the image completely fills the 0-255 range [A6].

The operator generally utilises histogram alterations to help improve the grey levels of the image and edge sharpening routines to help locate the 'exact' position of boundaries of

interest. The process of histogram and, thus, image modification is normally used to aid the human eye in distinguishing individual features.

A6.7 Thresholding

The process of identifying features by distinguishing discrete ranges of grey level associated with the features can involve thresholding, density slicing or binary segmentation. Each term has a slightly different meaning [A7]. Proper setting of the threshold detection is somewhat subjective at times and is a major source of error and lack of reproducibility [A2].

Thresholding includes the other two procedures, and involves the establishment of specific grey levels, or thresholds, so that features exhibiting intensities between two thresholds are assigned to the same class. Density slicing is a similar process in which limits on grey level are selected, and the grey levels of features outside those limits are set to black. Binary segmentation involves the selection of two categories of features lying on either side of a single threshold. This is a very important step in ensuring quantitiveness, since it requires quantitatively locating boundaries of interest and forming a binary (black on white) image of the different structural features that are to be analysed in the image [A6]. Binary segmentation works well for two-phase images [A7].

A6.8 Filters

Edge detection plays an important part in analysis. The problem of edge detection arises from the intermediate values of grey level that are measured for the pixels on a feature boundary. The ability to accurately identify an edge can significantly improve the accuracy of an analysis. As the size of the pixel relative to the size of the feature increases, the ability to detect edges is degraded. Careful detection during the image processing stage is critical to developing a consistent representation for feature edges [A7]. Edge enhancement filters can be an important consideration here. Laplacian filters are based on calculating second order derivatives – they indicate the rate of change of grey scale all over the image boundary. Gradient filters are more directional since they only affect one portion of the image. In addition to enhancing boundaries and edges, these filters also remove slowly varying background shading. They may also enhance noise [A6].

A6.9 Image Segmentation

The objective of all image enhancement is to produce the most suitable image which can be segmented into well defined meaningful regions and image features for later analysis. The most important segmentation procedure is to divide the image into specific bands of unique grey value and use thresholds to obtain boundary outlines. In this method, a binary image is created for each grey value of interest. The usual procedure is to make the region of interest white, but it does not really matter as long as the region of interest is accurately discriminated from the background [A6].

Discrimination is not always easy, and since it is the most critical part of the image analysis process it is especially important to understand where there can be errors. The most important problem is of truly finding the boundary between the region of interest and the background [A6]. A major difficulty is to define the grey levels that are used in setting the threshold so that the width of the binary object is exactly equal to the width of the grey level image.

Quantification depends on an accurate match. The best method is to overlay the binary image onto the grey level image and observe the closeness of fit [A6].

Errors can occur if there are differences in size between images of the same grey level. There is no problem with the grey level of a larger object being properly recorded; however, with a small object there may be a problem. By the time the scanning spot has reached its full height, it has started to move into the surrounding material and the fully digitised grey level of the small object is not attained. Errors in thresholding can also exist when comparing similar sized objects of slightly varying or almost comparable grey levels [A6].

A6.10 Feature Recognition

Specific features for measurement next have to be defined and extracted. The operator determines which features are important to measure. Many commercial image analysis programs have the ability to identify and catalogue the properties of surface features once thresholding has been completed [A7].

However, additional steps may be required for feature recognition even once features have been identified. There may be regions of a feature that are not correctly assigned because of artefacts in the image or variations in the signal due to noise, or even the basic nature of the imaging process. The operator may then have to use steps such as erosion (removal of pixels on the perimeter of a feature) or dilation (addition of pixels) to obtain better feature definition, or apply other techniques to join portions of features that should be treated as contiguous, or separate agglomerations of particles that appear to touch [A7].

A6.11 Data Analysis

Image analysers allow the operator to make a wide range of measurements that characterise the image. Such measurements are usually divided into three broad categories: field specific, chord specific and object specific. Field specific measurements include details about the entire field of view, including number of objects in the field, volume measurements, area fraction of the discriminated objects. Object specific properties include the distribution of object characteristics, such as areas, perimeters, diameters, aspect ratio and orientation [A6].

A6.12 Application to Difficult Structures

The measurement of average ASTM grain size in a ferrous material was one of the earliest applications for image analysis in the metals laboratory. Various methods have been used by different manufacturers to provide this data with their systems. However, all the systems depended upon having an image possessing a high degree of contrast between the lighter polished grains and the darker grain boundaries. As such, the ability to accurately analyse the material was highly dependent on the quality of the sample preparation. No residual scratches should remain, and the grain structure must be accurately revealed. This can be difficult to achieve, as often the boundaries are not completely revealed, and in other cases there is a surplus of sub-grain boundaries such as twins. This requires manual modification of the image prior to analysis. These manual techniques, using a light pen, track ball or mouse are very time consuming. In addition, a high degree of operator familiarity with both the material structure and the operation of the image analysis equipment is required. to successfully modify the image [A8].

Not all materials, even when properly prepared and etched, exhibit a high degree of contrast between grains and grain boundaries. The boundaries between adjacent grains are determined more by inference between areas of slightly different grey values [A8].

Most modern image analysis systems contain methods for transforming the image into a form that is more easily analysed. These processes are frequently referred to as 'transformations', 'convolutions', or 'grey level operations'. These transformation functions allow the image to be viewed on a pixel by pixel basis. Each target pixel is viewed with regard to the 8 (3×3 matrix) or 24 (5×5 matrix) pixels that surround it. A series of mathematical operations is performed on these surrounding pixels to determine the new grey level of the target pixel. The value of this new pixel is then assigned to a new image. This process is repeated on a pixel by pixel basis for all pixels in the incoming range. From these data, a completely new image is constructed in temporary memory in the image analysis system. When finished, this new or 'transformed' image is displayed on the monitor screen.

Once the raw grain boundary structure has been revealed, a further series of image processing steps can be undertaken to perfect the image, thin the boundaries, and reconstruct some boundaries that were inadequately revealed. Thinning operations can be performed to reduce thick boundaries to picture point thick lines. An image inversion operation can then be performed so that the bodies of the grains are detected instead of the grain boundaries. Separation techniques reduce detected areas to a single or a series of centres of gravity and then expands from these centres of gravity as separate detected particles to their current boundaries. This effectively re-establishes these separately detected grains while 'drawing in' new boundaries where logic dictates that they should occur. While not infallible, this process results in a much more accurate rendition of the original image and an automatic and accurate estimation of the average of the ASTM grain size value of the field. Once the sequence of image processing steps has been established, the analysis can be performed equally well by any operator [A8].

Once an image has been processed into a form suitable for analysis, the ASTM grain size value for each individual grain can be determined, hence making the grain size distribution available. This data can be rapidly accumulated over a number of fields of view on the same sample for greater statistical accuracy. In addition, the image can be colour-coded to correspond to size ranges of ASTM values. This gives a rapid estimation of the distribution of grain sizes in the field of view, and allows further classification as to the number and area percentage of each grain size range [A8].

Fully automated systems may fail to count some boundaries when they are of low contrast or unusually thin, but they may also count intersections with inclusions and second phase particles even in a single phase material. The fraction of such false counts must be periodically determined and kept within acceptable limits. These errors should individually be held within 5% of the count for confidence that the specimen average is accurate to the nearest 0.5 ASTM number and within 1% for confidence to 0.1 ASTM number. If the additional counts due to inclusions cannot be kept to this level, the material should be treated as two-phase instead of single phase [A4].

A6.13 Measuring Procedures and Analysis of Results

Once a satisfactory image has been obtained, the methods of measuring the grain size and grain size distribution are the same as those given for manual methods, i.e. using either grain

counting (planimetric), linear intercept, or grain area methods. ASTM Standard E-1382 outlines the proper procedures for these methods, and also gives examples of both suitable and unsuitable microstructures [A9]. The analysis of the results is also the same as for manual methods.

However, if completely automatic image analysis is being used, extra care must be taken when using the grain intercept lengths or grain areas to estimate the grain size, to delete from the image all those grains that intersect the test area border in order to eliminate edge effects. Failure to delete these grain will produce measurement bias, which will increase as the magnification is increased, as a greater proportion of the grains will intersect the border. If the number of grain boundary intersections per unit length of scan line or the total length of grain boundaries per unit area is to be determined, it is not necessary to delete the grains that intersect the border, so long as the field contains a large number of grains. Particles within the grains should also be removed before measurement [A9].

A7 Comparisons of the different measurement techniques

The most efficient method for measuring grain size is always the one which gives the greatest accuracy for the least effort – and effort generally equates to time taken.

The Jeffries method is much slower than the intercept method because of the need to keep track of the grains being counted. However, for a single measurement field, the Jeffries procedure produces a lower standard deviation. For equal accuracy, the Jeffries method requires about 60% more time than the intercept (Heyn) method. Working to an accuracy of 0.5 ASTM unit, the intercept method takes no longer than the comparison method.

A round robin was conducted by ASTM Committee E-4 on Metallography in the early 1990s on these three different manual grain size measurement techniques. It was found that the planimetric and intercept methods gave excellent agreement with no bias. However, the comparison method showed a consistent bias, with the comparison method giving an apparently coarser grain size (ASTM grain size number G is larger).

An assessment of results obtained in an analysis of a stainless steel using manual methods and computer image analysis [A10] showed that although both methods led to similar material classification, manual measurement overestimated the grain size as compared to computer analysis in the majority of cases. The manual measurements, conducted by six independent workers, were made from printouts of the images used for image analysis so that the structures measured were identical. The number of grains was determined by using the Jeffries method (number of grains per unit area) in all cases. For the image analysis investigation, four different algorithms were developed for processing the image to enable a grain edge to be determined uniquely. Each different algorithm was found to lead to some errors, either with missing edges or excessive division, but as only about 1% of boundaries were incorrectly detected, their effect on the final result was considered to be negligible. The analysis of the final image was carried out in the same way for all four algorithms. Although the computer algorithms can take a considerable time to set up initially, they can then be run fully automatically, thus saving a considerable amount of operator time when compared with manual methods. An established algorithm is also fully repeatable, whereas different results are likely to be obtained every time a manual measurement is repeated [A10]. For eight of the twelve samples, manual methods gave obviously larger mean grain areas, whereas for the

other four samples, there appeared to be little difference in the measured results, with the maxima and minima for the manual measurement falling within those for image analysis.

A8 Grain size of non-equiaxed structures

When the grain shape is noticeably distorted, size measurements should be made on the transverse, longitudinal and planar (rolling plane) surfaces with respect to the deformation direction [A2].

If a Jeffries count of the number of grains per square millimetre at $\times 1$ is made on the three principal planes yielding values of $N_{A,t}$, $N_{A,l}$ and $N_{A,p}$, the average value of the number of grains per square millimetre is given by

$$\bar{N}_A = (N_{A,t} N_{A,l} N_{A,p})^{1/3} \quad (\text{A15})$$

and the ASTM grain size, G , is calculated from equation (A11).

If the intercept method is used, straight lines are preferable to a circle. The average $\bar{N}_L$ value is obtained by

$$\bar{N}_L = \frac{1}{3} (N_{L,t} + N_{L,l} + N_{L,p}) \quad (\text{A16})$$

This value of $\bar{N}_L$ is used to calculate $\bar{L}$ as $1/\bar{N}_L$ and the ASTM grain size is calculated from equation (A13).

A9 Grain size of duplex structures

The term duplex structure can cover two situations:

- Material containing a single phase with grains of two or more significantly different sizes. Assigning an average grain size value to a duplex grain size specimen does not adequately characterise the specimen, and may even misrepresent its appearance. Averaging two distinctly different grain sizes may result in reporting a size that does not actually exist anywhere in the specimen [A10].
- Material containing two or more phases, which may or may not have similar grain sizes.

Two main types of duplex grain size structures can be observed.

1. Agglomerations of small grains within a matrix of large grains, or vice versa. This case includes necklace structures and specimens showing banding or a systematic variation across the section [A10].
2. Analysis can be handled by determining the volume fraction of the fine and coarse regions by point counting using randomly selected fields. Then, the grain size within each region can be analysed by selectively measuring each area. This is a reasonably simple procedure, although some subjectivity is involved. A low magnification would normally

be used for the volume fraction determination, whilst higher magnifications are needed for the selective grain size estimates [A2].

3. A continuous random dispersion with a small amount of grains scattered through the matrix that are outside the normal distribution, or the presence of an unusually wide range of grain sizes. This is a much more difficult situation. The best procedure may be to develop a size distribution using either grain areas, diameters, or intercepts. For a manual analysis, a frequency distribution of intercept lengths is easiest to perform [A2]. Any measurement parameter can be used with semiautomatic or automatic image analysers. Single coarse grains can be measured individually, and ASTM Standard E-930-89 sets out several methods for estimation of the largest grain size observed [A11].

Estimation of the area fractions occupied by the distinct grain sizes in a given specimen is the most subjective part of characterising duplex grain sizes, and is the part most prone to error [A10]. Four procedures for estimating area fraction are described in the ASTM Standard E-1181, each of which results in a different precision level. The four procedures recommended in the standard are as follows.

1. Comparison procedure: Use area fraction comparison charts, using the lowest magnification that allows visual resolution of the coarse and fine grained areas.
2. Point counting procedure: Count the number of grid points falling within the particular grain size region being evaluated. Any points falling on the boundary should be counted as half.
3. Planimetric procedure: A planimeter, a device which measures areas of irregular polygons, may be used to measure the area fractions occupied by the distinct grain sizes.
4. Direct measurement procedure: This procedure may be used with specimens which show banding or where there are surface layers of differing grain size.

A10 Two-phase structures

If the second phase is similar in size to the matrix phase, comparison charts can be used to estimate the matrix grain size. If a more accurate estimate is desired, or the two phases are not of similar size, either the planimetric or the intercept method can be used. If the planimetric method is chosen, the number of matrix grains that are within the test area and intersect the perimeter are counted as normal, but the percentage of the test area containing these grains must also be measured. This can be done by point counting or lineal analysis within the test area. The corrected test area is then used in the calculations. This is not a particularly convenient method [A2].

The intercept method is simpler and yields excellent results. The volume fraction of the matrix should first be determined either by point counting or lineal analysis. This measurement should be made on the second phase, and the matrix volume fraction determined by difference. Next a linear test grid is applied to the structure and the number of matrix grains, N_α , intercepted by the test lines is counted. It is better to count the number of intercepted grains than to count the number of grain-boundary intersections. These two measurements are not equal for two-phase structures. The mean linear intercept of the matrix (α) phase is calculated according to

$$\bar{L}_\alpha = \frac{V_{V,\alpha} L_T}{N_\alpha} \quad (\text{A17})$$

where L_T is the total line length calculated at a magnification of $1\times$ (e.g. a line length of 500 mm when viewed at a magnification of $\times 250$ gives an L_T value of $500/250 = 2\text{mm}$).

A11 Volume Fraction Measurement

The volume fraction, V_v , of a constituent is the volume of the constituent divided by the specimen test volume. There is no simple, direct approach for determining volume fractions. Instead, indirect approaches are used. Techniques for volume-fraction analyses are based on one or more of the following principles [A12]:-

1. *Areal analysis.* The areal fraction of a three dimensional feature intercepted by a random plane provides an unbiased estimate of the volume fraction of that feature.
2. *Lineal analysis.* The fractional intercept on a line passing at random through a two- or three- dimensional feature provides an unbiased estimate of, respectively, the areal or volume fraction of that feature.
3. *Point count analysis.* The fractional number of randomly or regularly dispersed points falling within the boundaries of a two-dimensional feature on a plane, or within the boundaries of a three-dimensional feature in a volume, provides an unbiased estimate of, respectively, the areal or volume fraction of that feature. [A8].

As a general rule, the volume fraction of particles is approximately equal to the surface area fraction even if the particles have different sizes, provided that they are equiaxed and uniformly distributed, and that a statistically significant number of fields are examined. If the particle distribution is not uniform then difficulties can arise. Specimen preparation is important, and the true microstructure must be revealed without any surface imperfections. Poor preparation can lead to misleading results, for example, relief effects can lead to systematic under- and over-estimation of sizes and volume fractions [A13].

A11.1 Areal analysis

The average areal fraction A_A obtained on a two-dimensional surface is an estimate of the volume fraction V_v :

$$V_v = \frac{\sum A_\alpha}{A_T} = A_A \quad (\text{A18})$$

where $\sum A_\alpha$ is the sum of the areas of the phase of interest and A_T is the total measurement area. The measurements can be made either by planimetry or by cutting out the areas of interest from a photograph. If σ_α is the standard deviation of the areas of individual α particles, then the measurement error, or proportional variance, E_α is given by [A14].

$$E_\alpha^2 = \frac{1}{n} \left[1 + \left(\frac{\sigma_\alpha}{\bar{A}} \right)^2 \right] \quad (\text{A19})$$

where n is the number of particles measured and $\bar{A}$ is the mean area measured. This equation was established on the assumption that the α -phase occurs as discrete particles randomly distributed in three dimensions, which implies that the volume fraction is small. It is not

considered to be valid when the volume fraction of second phase approaches 30% or more. The error is minimised for large n and is expected to be smaller for uniform microstructures (small σ_α).

If it is assumed that the α phase particles are uniform diameter spheres, it can be shown that [A14]

$$\left(\frac{\sigma_\alpha}{A}\right)^2 \cong 0.2 \quad (\text{A20})$$

Equation (A19) then reduces to

$$E_\alpha \cong \frac{1.1}{\sqrt{n}} \quad (\text{A21})$$

The number of α -phase areas which have to be measured to achieve a given relative error can thus be determined [A14].

Relative error %	Number of areas to be counted for:	
	67% confidence	95% confidence
± 1.0	12000	48000
± 2.0	3000	12000
± 4.0	750	3000
± 5.0	480	1920
± 10.0	120	480

However, the assumption of uniform spherical particles is unrealistic in most cases, and equation (A21) gives the most optimistic estimate of the relative error. Hence, even more particles than given above may need to be measured. The method thus involves very considerable time and effort, and is rarely used today.

A11.2 Lineal analysis

The total length of randomly placed lines within the phase of interest, ΣL_α , is divided by the total line length L_T to obtain the lineal fraction:

$$L_L = \frac{\Sigma L_\alpha}{L_T} = V_V \quad (\text{A22})$$

The total line length, L_T , must be large with respect to the dimensions of the microstructural features since equation (A22) is only valid when the total length of intercept is sufficient to guarantee a representative sample. The proportional variance is given by

$$E_L^2 = \frac{1}{n} \left[1 + \left(\frac{\sigma_L^\alpha}{\bar{L}_\alpha} \right)^2 \right] \quad (\text{A23})$$

where n is the number of intercepts in the α -phase particles, σ_L^α is the standard deviation of intercept lengths in individual particles, and $\bar{L}_\alpha$ is the mean intercept length of the particles. This equation is only valid for small volume fractions of discrete, randomly distributed particles, and a more general relationship for α -phase particles occurring in a β -phase matrix is given by [A14]

$$E_L^2 = \frac{1}{n}(1-V_V)^2 \left[\left(\frac{\sigma_L^\alpha}{\bar{L}_\alpha} \right)^2 + \left(\frac{\sigma_L^\beta}{\bar{L}_\beta} \right)^2 \right] \quad (\text{A24})$$

However, the standard deviation of the intercept is equal to the mean intercept length for either phase, i.e. $\sigma_L^\alpha = \bar{L}_\alpha$ and $\sigma_L^\beta = \bar{L}_\beta$ [15], so that equation 36 simplifies to:

$$E_L = (1-V_V) \sqrt{\frac{2}{n}} \quad (\text{A25})$$

This method tends to give poorer accuracy than areal analysis, but is quicker to carry out, thus allowing more extensive regions to be measured without incurring an excessive time penalty.

A11.3 Point counting

The most efficient method for manual determination is the point count procedure. The accuracy is close to that of areal analysis, but much less time is required [A5,A12] The probability that a test point placed in the specimen volume is contained in the phase of interest is exactly equal to the fraction of the specimen volume occupied by that phase.

A grid with a number of systematically spaced test points is placed over the structure. The points lying over the constituent of interest are counted and divided by the total number of grid points to obtain a point fraction, P_p . Points lying on the interface are counted as 0.5. In order to minimise bias, any point that is doubtful as to whether it is inside or outside the boundary should also be counted as 0.5 [A16]. This process is repeated for a number of randomly chosen fields. The point fraction P_p is given by

$$P_p = \frac{\sum P_\alpha}{nP_o} \quad (\text{A26})$$

where n is the number of fields measured and P_o is the number of grid points [A2] The volume fraction is then expressed as a percentage by multiplying P_p by 100. The measurement error is given by [A14]

$$E_p^2 = \frac{1}{P_\alpha} \quad (\text{A27})$$

The error is independent of the size distribution of the α -phase, and is also less than the errors arising from areal analysis.

The quantity of interest is the value of the test point fraction averaged over the whole specimen, and not its average value in a given field or frame of view. Thus, it is better to use a grid with a small number of test points and place it over a large number of fields of view than to have a grid with a large number of test points placed on a very small number of fields of view. Too high a density of points per field will greatly increase the effort required in counting without giving any appreciable increase in accuracy [A12]. To ensure maximum efficiency, it is considered essential that the magnification relative to grid spacing should be such that the majority of the structural features should not occupy more than one grid corner. The ASTM Standard E-562-89 recommends that the magnification should be chosen such that the average feature or constituent size is approximately one half of the grid spacing [A16].

It has been shown [A12] that a grid with a systematic two-dimensional point array is most efficient, and that the optimum number of grid points is given by $3/V_v$. Thus, for volume fractions from 0.5 to 0.01 (50% to 1%), the optimum grid test point density will vary from 6 to 300 for manual measurements [A5]. The points in constituents with the lowest volume fraction should always be counted in order to minimise effort [A2].

A point counting experiment was carried out [A2] using three microstructures each having a different volume fraction of minor phase (3%, 30%, 45%), and four different grid point densities (9, 25, 64, 100). Three counting conditions were used, namely:

1. a constant number of fields,
2. a constant total number of grid points,
3. a constant measuring time.

In each case, the results were translated into percentage point fractions $100P_p$, where P_p is defined as in equation (A26). The mean, standard deviation, 95% confidence limit and % relative accuracy were calculated. The total measurement time, including set-up and calculations, was determined for each case. As would be expected, for a constant number of grid points, measurement time decreased with increasing grid point density, since fewer fields were required, but for a constant number of fields, time increased with increasing grid point density. Measurement time increased with increasing volume fraction of the phase to be measured.

The ASTM Standard E-562-89 gives guidelines for grid selection as follows:

Visual estimate of area fraction (%)	Grid size (number of points)
2 to 5%	100
5 to 10%	49
10 to 20%	25
> 20%	16
These guidelines represent an optimum for efficiency for the time spent counting and for the statistical information obtained per grid placement.	

The relative accuracy of the measurements improves with increasing grid point density and increasing volume fraction. For a constant total number of grid points, the relative accuracy remains essentially constant. As the volume fraction increases, the relative accuracy shows less difference with grid point density, because the low density grids are more efficient for

high volume fractions. The high density grid is clearly superior for low volume fraction, confirming that the optimum grid density is in inverse proportion to the volume fraction.

In any evaluation of real materials, both material factors and test factors influence the precision and reproducibility of the measurements. For heterogeneous structures, it is better to spend less time measuring each field, and measure more fields. Hence, a lower density grid is more efficient [A5].

The balance between accuracy and time taken to undertake the measurements can be critical. The ASTM standard E-562-89, which defines a standard test method for determining volume fraction by manual point counting, includes a table showing the number of fields which need to be counted to give different levels of relative accuracy.

Volume fraction, $V_V\%$	33% RA		20% RA		10% RA	
	Grid of 25 points	Grid of 100 points	Grid of 25 points	Grid of 100 points	Grid of 25 points	Grid of 100 points
2	75	20	200	50	800	200
5	30	8	80	20	320	80
10	15	4	40	10	160	40
20	10	4	20	5	80	20

These values were calculated using the equation

$$n \cong \frac{4}{E^2} \cdot \frac{100 - V_V}{V_V} \quad (\text{A30})$$

where $E = 0.01 \times \text{RA}\%$ and V_V is expressed in %. The table shows that for small volume fractions, the number of fields that need to be measured to achieve high accuracy becomes extremely large, and therefore very time consuming. As a guideline, to reduce the %RA by 50%, a total of four times the original number of fields should be measured [A16].

For isotropic and homogeneous microstructures, the volume fraction can be precisely estimated from just one metallographic plane. If the structure is anisotropic, the choice of sectioning plane orientation becomes very important. If the structure contains a gradient, the metallographic plane orientation should contain the direction of the gradient.

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Appendix B

14

IN THIS CHAPTER

- Electron Backscattered Diffraction (EBSD)
- Method and Limitations
- Measurement Uncertainties
- Underpinning Case Studies

APPENDIX B Electron Backscattered Diffraction

B1 Introduction

Electron Backscattered Diffraction (EBSD) has rapidly grown in use in recent years as a tool to characterise microstructure. Improvements to both software and hardware have enabled detailed crystallographic information to be acquired automatically from large areas of a polished specimen within a relatively short time. Selection of the appropriate operating parameters before acquisition and choice of methods of manipulating and interpreting the large quantity of data at the end of the map all affect the final results significantly, even on relatively simple single or two phase materials. The distribution of orientations or of phases in the specimen can be obtained, providing a basis for detailed quantitative evaluation of the microstructure [B1-B8 and EBSD bibliography].

A new ISO standard on the EBSD method is being developed by ISO/TC 202 (Microbeam Analysis) and further information can be obtained from national representatives appropriate to the ISO/TC 202 committee (ISO/TC 202 N137 ISO/WD). Also ASTM committee E04 (Metallography) is developing a standard for the measurement of grain size by EBSD (ASTM WK3577).

EBSD is an electron microscopy technique for identification (with high spatial resolution) of the crystallographic orientation of an array of points on the surface of a sample. It is based on the formation of diffraction or Kikuchi patterns produced when an electron beam strikes the sample surface at low incident angles. Electrons scattered from the beam that fulfil the Bragg Diffraction conditions for the crystal lattice planes in the sample produce flat cones of high intensity in the same manner as X-ray diffraction. The cones intersect a suitably positioned phosphor screen and are seen as pairs of lines with very slight curvature. The combination of line pairs produced by the different lattices planes forms the Kikuchi pattern. By indexing the lines and measuring their relative orientation the crystallographic orientation of the point on the sample under the beam that is being interrogated can be calculated. The hardware generally comprises a camera and an image processing system for pattern averaging and background subtraction. The software controls the data acquisition, solves the diffraction patterns and stores the data. Further software is required to analyse, manipulate and display the data.

Because the diffraction cones/lines result from electrons scattered with very little energy loss they are formed only from lattice planes close (within ≈ 20 nm) to the surface. The diffraction pattern formed is representative of an area of similar order to that of the electron beam, typically <200 nm and in high resolution microscopes <50 nm. Also as a consequence of the region of interaction the technique can examine the surface of bulk samples but the surface must be flat and free from damage induced in the crystal structure by the process used to produce the flat surface. Any damage to the crystal structure reduces the intensity of the diffracted electrons and may prevent indexing of the pattern.

By stepping the electron beam in both x and y directions across the polished surface of a sample tilted at (typically) 70° to the beam, and analysing the diffraction pattern produced at each step a map of the crystallographic orientation of the sample can be built up. Software can step and index the patterns automatically; subsequent analysis of the crystallographic data set allows identification of changes in orientation between pixels, and hence grains and grain

boundaries can be defined by grouping of pixels of similar orientation within a boundary set between pixels with a misorientation greater than a defined value. The data stored with each pixel enables automatic identification of, for example, twin boundaries, and automatic calculation of grain size, either by area or by linear intercept.

There are a number of factors which have to be taken into account when deciding if EBSD can be successfully used for a particular investigation [B3, B4].

- **The specimen.** The EBSD signal and the quality of the diffraction pattern increase with atomic number. In order to obtain an analysable pattern, the region of the specimen from which the pattern is obtained must have a single crystallographic orientation. Thus the smallest grain or subgrain size which can be measured is related to the spatial resolution of the technique.
- **Speed.** The time to acquire a data point depends on the slowest of:
 - The time required to obtain an analysable diffraction pattern. Typical times are 0.05 to 0.3 seconds.
 - Time required to analyse the pattern. This depends on the processing speed of the computer and the speed of the pattern-solving algorithm, which is typically 0.1 to 1 second. Patterns that cannot be analysed can significantly increase the time required to produce a map.
 - Time to reposition the beam or stage. Scanning can be done by moving either the electron beam or the specimen stage. The repositioning time is negligible for beam scanning, but may be greater than 1 second for stage scanning.
- **Spatial resolution.** EBSD is carried out on specimens tilted at about 70° from the horizontal. The area from which the pattern is obtained is elliptical, with the major axis, which is perpendicular to the tilt axis, being about three times that of the minor axis. The resolution parallel to the tilt axis is typically in the region of 200-500 nm for aluminium, but it varies with the material, the beam accelerating voltage, the specimen tilt angle and the probe size. Better spatial resolutions can be obtained with very small probes, but data acquisition times become very long.
- **Angular resolution.** The accuracy of determination of the absolute orientation is typically about 1°. The number of pixels into which the acquired pattern is digitised determines both the angular resolution and the time for pattern analysis. The limited angular resolution results in 'orientation noise' in the data which has the consequence that there is a lower limit to misorientations between grains which can be recognised above the noise threshold.
- **Non-indexed data points.** Each point on the specimen from which a diffraction pattern is collected does not necessarily produce data because the pattern quality may be too poor to analyse. Alternatively, the software may not be able to distinguish between overlapping patterns at grain or phase boundaries. If the number of such non-indexed points is large then it may be difficult to obtain quantitative microstructural data.
- **Software limitations.** The vast majority of EBSD installations rely on commercially available software and the user may have little control over the speed and accuracy of pattern analysis. Similarly, there may be little flexibility in the methods by which the data scans are obtained or how the results are analysed.

In addition it is important to note that there are two different colour schemes for representing differences in orientation (Euler and IPF). These are shown in Fig B1. Euler especially can

result in misleading artefacts where a small (1-2°) difference can result in very abrupt colour changes (at for example the 360° boundary).

With respect to other issues concerning this current Good Practice Guide then choice of step size in producing EBSD maps of hot deformed alloys is critical, especially in cases of partial recrystallisation where a bimodal distribution exists. Inappropriate use of noise reduction methods can lead to significant changes in reported grain size. Methods of reporting grain size distributions over large areas with gradients in grain size are important too, and the ability of EBSD to select particular subgroups by grain size, orientation or aspect ratio can be very useful in enabling visualisation of the effects of deformation. The higher resolution of EBSD in comparison with optical methods can lead to smaller grain sizes being reported by EBSD, although broad trends may be followed by both techniques.

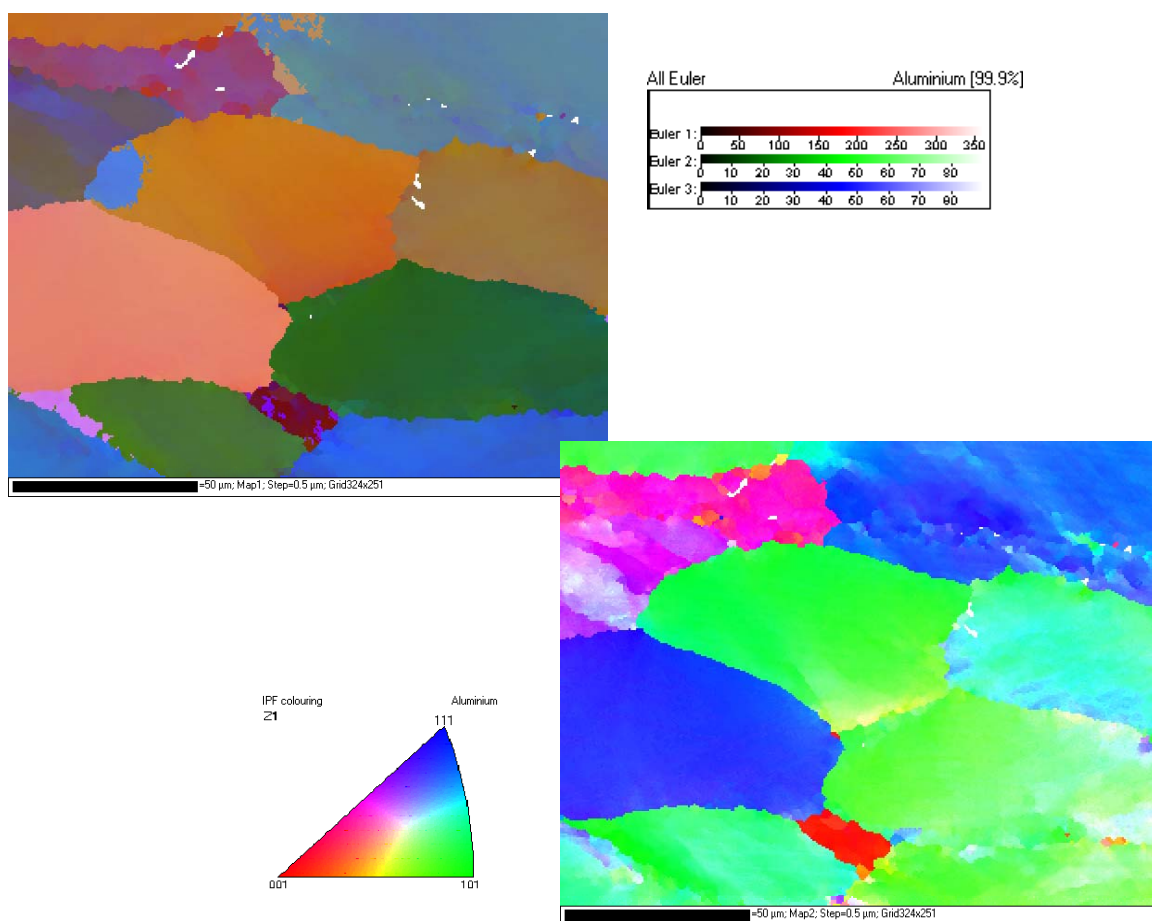


Figure B1 Example comparison of different EBSD colour schemes.

B1.1 Sample Preparation

Because diffracted electrons escape from within only a few tens of nanometres of the specimen surface, any surface defects, such as surface deformation, and surface contaminants and oxide or reaction product layers, will influence the electron diffraction signal resulting in patterns of either low quality or spurious origin. To avoid this, careful preparation is needed

for most bulk specimens to obtain useable diffraction patterns. Some specimens however, such as crystal films, require no preparation prior to analysis. Each material under investigation should be considered on an individual basis and the preparation scheme be chosen appropriately. (For more details see ASTM E 3-2001: Standard Practice for Preparation of Metallographic Specimens and ASTM E 1558-1993: Standard Guide for Electrolytic Polishing of Metallographic Specimens). Two thorough summaries of procedures for metals and ceramics have been published by Katrakova and Mücklich [B7, B8].

The sample surface must be crystalline and without excessive plastic deformation. In the case of metals, this can be obtained by electropolishing the surface of the metallic specimen after polishing if conventional polishing proves to be inadequate. Ion sputtering can be used to remove the work hardening layer formed as a result of the grinding and polishing on the surface of a metal or non-metal. The sample not only must be crystalline in the subsurface area, but the crystal features (such as grain or sub grain size, twins, and deformation state, if this is to be quantified) should be the same as in the bulk, to avoid artefacts due to preparation effects.

B1.1.1 Cutting

When cutting specimens for EBSD, the sample axes orientation should be preserved. For rolled sheets, the significant sample directions (rolling direction, transverse direction and sample normal direction) should all be recorded. The cutting process must not damage or change the sample. Avoid aggressive cutting methods that generate heat or cause deformation at the cut surface. Severe damage induced at this stage may extend so deep into the material that it is not removed by subsequent grinding and polishing. Heating caused during cutting may cause changes to the microstructure - phase transformations or precipitation/diffusion may take place. Therefore, heating must be avoided.

B1.1.2 Mounting

Small specimens generally require mounting so that the specimen is supported in a stable medium for grinding and polishing. The medium chosen can be either a cold curing resin or a hot mounting compound. Mounting is needed for polishing but then could be eliminated to prevent charging effects. A conductive mounting material is preferred for EBSD analysis as it helps to minimize electron beam charging effects which can cause intensity blooming in the EBSD patterns and image drift and distortion in orientation maps. Often even non-conductive specimens can be mounted in such materials and observed using EBSD without requiring a conductive coating. If the specimens cannot withstand the heat/pressure required for resin mounting then the use of other mounting materials may be necessary. In this case, a cold-mounting epoxy may be more appropriate. During analysis, the non-conductive surface of the epoxy may be painted with conductive silver or carbon paint to minimize charging effects.

B1.1.3 Grinding

Grinding can be achieved in a variety of ways, using a variety of abrasives. Fixed abrasive surfaces are available using SiC, Al₂O₃, diamond or cubic nitride abrasives. Grinding on a surface that has blunt abrasives causes a great deal of surface damage by smearing, 'burnishing' and local heating. Damage imparted during grinding may be invisible in the polished surface, but serve to distort the EBSD result or even completely suppress pattern formation. Therefore, in order to maintain sharp abrasive particles, grinding papers should be

changed frequently during the specimen preparation process. Also, the selection of grinding material and conditions should be specific for a given specimen.

After every grinding stage it is advisable to inspect the ground surface using a light microscope in order to ensure that all damage from the previous stage, whether that be a cutting or grinding stage, is completely removed. Proceed in this manner to the finest abrasive size required ready for polishing.

B1.1.4 Polishing

It is usually the case that polishing is started on a hard cloth with a coarser abrasive and finished on a softer cloth with a finer abrasive. Final polishing should not be prolonged, but just sufficient to achieve the desired surface finish without causing excessive relief. Extreme undulation or relief in the polished surface is to be avoided.

Diamond polishing compounds or slurries are good for preliminary stages for most materials. Typically, diamond polishing is carried out down to a 1 or 0,25 μm particle size and this is followed by polishing with an 0,05 μm colloidal silica slurry. A polishing time for this final step of between a few minutes and about 20 minutes is common for metal. Harder polishing surfaces or cloth produce a flatter or 'plane' surface but may leave polishing damage at the surface of the material, and promote superficial scratching. For metal specimens having hard second phases such as carbide, the above procedure can also produce better results. Colloidal silica is a chemo-mechanical polish, i.e. it combines the effect of mechanical polishing with etching. This type of stock removal is ideal in many cases for EBSD, as a damage free surface can be obtained with little effort. Meticulous attention to avoiding contamination of wheels is an important aspect in achieving the best results. Use of colloidal silica generally produces good results with nearly all materials.

B1.1.5 Etching

Polished surfaces can be inspected using EBSD, but in many cases the pattern quality is improved by etching. Additionally, etching delineates the grain structure, which is of obvious benefit. However, etching may attack a second phase preferentially, or attack grain boundaries excessively. Materials that are difficult to polish may benefit from repeated etching and repolishing. This method can expose an undamaged surface suitable for EBSD when conventional polishing and etching fail to achieve an adequate surface. Any etchant that is used must dissolve the specimen surface in an even manner, and not leave behind any oxide or reaction product layers. Such layers can completely suppress diffraction. Many etchants listed in metallographic text books are 'contrast etches' which rely on the formation of different thickness oxide layers to generate colours visible using a light microscope. Therefore such etches are generally not suitable for EBSD.

Electrolytic polishing and etching are alternative and effective methods which can dissolve the surface metal of deformed layer by controlling voltage and time applied to specimen in an electrolytic cell.

Plasma etching is a process in reverse compared with sputter coating. Generally it should be used as a means of cleaning or improving a mechanically or electrolytically prepared surface.

B1.1.6 Ion beam techniques

Ion milling is a process applied to a specimen under vacuum whereby a selected area of the surface is bombarded by an energetic beam of ions. Generally the use of rotation and grazing bombardment angles promotes even erosion of the specimen surface and minimizes damage effects. However, certain grain orientations, grain boundaries and phases can erode at different rates. Ion milling can give reasonably high rates of material removal and this can be enhanced by the use of reactive gases, such as iodine in the reaction chamber. Ion milling can produce surfaces suitable for EBSD with minimal prior preparation, especially on materials that are difficult to prepare by conventional metallography, such as zirconium and zircalloys.

B1.2 Grain Size Measurements

Both grain sizes and shapes may, in principle, be obtained by automated EBSD and there are two main size parameters, one determined by the use of line intercepts and one obtained from the diameter of a circle of equivalent area for each specified grain.

B1.2.1 Line Intercept Method (EBSD)

In the intercept method a line of data points in the x-direction is analysed by comparing the orientations of adjacent points and noting the number of high-angle boundaries detected along the line. A prior decision must be made as to what misorientation constitutes a high-angle boundary, and 15° is often used. The ability to precisely define the nature of the boundaries constitutes a significant advantage over optical methods, where the visibility of the boundary is a function of technique, and where all visible boundaries must be counted [B3, B4].

Non-indexing of patterns sometimes occurs at grain boundaries for various reasons, including deformation, poor focusing, scratches, dirt or the presence of other phases. In these cases the orientations of the indexed points on either side of the non-indexed point are compared. This will only lead to inaccuracy if the amount of non-indexing is such as to cause a complete grain to be missed, such as when the grain size is very small.

A minimum of about 200 grains is typically required for a determination of grain size, and about 10 points across a grain are required to define the grain with sufficient accuracy [B6]. Thus, about 2,000 data points are required to measure the grain size in one direction from a line intercept. At a speed of 0.3 seconds per point, this should take about 10 minutes. To fully define the grain size and shape in both directions, a matrix of 100 points is needed for each grain. This suggests that a map of 20,000 points is required, which would take under 2 h. This is a significant amount of machine time, but since the data acquisition and processing procedures are fully automated, little operator time is required [B3, B4].

Backscattered imaging reveals both high and low angle boundaries and gives a smaller grain size compared with optical microscopy where only the high angle boundaries are revealed. The operator can select the degree of misorientation before beginning the scan depending on whether all boundaries or high-angle boundaries only are to be counted. If required, data for other boundaries such as twin boundaries can be acquired using this method [B3, B4].

B1.2.2 Grain Reconstruction

An alternative method of analysing the grain structures is by grain reconstruction. For each data point, the misorientations of neighbouring points are checked, and if they are within a specified limit, they are assigned to the same grain as the reference point. This procedure is continued until a complete grain has been defined. This type of analysis is capable of producing more complete information on grain areas and shapes than the intercept method, but because it relies on obtaining a high resolution orientation map, it is considerably slower [B3]. A grain size parameter, approximately equivalent to the line intercept value, is obtained by calculating the diameter of a circle of equivalent area.

B2 Measurement uncertainty using EBSD

The acceptable level of uncertainty in an EBSD measurement and the requirements for achieving this depend on the type of EBSD analysis being conducted, the material being analysed and the purpose for which the result is to be used. Two key aspects are:

Absolute orientation: uncertainty in the measurement of the orientation of a crystal with respect to the sample coordinate system.

Relative orientation (or misorientation): uncertainty in the measurement of the orientation of one crystal with respect to another (i.e. the misorientation between two crystals). For small misorientation angles ($\sim 5^\circ$ or less), the uncertainty in the misorientation axis becomes very large.

B2.1 Measurement of absolute orientation

The uncertainty in the measurement of the orientation of a crystal with respect to the sample coordinate system is a result of the following sources of error.

- Uncertainty in the specification of the sample coordinate system
- Uncertainty in the specification of the alignment between the sample coordinate system and the coordinate system of the EBSD detector
- Uncertainty in acquiring the EBSP
- Uncertainty in indexing the EBSP.

Each of these sources of uncertainty is discussed in more detail below.

Uncertainty in the specification of the sample coordinate system

This includes uncertainty in cutting and mounting the specimen, if the specimen surface and edges are used to define the specimen axes, or uncertainty in marking the directions of the axes on the specimen (e.g. using micro-indenters or scribing lines), if these methods are used. Due to the wide range of possible cutting and mounting techniques, this component of the orientational uncertainty may vary widely (between about 1 and 10 degrees) and specification of the uncertainties associated with each possible cutting and mounting technique is beyond the scope of this standard.

Where the dominant components of the bulk texture of the sample are well known and the analysis has been carried out over a region that is sufficiently large and appropriately located to be representative of the bulk specimen, compensation for the uncertainty generated by cutting and mounting can be conducted after the acquisition of the data, during data processing. This might be the case when studying orientational deviations within a single crystal, where the principal orientation of the single crystal is well known. Another example might involve investigations of small changes in the textures of cold-rolled or extruded metals with well-known characteristic textures. In these cases, the deviation of the well-known dominant components from their usual positions could be assumed to be the error due to specimen cutting and mounting and all the orientations in the dataset could be rotated to bring the dominant texture components into their well-known accepted positions. Use of this method can potentially mask real changes in texture produced by processing variation. Consequently, it should only be used on strongly-textured specimens and only after careful consideration of whether the deviation of the dominant texture component from its usual position is almost certainly due to cutting or mounting error, or whether this deviation could be the result of a real deviation in the texture of the specimen.

Uncertainty in the specification of the alignment between the sample coordinate system and the coordinate system of the EBSD detector

This includes uncertainty in the alignment of the sample within the microscope and uncertainty in the calibration of the EBSD system. For measurements involving significant movement of the beam's point of impingement on the sample surface away from that used for calibration (e.g. in orientation mapping at low magnifications), there is an additional uncertainty associated with the adjustment of the EBSD system calibration to account for the resulting change in geometry between the point of beam impingement and the detector screen (dynamic calibration).

This uncertainty can be addressed by mounting a single crystal of known orientation (EBSD system calibration sample) adjacent to the sample to be analysed and in such a way that the surfaces of the calibration sample and the specimen to be analysed are at the same height. The mounting should be done in a way that ensures that the acquisition surface of the two specimens are coplanar and that the reference directions of the two specimens within this plane (for example, the $\langle 110 \rangle$ direction of the single crystal and the X direction of the sample coordinate system) are parallel, with the best achievable level of accuracy. The latter alignment can be assisted by ensuring that the edges of the specimen to be analysed have been cut parallel to the sample coordinate system as accurately as possible and by pressing the edge corresponding to the reference direction of the single crystal up against an edge of the specimen that corresponds to a specimen reference direction.

The reference directions of the calibration sample and the sample to be analysed should then be aligned with the microscope tilt axis. Once this has been done, the stage and scan rotations should not be changed until the EBSD analysis has been completed. Also, the accelerating voltage of the electron beam, the tilt angle of the specimen, the insertion distance of the EBSD detector and the working distance should not be changed until the EBSD analysis has been completed. Calibrate the EBSD software using the calibration sample. Then move the stage within the plane of tilt to bring the area of interest on the surface of the sample to be analysed under the electron beam at the same working distance just used for the calibration sample. Perform the EBSD analysis.

The above method, using a calibration performed on a single crystal calibration sample at exactly the same conditions as those used for the EBSD analysis, minimises the uncertainty in alignment between the sample coordinate system and the coordinate system of the EBSD detector. For most analyses, where minimal uncertainty in absolute orientation is not critical, or for analyses where only relative orientation measurement is desired, then it is common to load a calibration conducted on a single crystal calibration sample at conditions that are close to those being used for analysis and refine this calibration on an EBSD collected from a known phase in the specimen to be analysed.

Uncertainty in acquiring the EBSD

This includes uncertainties related to the sharpness of the EBSD produced by the diffraction process and the resolution and contrast of the captured EBSD. Some of these uncertainties are related to the condition of the specimen (such as the degree of lattice perfection within the volume of material from which the EBSD is produced). Others are related to the characteristics of the electron beam (for example, the accelerating voltage and probe current, which determine the volume of material from which the EBSD is collected) and the SEM design (with some SEM designs there can be distortion of the EBSD by magnetic fields within the SEM chamber). In addition to these effects, the accuracy of the acquired EBSD is influenced by the EBSD hardware and software and the user-adjustable parameters (such as detector gain, EBSD frame averaging and EBSD pixel binning) that control the EBSD acquisition process.

Uncertainties related to the condition of the specimen can be minimised by good specimen preparation. However, when analysing deformed materials, lattice defects within the volume of material from which the EBSD is produced will reduce the sharpness of the EBSD and contribute to measurement uncertainty, even when the specimens have been prepared in an ideal manner. An increase in accelerating voltage can sometimes be used to provide clearer patterns on specimens that have a very thin (of the order of 10 nm) surface deformation layer, oxide layer or coating. When analysing very fine-grained specimens or deformed specimens, the acquisition of high quality EBDs (and consequently lower measurement uncertainty) requires a well-focused, small diameter beam, still with sufficient probe current to provide EBSD acquisition in a reasonable time. Distortion of the EBSD by magnetic fields within the SEM chamber can be eliminated by using “field free” or “out-of-lens” modes in SEMs that have these modes as well as in-lens or semi-in-lens modes, or by using a SEM with an out-of-lens design. Alternatively, it can be minimised by the use of EBSD software that includes an accurate EBSD distortion correction algorithm. Measurement uncertainty due to EBSD acquisition can be minimised by collecting high resolution EBDs (i.e. using no pixel binning, or low binning levels) and by using low camera gain and averaging over a high number of frames when collecting the EBSD. However, these settings also cause the EBDs to take longer to acquire, so a compromise between speed and accuracy is always required.

Uncertainty in indexing the EBSD

This includes uncertainties in the algorithms used for detection of the bands within the EBSD, the algorithms used for calculation of theoretical band positions from crystal structure data and the algorithms used for comparing the detected and calculated band positions and selecting the phase and orientation that best matches the acquired EBSD. The algorithms used for EBSD band detection and indexing vary between manufacturers and are central to the accuracy and reliability of the results produced by the EBSD system. Consequently, very little

information about these algorithms is made available to the public. This component of uncertainty will depend not only on the EBSD system used, but also on user-selectable parameters for band detection and indexing.

Uncertainty in the band detection process can be minimised by an appropriate choice of Hough resolution. The accuracy of band detection on high resolution EBSPs can be improved by increasing the Hough resolution from lower to higher levels. However, the time required for the band detection process will also increase with increasing Hough resolution. The use of high Hough resolutions on highly-binned EBSPs is unproductive and can actually reduce the accuracy of the band detection process. Uncertainty in indexing can be reduced by the use of advanced fitting algorithms that refine the matching and ranking of candidate solutions using direct comparison of the simulated reflectors with the actual EBSP, rather than just using the Hough transform. While this method provides greater accuracy, it takes longer, so there is a need for the user to make a choice between the relative importance of speed and accuracy for a given analysis.

B2.2 Measurement of relative orientation (or misorientation)

Uncertainty in the measurement of the orientation of one crystal with respect to another (i.e. the misorientation between two crystals) is independent of the uncertainty in the specification of the sample coordinate system. It is also independent of the uncertainty in the specification of the alignment between the sample coordinate system and the coordinate system of the EBSD detector, except for the uncertainty associated with the adjustment of the EBSD system calibration to account for the resulting change in geometry between the point of beam impingement and the detector screen (dynamic calibration). Uncertainty in the measurement of relative orientation is dependent on the uncertainties related to EBSP acquisition and indexing in the same way as described for absolute orientation measurements in section B3.1, except that the measurement of relative orientation will not be affected by any components of the uncertainties that are systematic (i.e. do not vary from point to point, or orientation to orientation), but only by those components that vary from point to point or orientation to orientation. Consequently, the uncertainty in the measurement of relative orientation between two crystals in a sample is typically much lower than the uncertainty in the measurement of the absolute orientation of each crystal with respect to the sample coordinate system.

B2.3 Additional Uncertainties

Two further additional sources of uncertainty arise from symmetry group considerations and users should be aware of their potential:

Pseudo-symmetry discrimination: uncertainty in the discrimination between two crystal orientations where the discrimination relies on the ability to correctly detect small changes in EBSP band position and width due to small differences between two lattice parameters.

Same Laue group Phase ID: uncertainty in the ability to discriminate between two phases belonging to the same Laue group, where the discrimination relies on the ability to correctly detect small changes in EBSP band widths due to small differences in the lattice parameters of the phases.

B3 EBSD Case Studies

Three representative engineering alloys were examined in underpinning EBSD case studies to support the development of this Good Practice Guide; an aluminium alloy AA5052, a Ni superalloy, Waspaloy, and a stainless steel, 316L. A titanium alloy, Ti6Al4V, was also studied, but the results are omitted in this publication due to the difficulties in resolving differences in microstructure by EBSD techniques. Samples of these alloys were forged under axisymmetric (AC) and plane strain conditions (PSC) and examples are shown of samples subjected to representative strain rates and temperature ranges. Some of the results of these studies have been published in more detail separately [B9-B12]. Table B1 gives details of the testing conditions used to generate a two by two matrix of deformed test specimens (high and low strain rate; lubricated and dry).

Table B1 Testing conditions

Test Number	Material	Initial Temp (°C)	Friction	Strain Rate (s ⁻¹)
W1	Waspaloy	1040	lubricated	0.01
W2	Waspaloy	1040	dry	0.01
W3	Waspaloy	1040	lubricated	1
W4	Waspaloy	1040	dry	1
A1	AA 5052	400	lubricated	0.01
A2	AA 5052	400	dry	0.01
A3	AA 5052	400	lubricated	1
A4	AA 5052	400	dry	1
S1	316L	950	lubricated	0.01
S2	316L	950	dry	0.01
S3	316L	950	lubricated	1
S4	316L	950	dry	1

Cross sections of the deformed testpieces were prepared by spark erosion, followed by mounting in resin and polishing by conventional metallographic techniques. Special attention, as for any EBSD examination, was paid to the final polishing stage to ensure a surface free from deformation caused the polishing. For the Ni and stainless steel alloys, this involved a long (60 minute) final polish with colloidal silica, and for the Al alloy, an electropolish with 30% Nitric Acid in methanol.

Samples were examined in either a Zeiss Supra or FEI Sirion FEG SEM operating at 15 kV, and with a spatial image resolution at least ten times better than the minimum step size of 75 nm used for the work conducted for this GPG. Both systems used Oxford Instruments HKL Nordlys EBSD detectors controlled with Channel 5 software.

B3.1 Analysis of Large Areas

Imaging large areas (up to several square mms) is important because microstructural heterogeneity, in PSC testpieces especially, can extend from top to bottom of the testpiece.

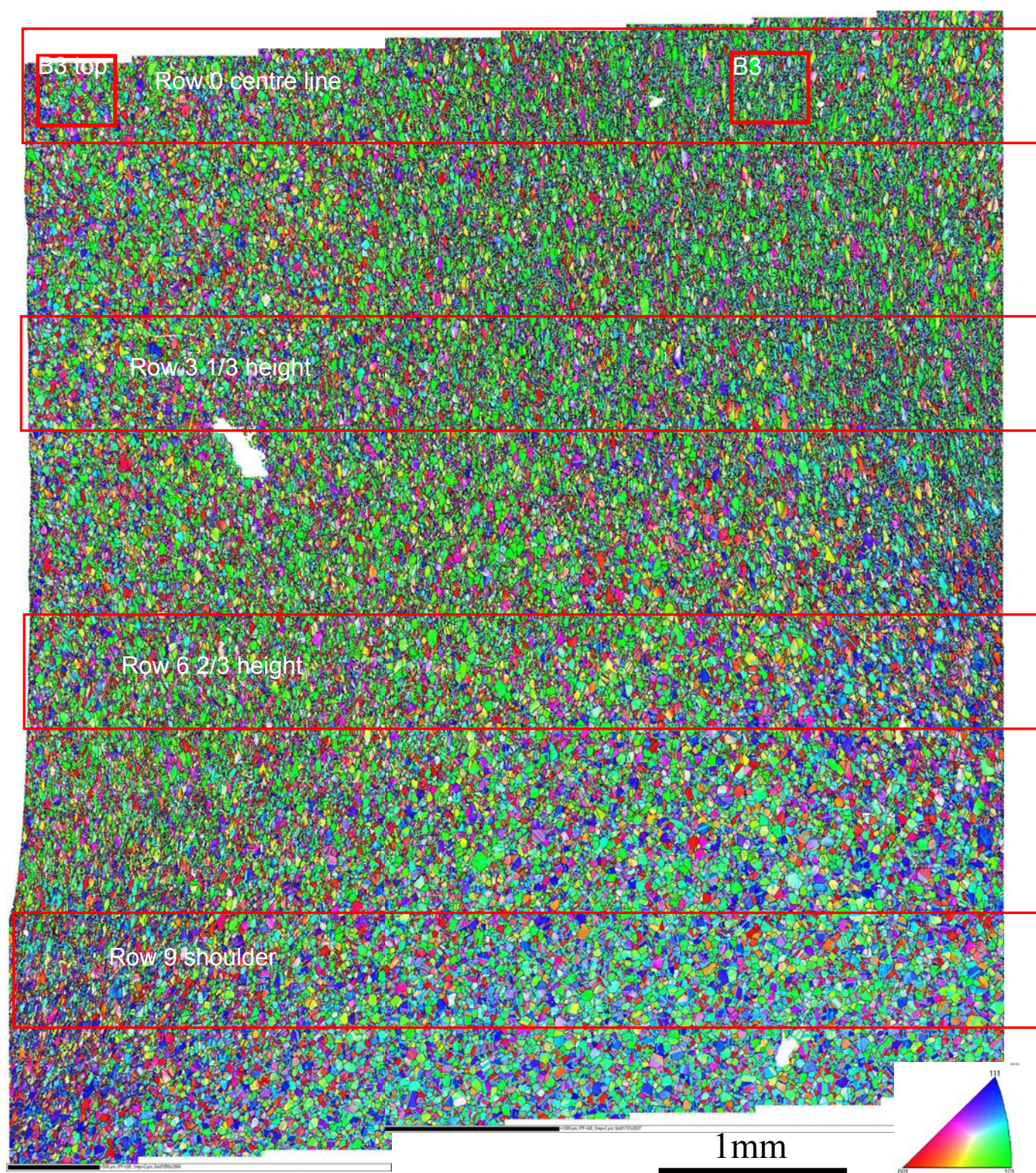


Figure B2a Large area map of Waspaloy PSC sample (5.6 x 5 mm area of PSC sample mapped at 2 μm step size). Inverse pole figure colour scheme used in direction of compression. The position of the map with respect to the whole PSC sample and direction of compression is shown in Fig B2b. Regions shown in Fig B3 are indicated by small boxes; elongated rectangles show approximate position of rows referred to in Figs B4-B9. The white region on the lower edge of row 3 is an area of damage on the sample surface which could not be mapped.

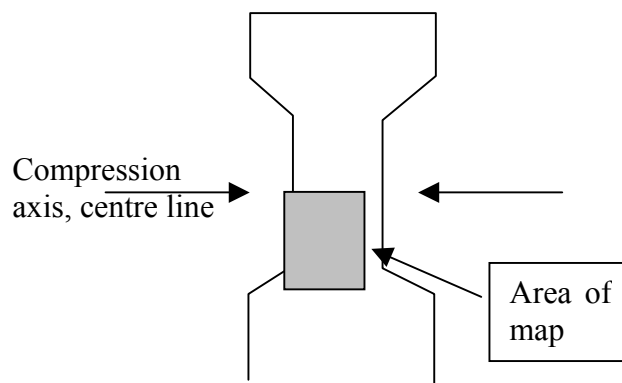


Figure B2b. Schematic of PSC specimen showing position of map in Figure B2 and direction of compression.

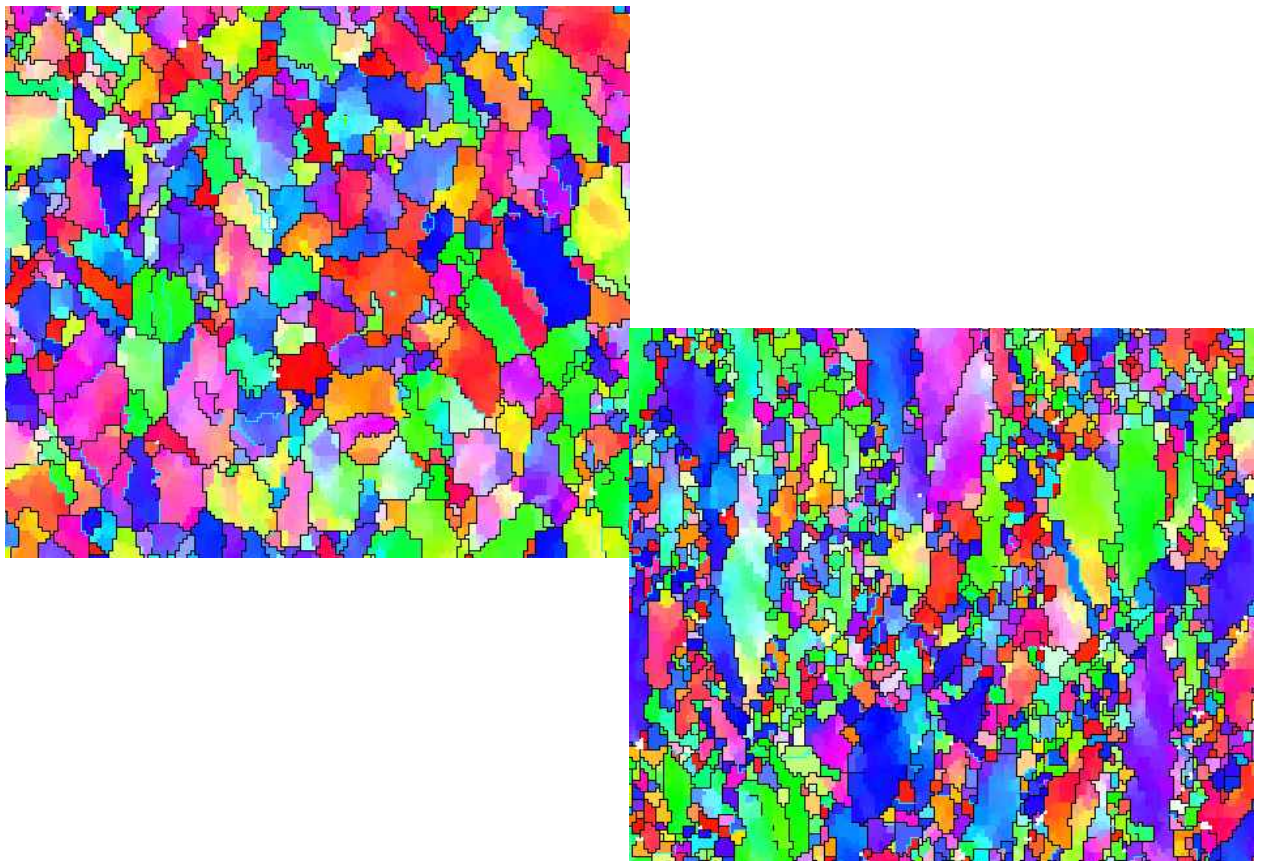


Figure B3 (Top) Enlarged area from Fig B2 in the zone close to the sample surface showing relatively undeformed microstructure with mostly equiaxed unrecrystallised large grains. Image width 320 μm

Figure B3 (Bottom) Enlarged area from Fig B2 near the centre of the sample showing partial recrystallisation with flattened unrecrystallised grains and a necklace structure of small recrystallised grains. Image width 320 μm

Large areas may best be visualised by selecting a particular fraction of the microstructure, especially if a complex microstructure is formed that varies in scale across this area. Individual areas can be mapped by EBSD at different scales (step sizes), but care must then be taken in comparing data from such areas because smaller step sizes generally lead to detection of more smaller grains and average properties then calculated can be biased by many small grains which account for only a small proportion of the area examined. Elimination of very small grains which may be considered noise arising from poor indexing is best done by preserving only those with 3 or more near neighbours.

Figure B2 shows an area of a Waspaloy PSC sample (1040 °C, dry non-lubricated conditions at strain rate of 1 s^{-1}) approximately $5.6 \times 5.0 \text{ mm}$ in area, mapped with a $2 \text{ }\mu\text{m}$ step size, giving approximately 10.5 million pixels. Individual maps $500 \times 700 \text{ }\mu\text{m}$ in size were produced with stage movement between each map area. With an acquisition and indexing at a rate of approximately 40 patterns/s, the map therefore took about 72 hours of continuous mapping to produce. A map of this size represents an enormous amount of data for over 150,000 grains which can then present a challenge for representing this information in a simple and useful manner.

The colour scale used in Fig B2 shows grain orientation according to the inverse pole figure colour legend for axes aligned with the compressive direction. Thus some indication of texture and grain size and shape can be seen but it is difficult to obtain a clear picture in this form. Fig B3 shows enlarged views of the map from the different regions marked, which gives an indication of the range of microstructures produced. The data stored with each pixel enables many possible ways, much of it automated, for both numerical quantification and mapping of the microstructure for correlation with models.

The effects of using a relatively coarse step size to enable mapping of the large area in Fig B2 can be seen in Fig B3 and have been considered briefly in Section 8. A further example is shown in Fig B4, which compares maps of the 316 stainless steel sample (SS2) made with step sizes of $0.2 \text{ }\mu\text{m}$ and $2 \text{ }\mu\text{m}$. Measurements from these maps are summarised in Table B2. Further results based on the high resolution map are presented in Figs B5 and B6. Figure B7 shows an example of the small grains clustered adjacent to deformed large grains. The misorientation profile confirms that these small grains do exist in the microstructure and are neither caused by artefacts nor by noise reduction. A SEM micrograph of SS2 (Fig B8) after electroetching using 1.1V DC in 60% HNO_3 acid provides supplementary evidence for small grains which cannot be revealed by the $2 \text{ }\mu\text{m}$ step size OIMs.

Table B2 Comparison of step size (maps shown in Fig B4)

Step size μm	Number of Grains	Mean d_{ceq} , μm	Median, μm	95% CL, μm
2	83	9.9	7.1	1.6
0.2	590	2.3	0.8	0.5

*grains smaller than 3 pixels in both maps are excluded

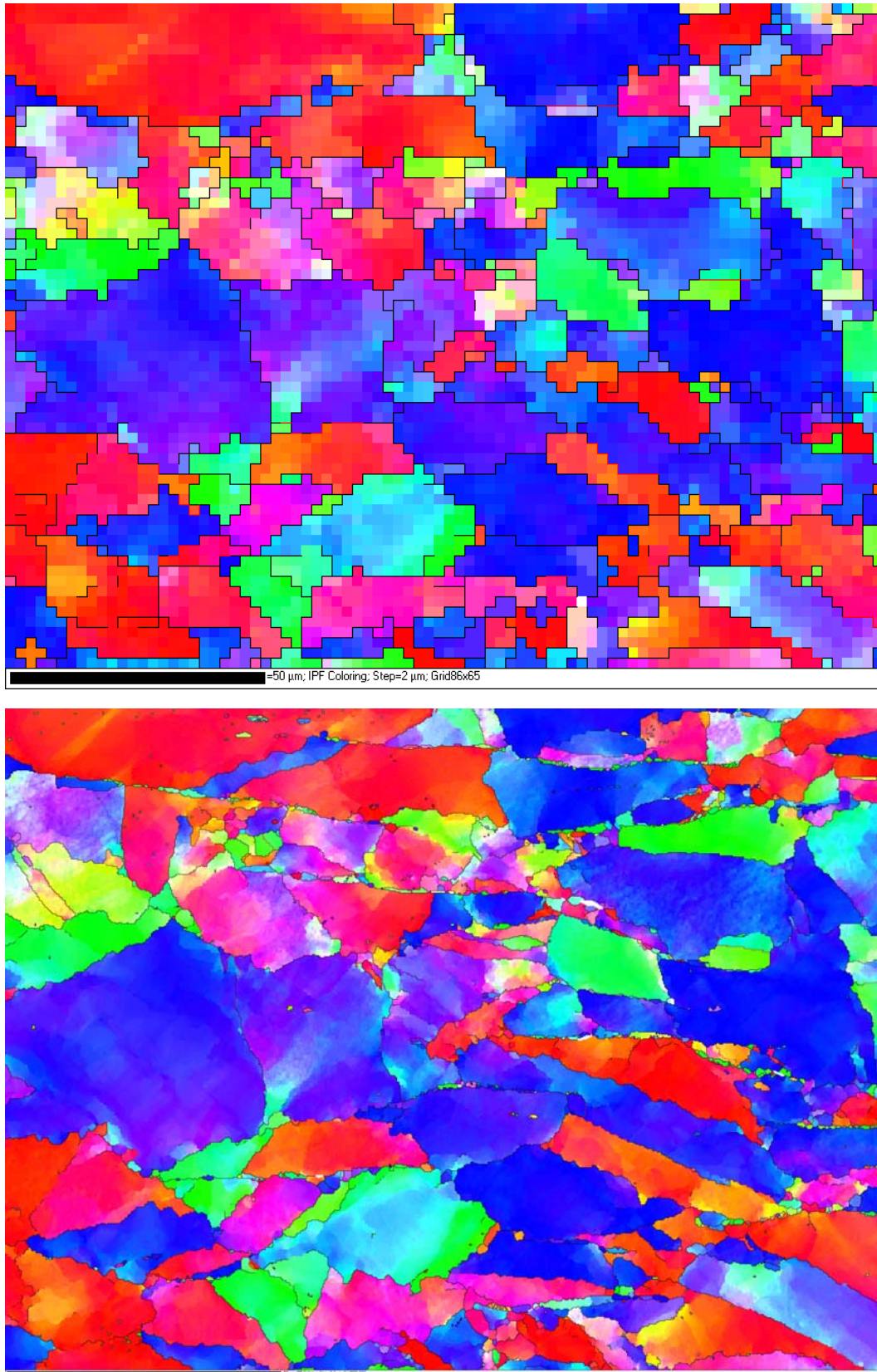


Figure B4 Comparison of same area of 316 stainless steel (SS2) using different step sizes, 2 µm and 0.2 µm

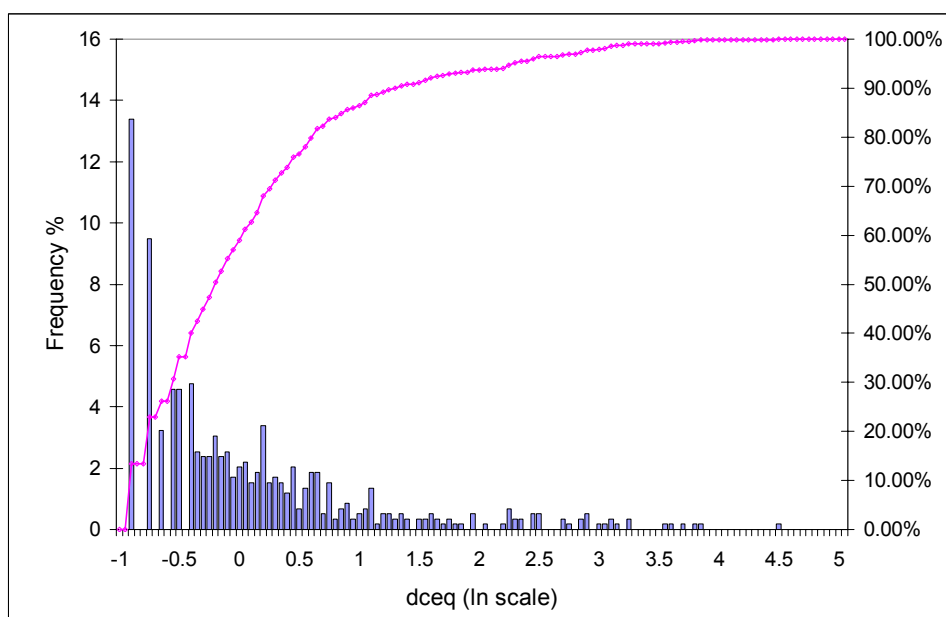


Figure B5 Histogram and cumulative frequency plot of grains larger than $0.12 \mu\text{m}^2$ in the map with step size of $0.2 \mu\text{m}$

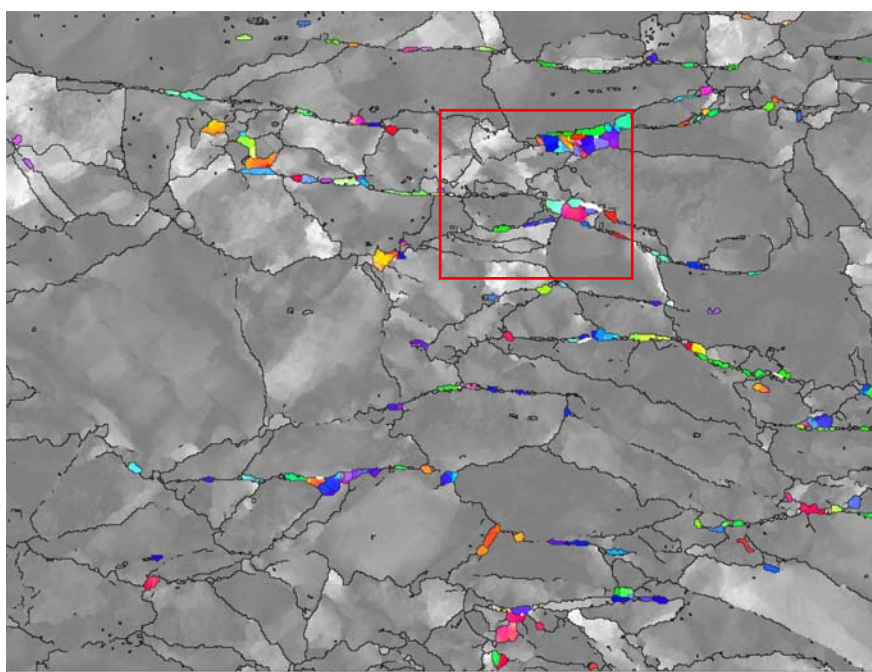


Figure B6 Small grains of area range between $1 \mu\text{m}^2$ (25 pixels,) and $12 \mu\text{m}^2$ (which is the cut-off value for $2 \mu\text{m}$ step size maps). These grains are missed out during the coarse step size mapping. Microbar is $50 \mu\text{m}$.

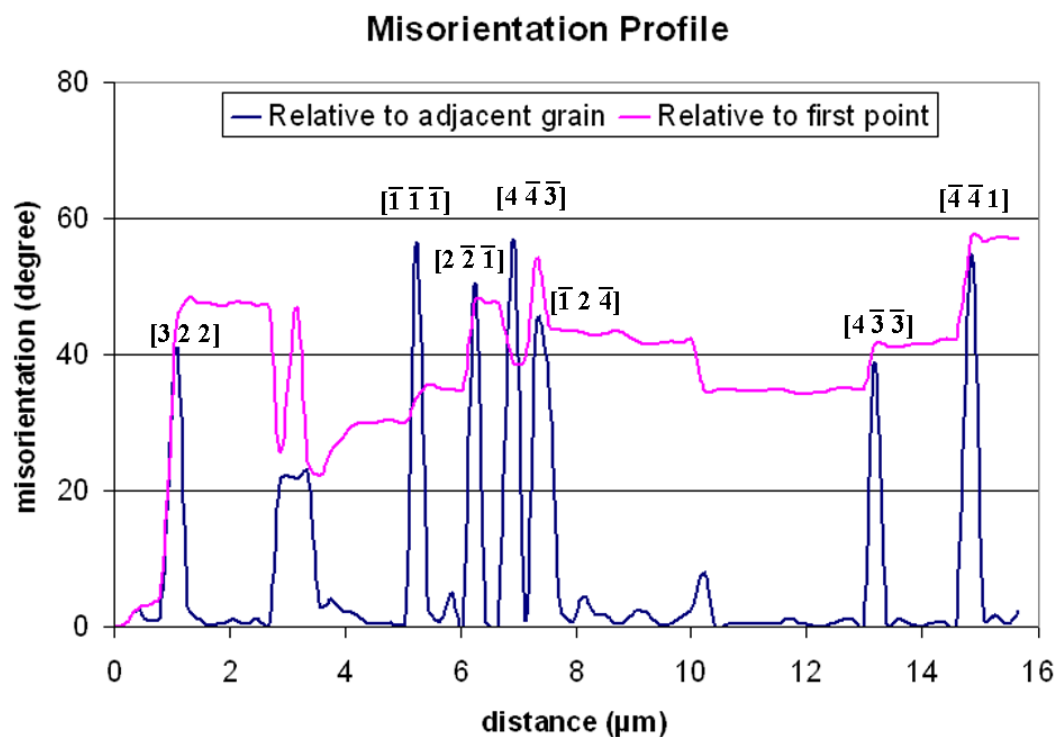
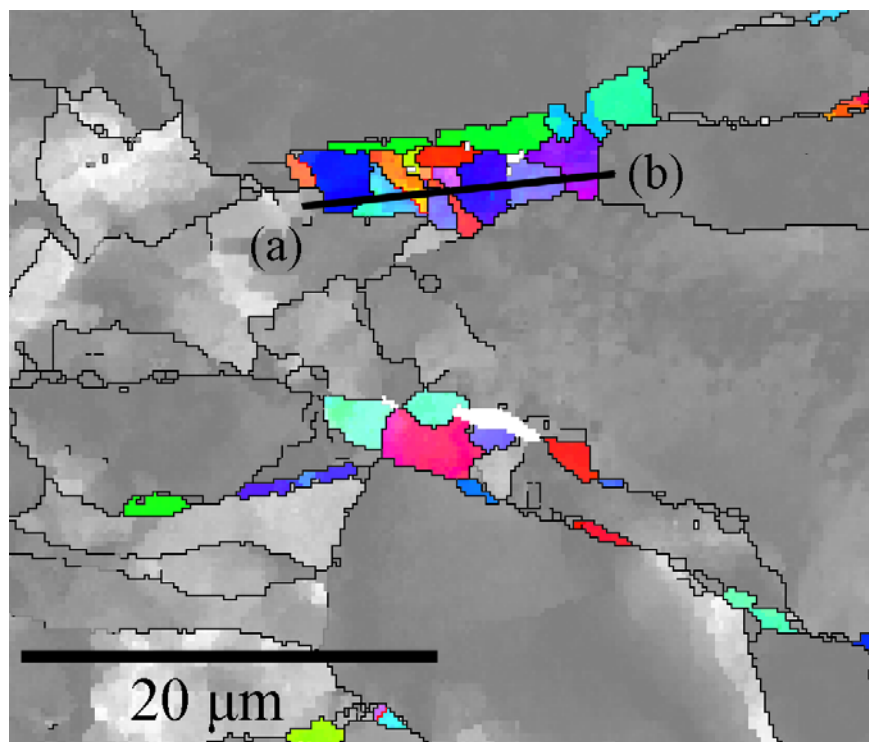


Figure B7 Misorientation profile from (a) to (b) long the black line in map above, which is subtracted from the red rectangle in Figure B6.

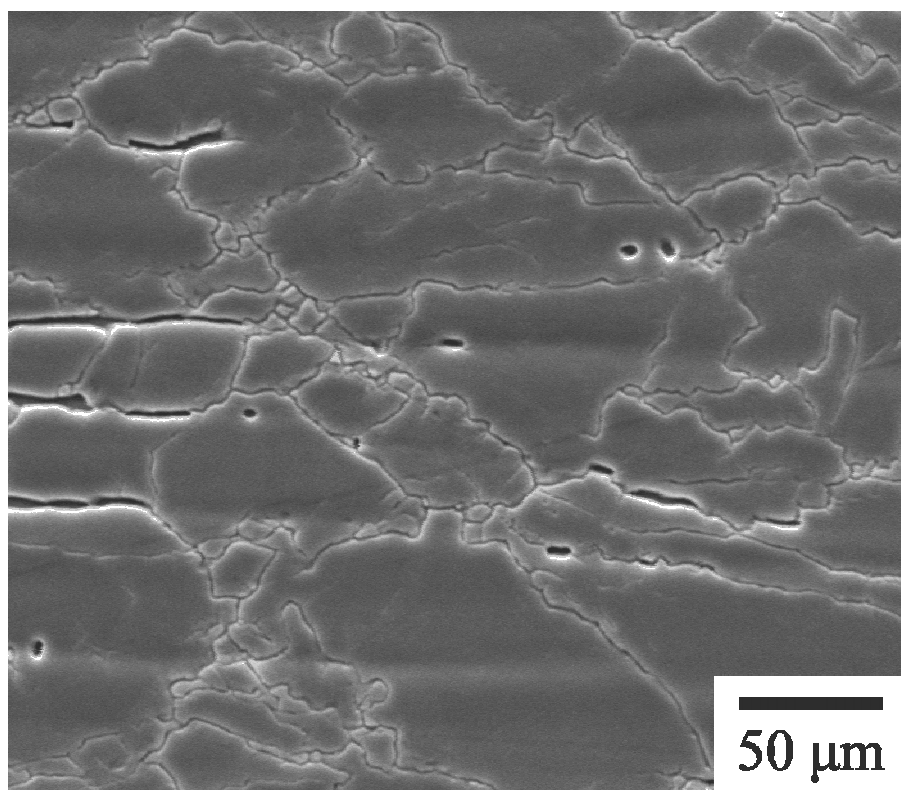
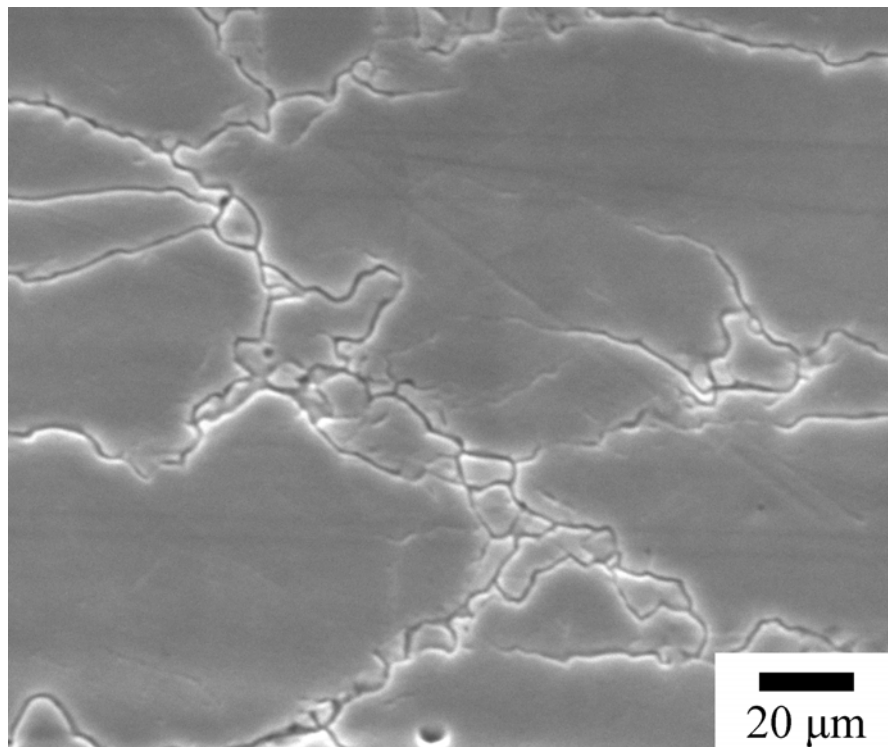


Figure B8 SEM micrographs show the existence of very small grains in testpiece SS2, after 90 seconds of electroetching using 60% HNO₃ acid and 1.1V. According to ASM handbook, this reveals only grain boundaries, but not twin boundaries.

B3.1.1 Noise Reduction for EBSD Maps

Before quantification of the microstructure can be done, and before producing the maps above, some post processing of the raw data is carried out to minimise “noise” arising from either misindexing of points or not indexing at all. This requires some considerable care to prevent introduction of artefacts into the data.

Misindexed points which exist as single pixels of dissimilar orientation to its neighbours can be removed easily. Non-indexed points (frequently at grain boundaries) can be indexed by assuming the same orientation as its near neighbours, if more than a minimum (chosen in this case as 5) number of neighbours have the same orientation as each other. A further factor involved in producing a map and quantifying grain size is the definition of a grain boundary as a particular minimum angular misorientation (typically 10 or 15°) between pixels. Grain boundaries thus identified which are incomplete because they do not terminate at a (in a 2-D cross section) triple point may be extended by relaxing the definition of a boundary to a lower misorientation, and this again can introduce changes to perceived grain size. In the case of the Ni and stainless steel, inconsistent treatment of twin boundaries which are conventionally omitted from calculations can also affect the results.

To compare the influence of post acquisition analysis on the quantitative metallography results, different noise reduction routines were applied to results from the 316 stainless steel PSC samples: routine 1, raw data with no data processing, routine 2, Remove wild spikes then grow into non-indexed area with 6 neighbours; and 3, Remove wildspikes then grow with 4 neighbours (twice). Grains less than 3 pixels were removed. Incomplete grains at map boundaries were excluded and grain boundaries were defined as 10° misorientation while twin boundaries were defined as within 5° of the ideal 60° misorientation about $\langle 111 \rangle$. Comparison between specimens SS1 and SS4 are shown in Tables B3 and B4. Further analysis based on noise reduction routine 3 is presented in Figures B9-B13.

Table B3 316 stainless steel PSC test conditions and OIM information

ID	Strain Rate	Friction	OIM size	Number of Grains*	d_{ceq}^* , μm	95%CL*, μm
SS1	0.01 s ⁻¹	Lubricated	2460 × 760 μm^2	7728	12.9	0.2
SS2	0.01 s ⁻¹	Dry	3250 × 1300 μm^2	15792	13.5	0.2
SS3	1 s ⁻¹	Lubricated	3450 × 1700 μm^2	23316	13.3	0.1
SS4	1 s ⁻¹	Dry	2850 × 1300 μm^2	15043	12.9	0.2

*after noise reduction routine 3 and 2 pixel grains removal.

Table B4 Comparison of noise reduction methods

Routine	Testpiece SS1			Testpiece SS4		
	% index	Mean d_{ceq} , μm	After removal, μm	% index	Mean d_{ceq} , μm	After removal, μm
1	81.4	5.5	10.8	87.5	5.8	11.7
2	92.0	11.4	12.1	96.1	11.7	12.5
3	99.2	12.2	12.9	99.7	12.1	12.9

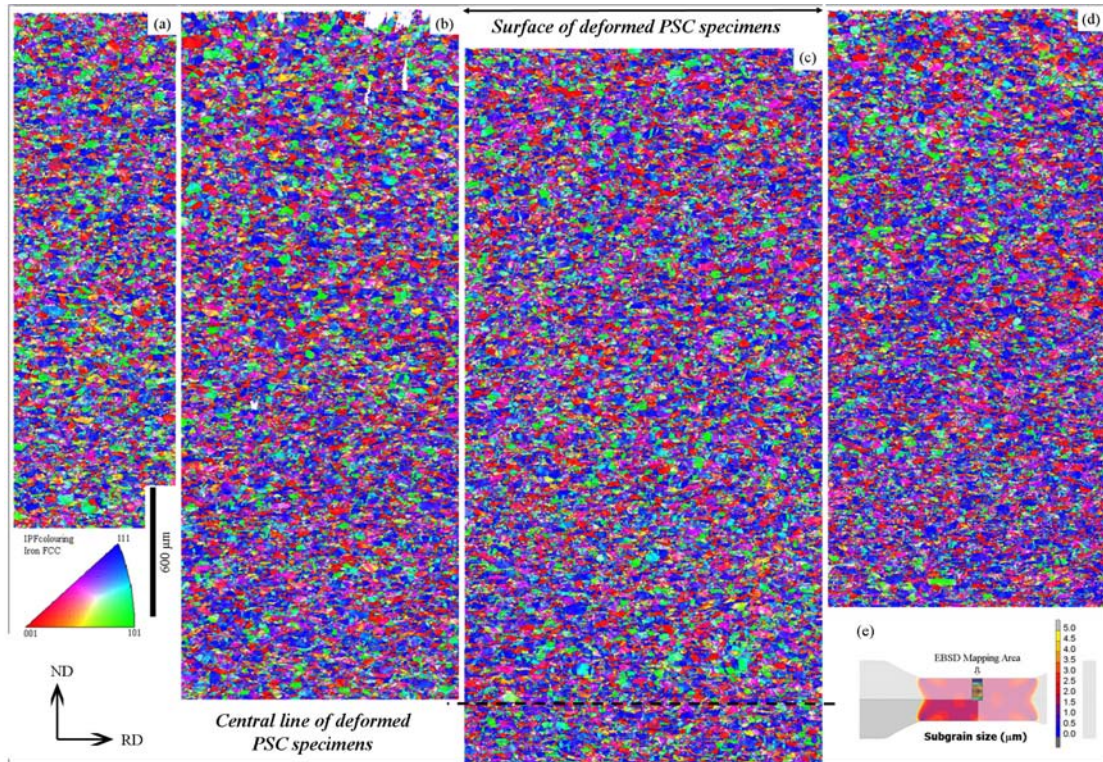


Figure B9 IPF coloured OIMs (a,b,c and d) of deformed PSC specimens; corresponding with test conditions SS1 to SS4 respectively, as summarised in Table B1; and the position of the maps is schematically illustrated in Fig B9(e).

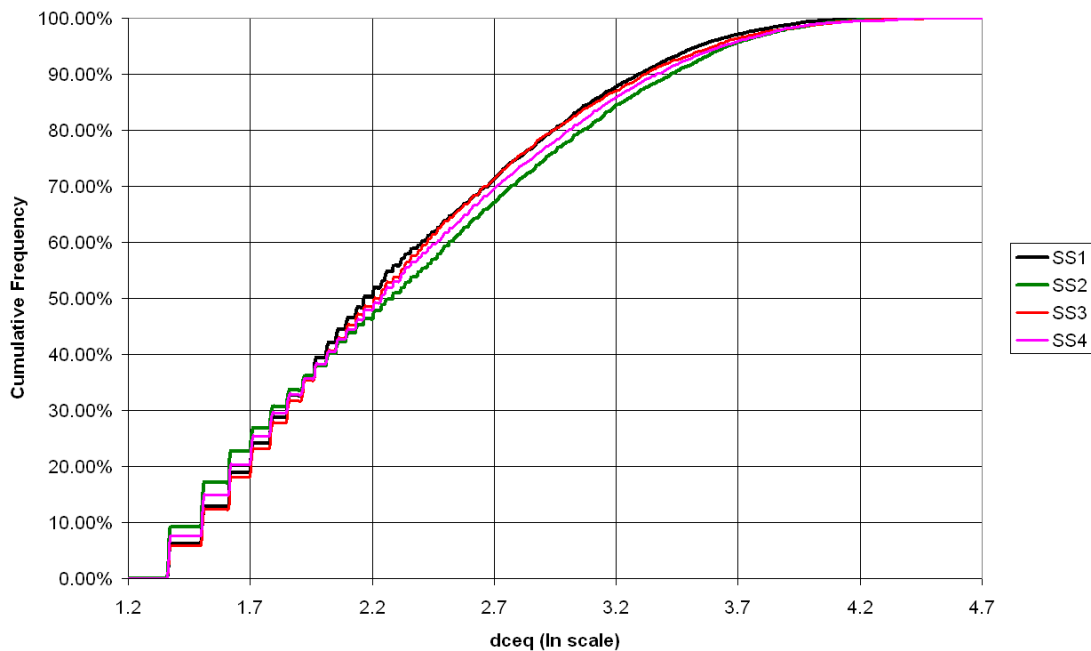


Figure B10 Cumulative number frequency plot of d_{ceq} in all four testpieces, step size of $2\ \mu\text{m}$, with grains smaller than $12\ \mu\text{m}^2$ (3 pixel) removed.

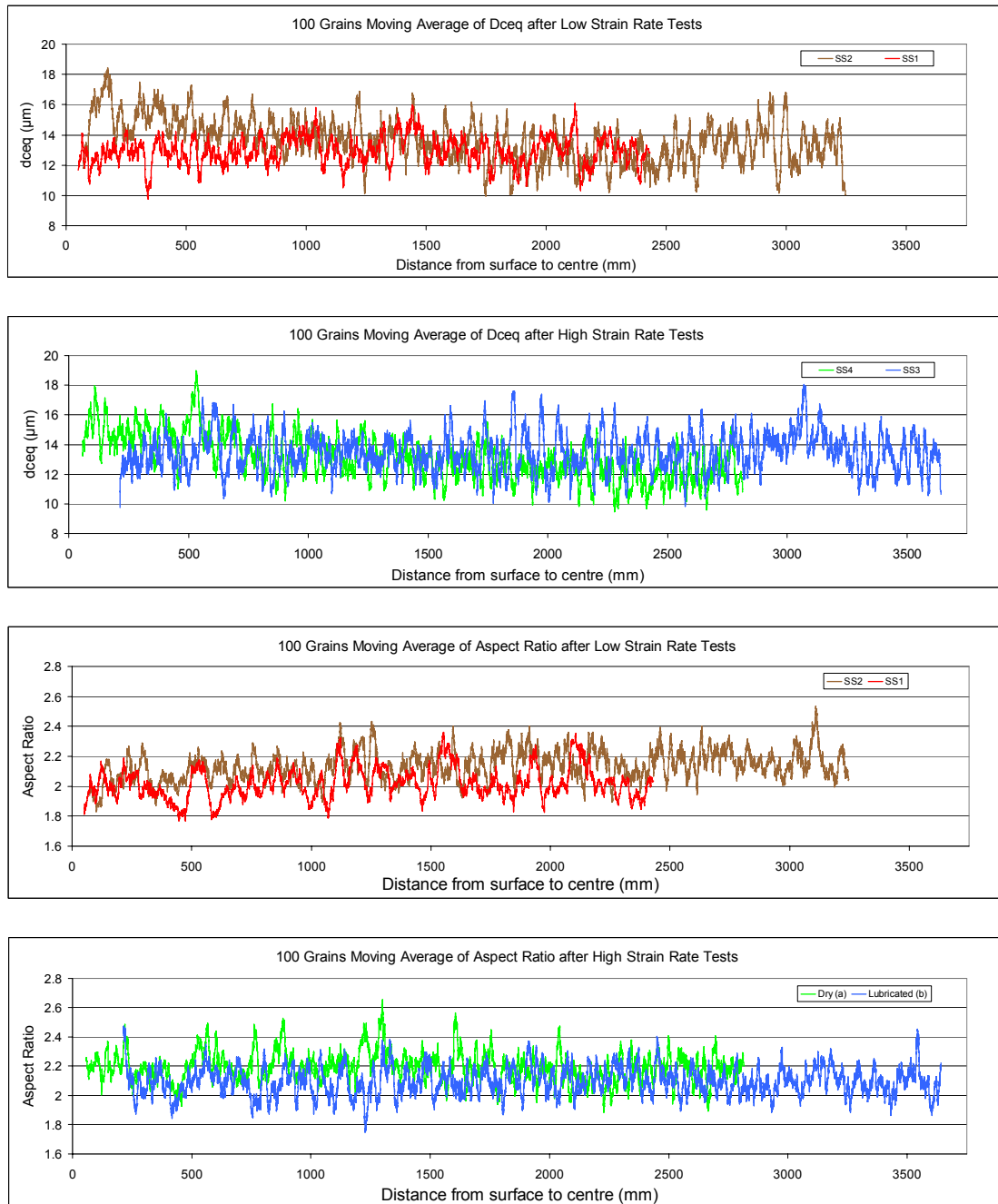


Figure B11 100 Grains moving average of d_{ceq} and aspect ratio plotted from surface to centre of the deformed PSC testpieces,

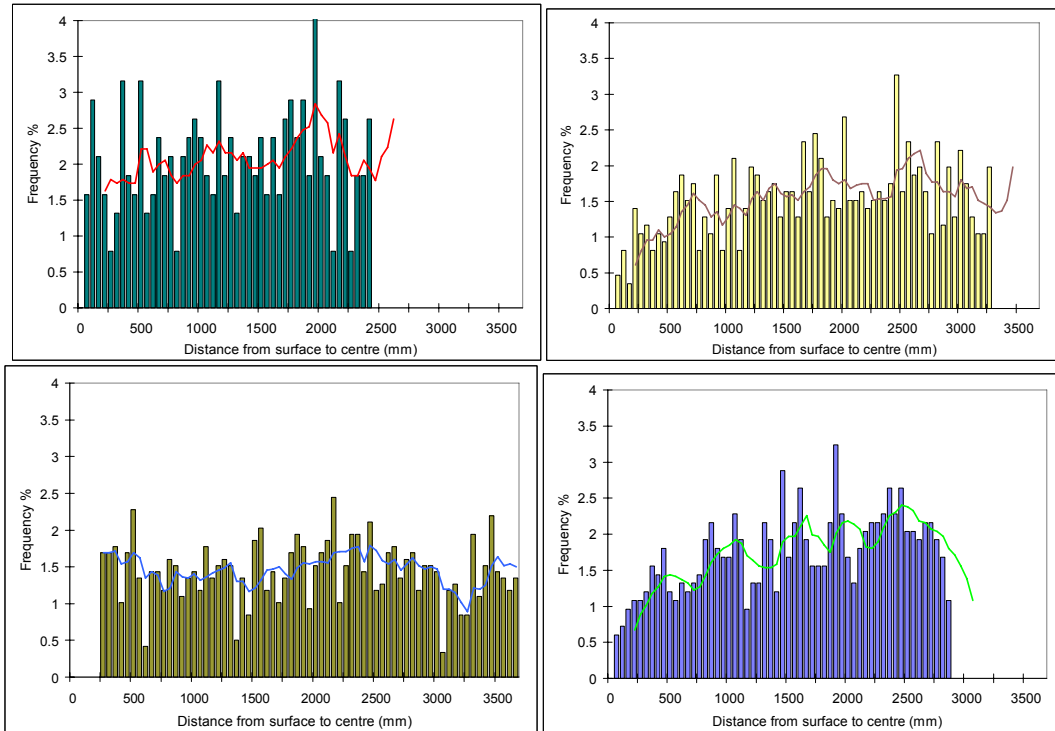


Figure B12 Histograms to show the change in frequency of occurrence with position of small equiaxed grains which have aspect ratio ≤ 1.1 and area $\leq 36 \mu\text{m}^2$ ($d_{\text{ceq}} \leq 6.8 \mu\text{m}$). Solid lines show the moving average (of 5) of the histogram data. Top left SS1, top right SS2; bottom left SS3, bottom right SS4.

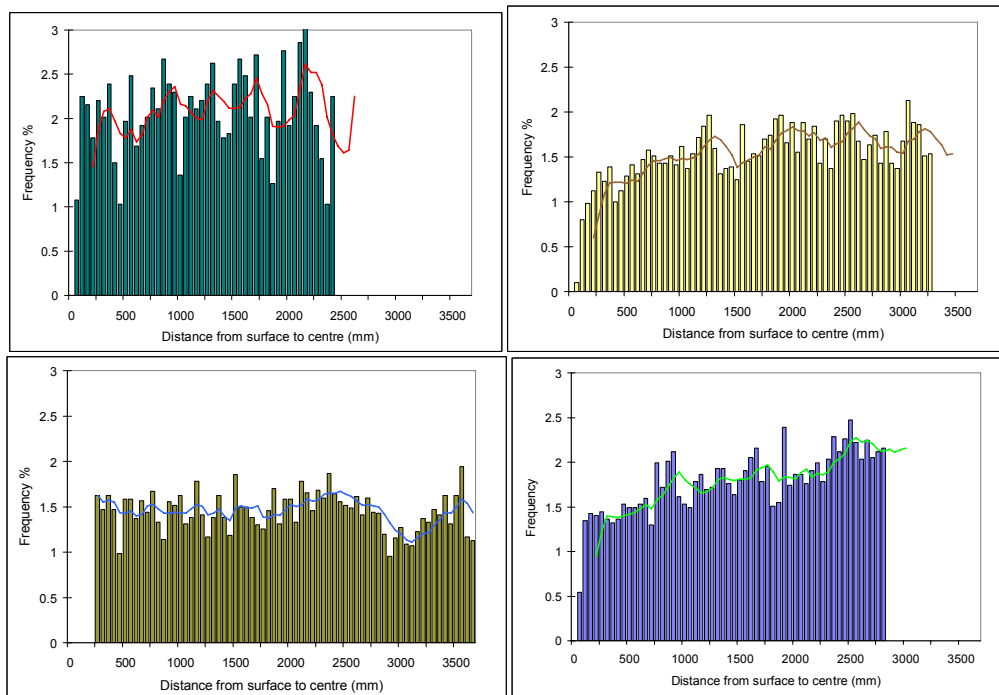


Figure B13 Histograms to show the change in frequency of occurrence with position of large elongated grains which have aspect ratio ≥ 1.9 and area $\geq 64 \mu\text{m}^2$ ($d_{\text{ceq}} \geq 9.0 \mu\text{m}$). Solid lines show the moving average (of 5) of the histogram data. Top left SS1, top right SS2; bottom left SS3, bottom right SS4

B3.1.2 Choice of Parameters to Quantify Microstructural Variation

The following gives examples of measurements from the map in Fig B2 made with grain boundary definitions of 10° as a minimum misorientation, and twin boundaries defined as within 5° of a 60° boundary and within 5° of the $[111]$ orientation. The entire area was split into map rows parallel to the compression direction, with each row made up of 8 maps stitched together; row 0 close to the centre line of the PSC specimen. Noise reduction steps were removal of single pixels, reindexing where 5 nearest neighbours exist, followed by a Kuwahara filter [B13].

Figure B14 plots parameters derived from each grain against distance from the top surface for row positions 0, 3, 6 and 9; the parameters are plotted as moving averages of 100 grains to enable trends to be more easily seen (see section B3.1.4 on the choice of the number of grains averaged). Grain size is given as the diameter of a circle of equivalent area to that of the grain, d_{ceq} ; mean misorientation is the average of the misorientations of all the pixels within a grain from the average orientation of that grain. The parameters which relate to the change of shape of the grains induced by deformation, aspect ratio and slope are derived from the feret diameters, where the slope is the angle of the maximum diameter to the compressive axis.

B3.1.3 Use of Data Subsets to Analyse Microstructural Variation

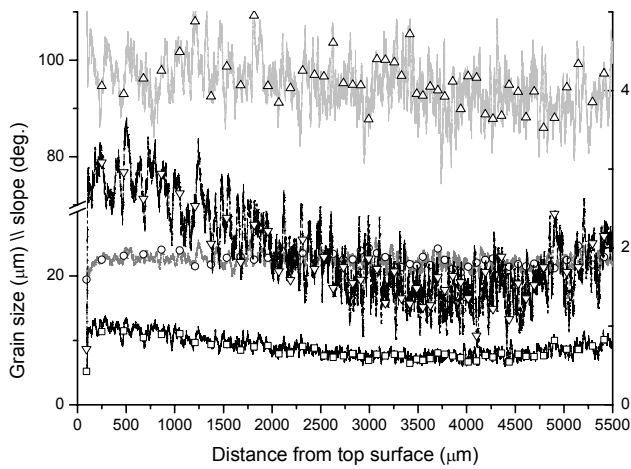
While significant differences can be seen in the trends of grain size and mean misorientation, the parameters relating to grain shape appear to show little trend with position. If, however, the data is split into sets of those grains $>10\ \mu\text{m}$ and $<10\ \mu\text{m}$ and the same graphs are plotted, different trends are observed and trends in slope and aspect ratio become more evident for the large ($>10\ \mu\text{m}$) grains. Indications of the density of each of the grain fractions can also be deduced from the variation with distance of the noise level of the moving averages. The changes seen by considering the different size fractions is not surprising when consideration is given to the relative number of grains in each fraction and the actual cross sectional area they represent. For example, in row 9, the grains less than $<10\ \mu\text{m}$ in diameter account for 48.4% of the total number but only 5.2% of the total area; in row 0 the figures for grains $<10\ \mu\text{m}$ are by 78.4% by number and 17.2% by area.

Conventionally, grain size has often been measured by linear intercept methods using grain boundaries manually identified from etched samples. Figure B15 shows equivalent linear intercept measurements derived automatically from the EBSD data; in this case the raw data is shown behind the moving averages. Two lines were superimposed on the row 0 map, giving 1134 intercepts, in comparison with a total of 24394 grains by area. The moving averages agree surprisingly well, even though the linear intercept method produces far fewer large measurements.

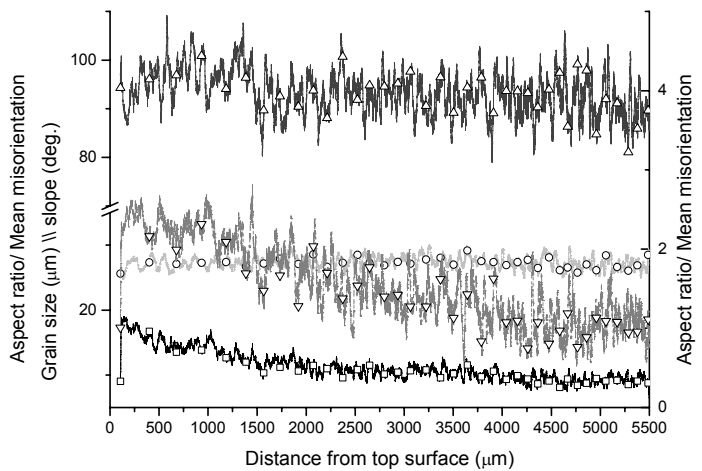
The improved visualisation of the process by examining subsets of the data is also more apparent if applied to the maps themselves. Figure B16 shows grains $>30\ \mu\text{m}$ in diameter (d_{ceq}), Fig B17 shows the mean misorientation plotted for each of the columns for all the data available and Fig B18 the grains with mean misorientations $<2^\circ$. The necklace structure of recrystallised grains, occurring in the shear band from bottom left of row 9 and running diagonally across the maps to peak towards the mid line of the PSC sample in row 0 is evident in Fig B18, as is the increasing deformation of large unrecrystallised grains in Fig B16.

The Ni alloy sample has clearly partially recrystallised, particularly in shear bands running from the PSC shoulder to the centre of the sample. As a result there is a wide range of grain sizes, from the small, largely recrystallised grains to the large highly strained grains. It has already been noted that when considered in terms of numbers, the small grains bias the results when presented as simple averages and obscure changes in the microstructure, and an arbitrary cut off of 10 μm was chosen to look at the effects on the majority of the sample in area terms. If in fact a map of sub 10 μm grains (Fig B19) is compared with a map of grains with mean misorientation $<2^\circ$, then a reasonable correspondence is seen, suggesting that the 10 μm cut off was a good choice to distinguish between recrystallised and non-recrystallised grains in this case. The data available from EBSD means that it is as easy to sort grains by their level of misorientation as it is by grain size, so it would be good practice to look at grain size variation by levels of misorientation in similar cases of partial recrystallisation. Similarly EBSD enables measurement of aspect ratio for individual grains and thus permits the variation of this parameter with grain size to be analysed.

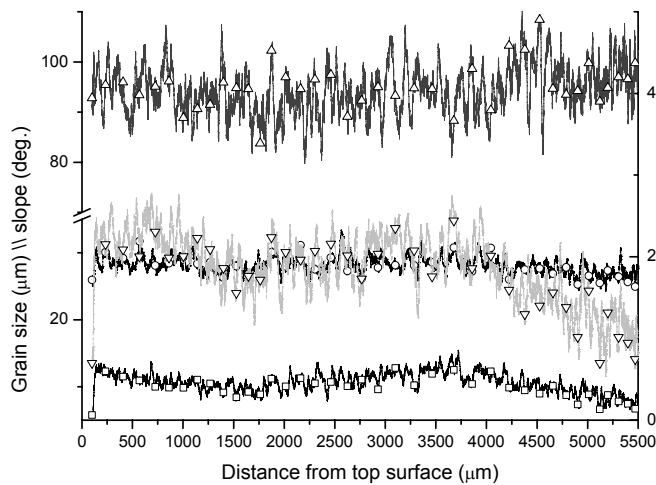
Figure B20 shows an example from the central axis of an Al alloy, equivalent to row 0 from the Ni superalloy map. This illustrates a number of further issues. Firstly and most obviously, significant regions were not indexed because of electropolishing problems caused by the presence of precipitates (as a result, re-indexing of non-indexed points was applied twice with a criterion of 4 near neighbours); where these non indexed regions cross entire grains then clearly grain size will be underestimated. Secondly, although it is not immediately visible in the map, the deformation of grains is such that there are a significant number of incomplete grain boundaries, so if extrapolation of these boundaries to join other boundaries is allowed, further changes can be made to the calculated grain size. The remaining point to make is that the average grain size is significantly larger than that of the Ni superalloy, although the step size used was identical. Figure B21 shows graphs similar to those of Fig B14, with the grain size, mean misorientation and aspect ratio plotted for all grains and then for grains $>10\mu\text{m}$ in diameter.



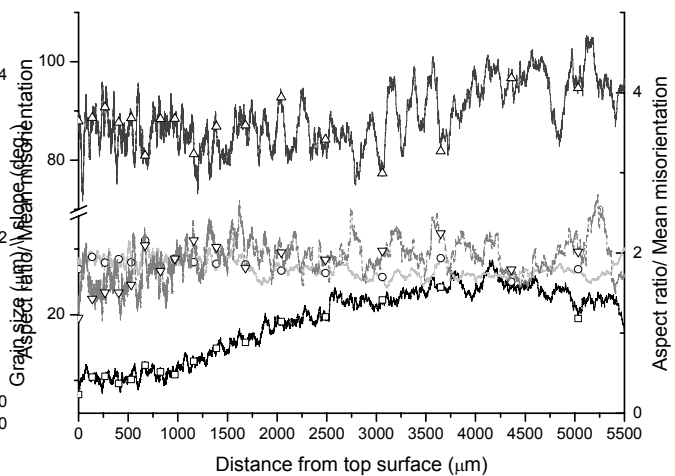
B14a centre line
(for legend see next page)



B14b 1/3 height from top

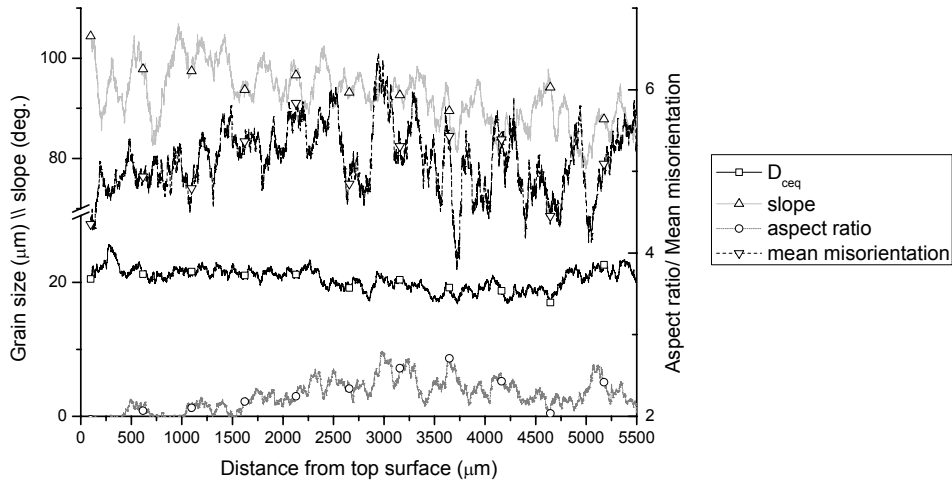


B14c 2/3 height from top

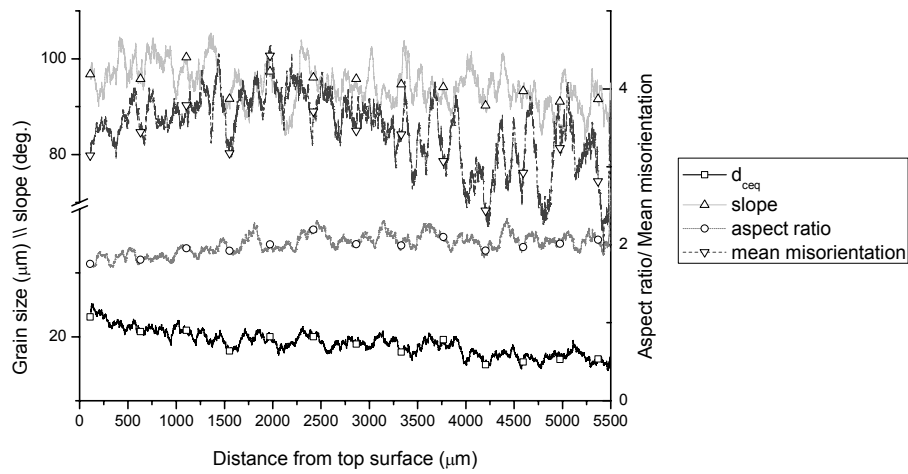


B14d shoulder

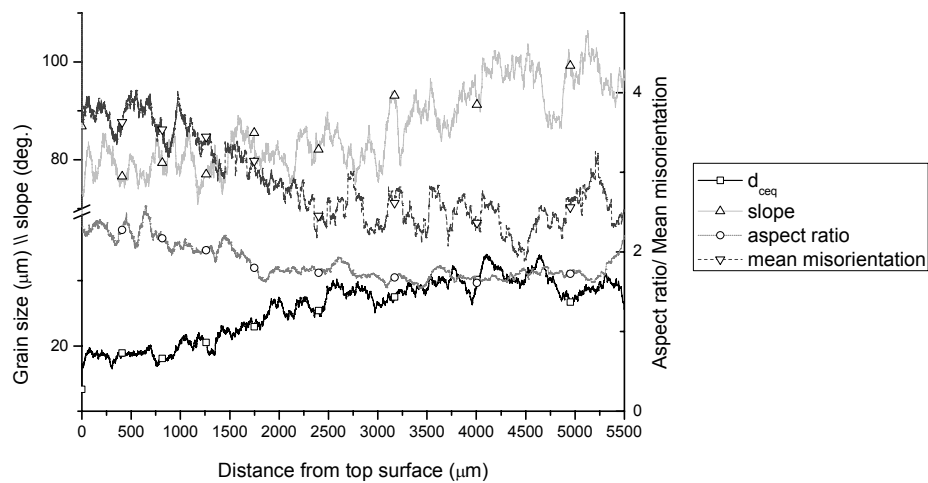
Figure B14 Microstructural parameters from strips indicated in Fig B2. Graphs show moving averages (100 grains) of circle equivalent diameter grain size, d_{ceq} , grain slope (angle of principle axis to compression direction), aspect ratio and mean misorientation for: B14a to B14d, rows 0, 3, 6 and 9 respectively, where row 0 is along the approximate centre line of the specimen, at the top of Fig B2, row 3 is at 1/3 height from top, row 6 at 2/3 height from top, and row 9 at the shoulder of the PSC specimen, at the bottom of Fig B2.



B14e centre line, >10 μm grain size data



B14f 1/3 height, >10 μm grain size data



B14g shoulder, >10 μm grain size data

Figure B14 Microstructural parameters from strips indicated in Figure B2 (cont.); e-g) grains with diameter (circle equivalent) >10 μm for rows 0, 3 and 9 respectively

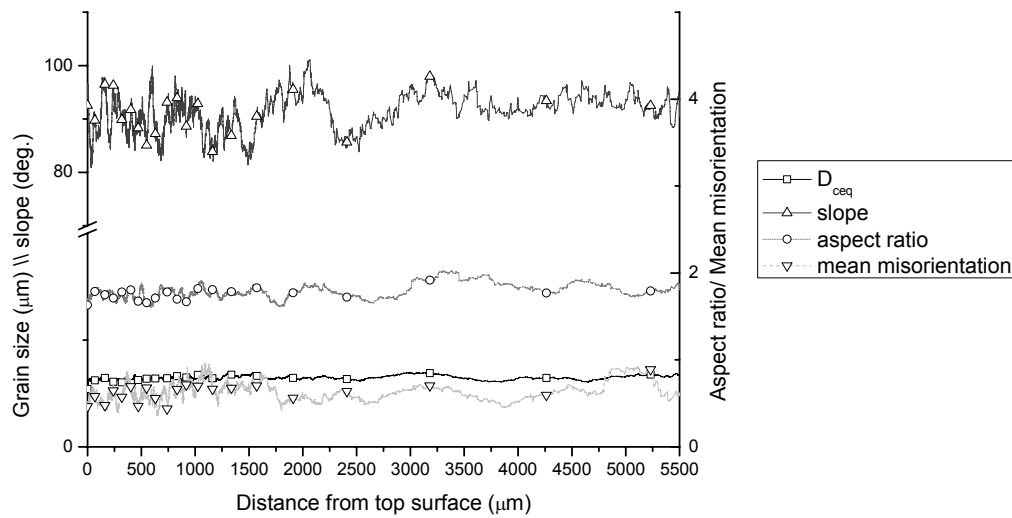


Figure B14h shoulder row, <10 μm grain size data

Figure B14 Microstructural parameters from strips indicated in Figure B2 (cont.); h) grains with diameter (circle equivalent) <10 μm for row 9.

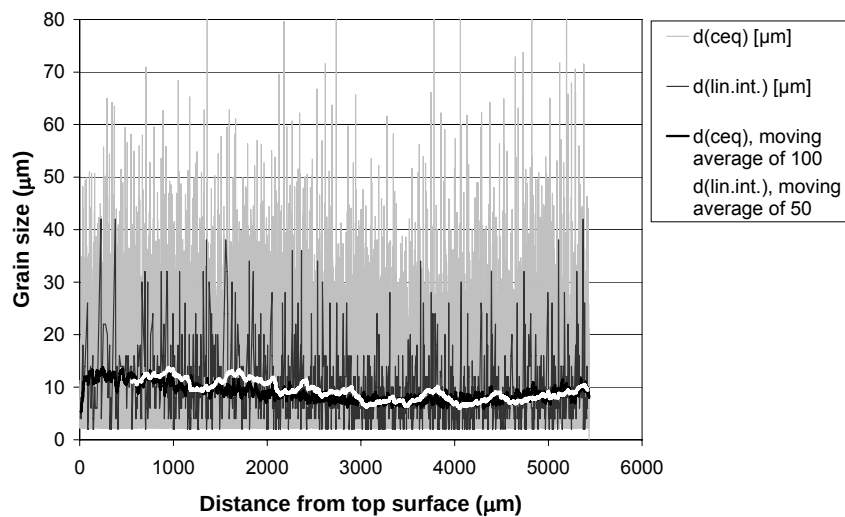


Figure B15 Graph showing individual grain size measurements and moving averages of grain size measured by both circle equivalent diameter d_{ceq} and linear intercept d_{lin} for row 0.

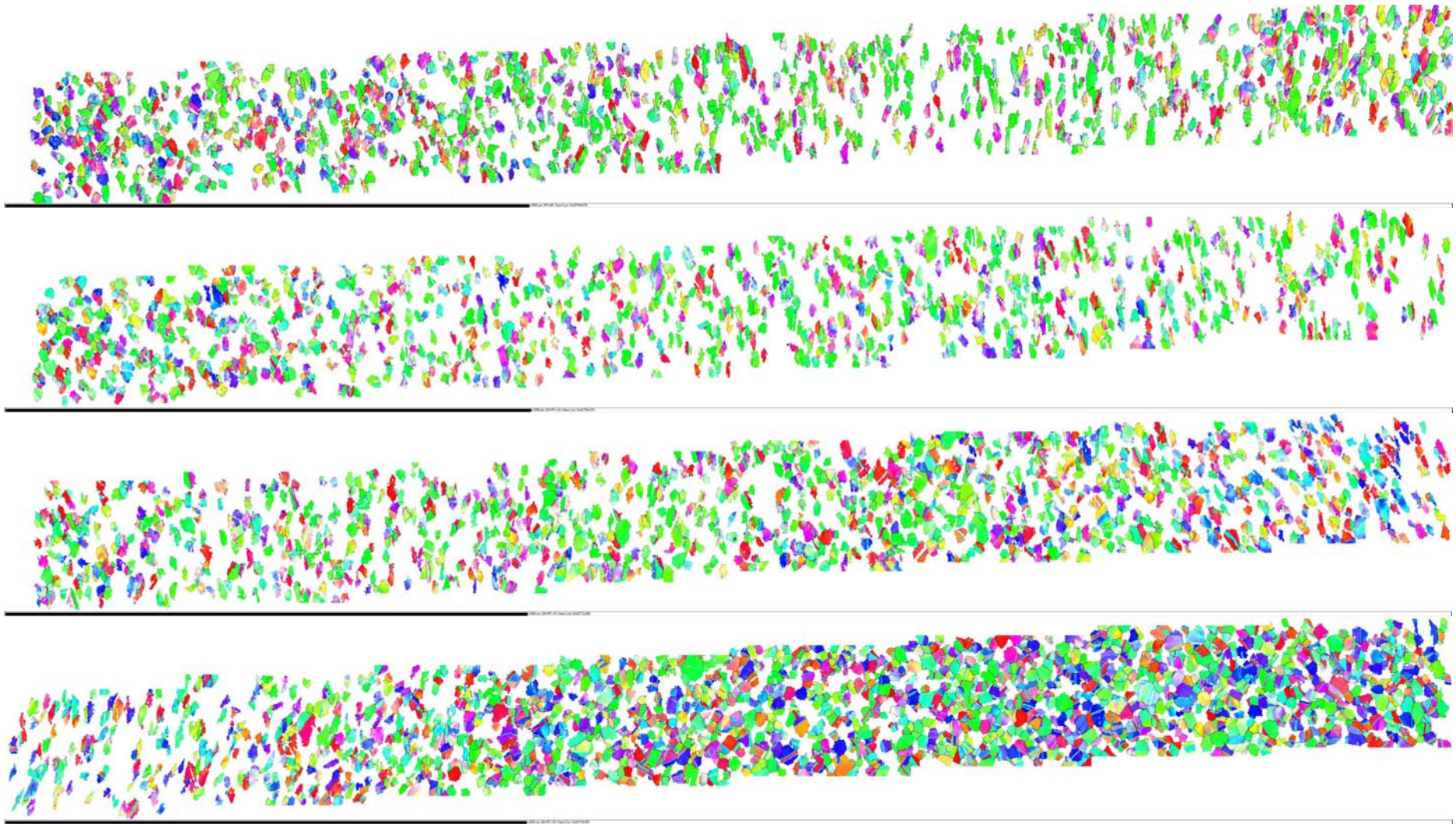


Figure B16 EBSD orientation maps showing grains $>30\mu\text{m}$ in diameter (d_{ceq}) in selected regions from Fig B2 for the same rows analysed in Fig B14. From top to bottom, maps are for rows 0, 3, 6 and 9 respectively. Inverse pole figure colour scheme shown in Fig B2 used. Scale marker = 2mm

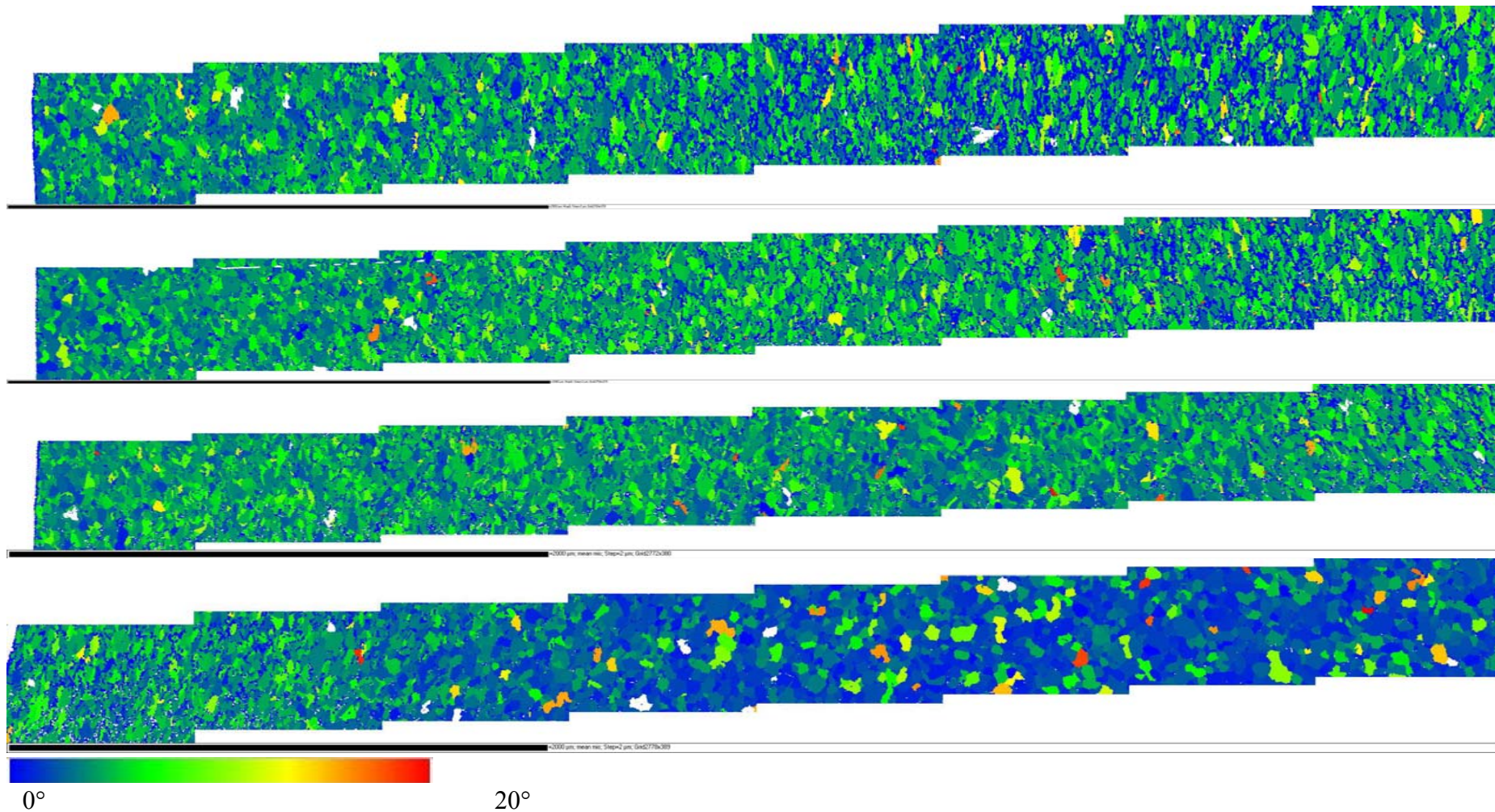


Figure B17 EBSD maps showing the mean misorientation in each grain, according to the colour scale shown with blue indicating unstrained (recrystallised) grains through to red, highly deformed grains with 20° misorientation. Map positions the same as those in Fig B14.

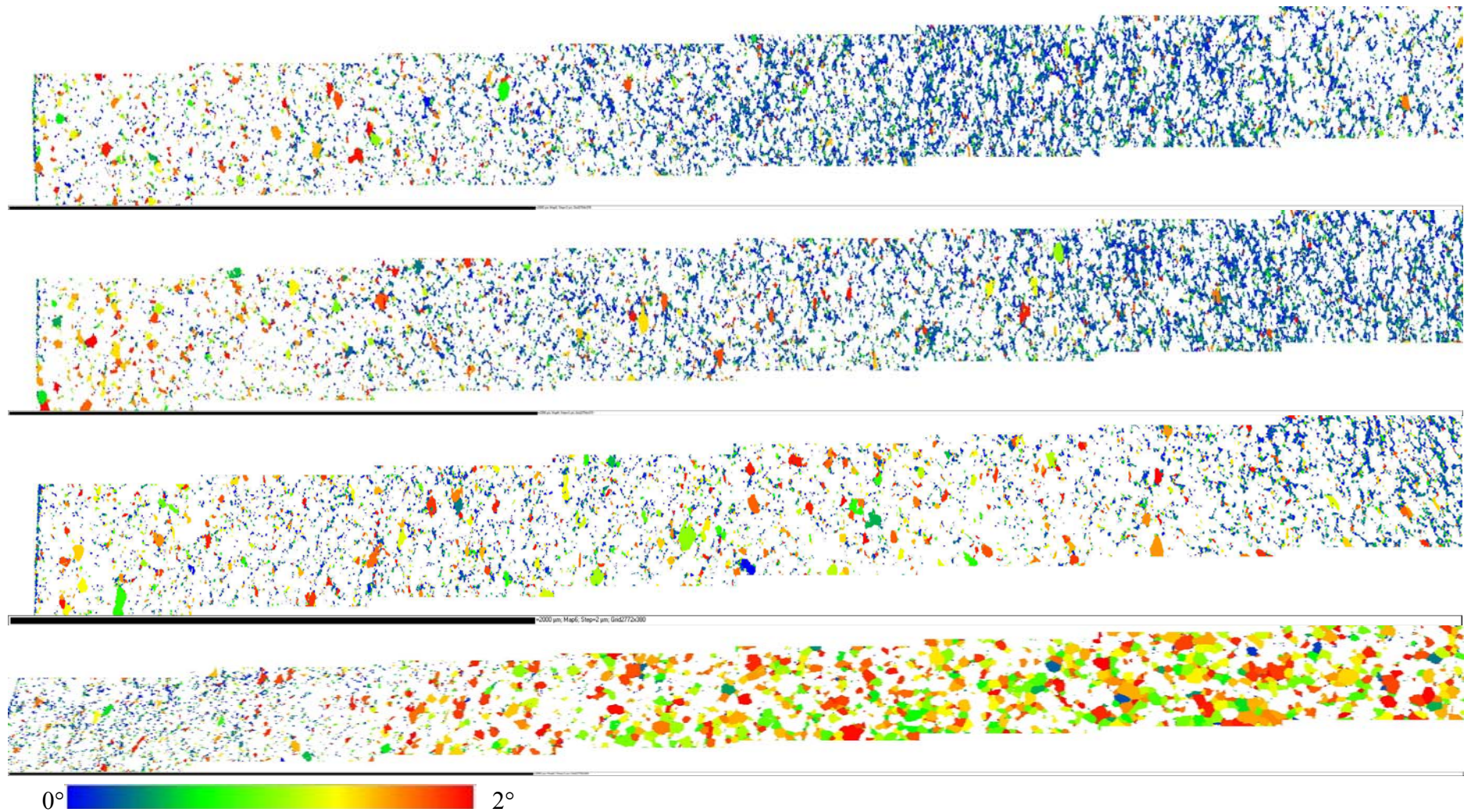


Figure B18 EBSD maps showing the mean misorientation in each grain only for grains with $<2^\circ$ misorientation, i.e. relatively unstrained grains, according to the colour scale shown. Map positions the same as those in Fig B14.

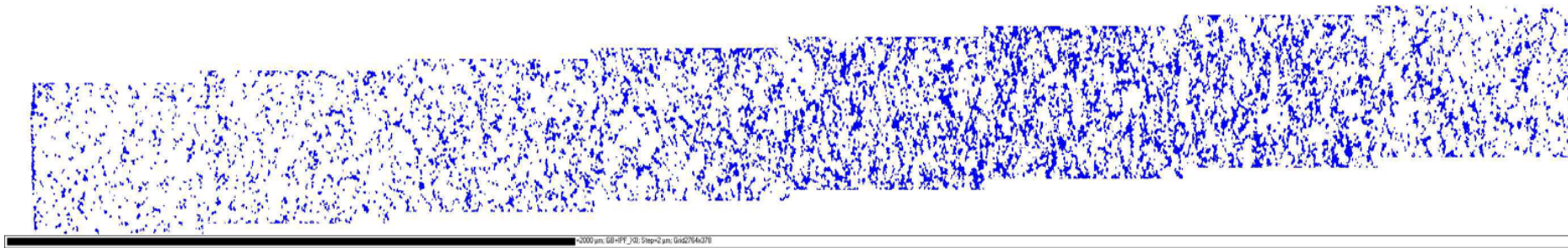


Figure B19 Map of sub 10 μm grains for comparison with grains with misorientations $< 2^\circ$ (Fig B18) in row 0.

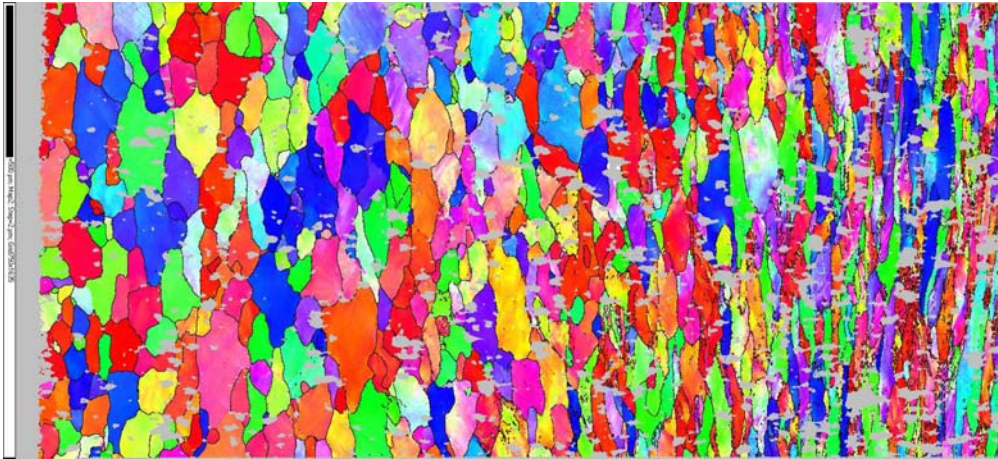
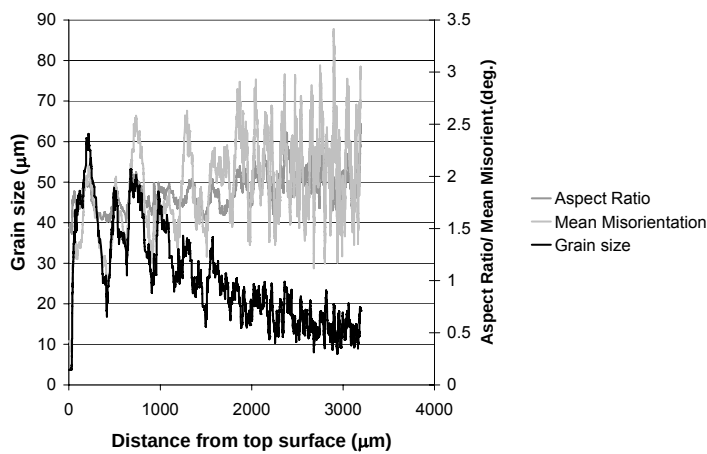
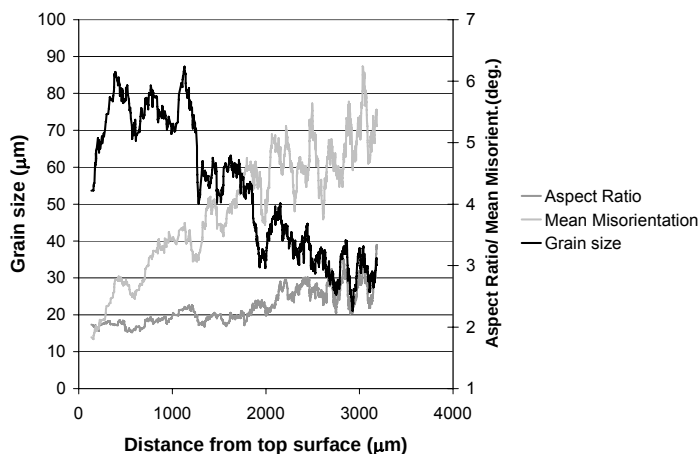


Figure B20 EBSD orientation map of Aluminium PSC sample taken from the surface to mid-height along the centre line of the sample. Mapped with a 2 μm step size. Scale marker 500 μm .



B11a



B11b

Figure B21 Graphs showing moving averages (100 grains) of grain size, grain orientation (angle of principle axis to compression direction), aspect ratio and mean misorientation for:

- all grains shown in Fig B20.
- only grains >10 μm in diameter (circle equivalent) in Fig B20.

B3.1.4 Averaging of Data

The example in Figure B20 showed very large changes in grain size from surface to centre of the sample. This can present issues in deciding how best to average the data, as will be illustrated by another example taken in this case from an axi-symmetric compression sample.

Figure B22 shows an EBSD orientation map for a Ni superalloy (Waspaloy) with inverse pole figure colouring. The map was acquired with a $1\mu\text{m}$ step size from the centre line of an axial cross section of an ASC sample. The sample was produced with relatively high friction between sample and platens, resulting in a region of large unrecrystallised grains close to the surface while the inner region consists of many smaller recrystallised grains. Noise reduction was performed by removing wildspikes of single pixels and then growing into non indexed pixels with 4 neighbouring pixels of the same orientation. Further analysis was carried out by removing all grains of 3 pixels ($d_{\text{ceq}} \approx 2\mu\text{m}$) or less.

Figure B23 shows the variation of d_{ceq} (prior to the removal of 3 pixel grains) with distance from the top surface by plotting the size of each grain (ignoring those impinging on map edges) against its distance from the top surface irrespective of its lateral position. Superimposed on the raw data is the moving average calculated over 50 grains which shows the trend in grain size more clearly and also emphasises the variation in the distribution from the edge to the centre of the sample.

Figure B24 plots the moving average line of Figure B23 together with the moving average line derived in the same way from the data in which all 3 pixel grains or smaller were removed. The peaks of both lines correspond very closely in the bulk of the sample but the effect of the small grains is seen close to the surface where the average is increased by their removal by over 25% in some places, and in the troughs of the moving average line further from the surface. This shows that removal of small clusters of pixels needs to be considered in relation to the region around them. If, relative to the average grain size, the cluster size is small, their removal can have a large effect as shown close to the surface in Figure B24. Their effect on reducing the troughs rather than the peaks suggests that the 3 pixel clusters are themselves clustered in regions or bands. These effects can be reduced if the moving average is taken over a smaller number of grains. Figure B25 repeats the moving average data after 3 pixel removal and adds lines to show moving averages over 25 and 10 grains. As is to be expected the plots become noisier as the number of grains averaged over is reduced, but the maxima are particularly accentuated near the surface where there are fewer large grains, so the overall change in size appears larger. The choice of moving average should thus be selected according to the number of grains analysed relative to the rate at which the average size changes. If there is rapid change then it may be more appropriate to divide a region into equal areas calculate the average for each area and then plot both average and number of grains for each area against position.



Figure B22 EBSD map of grain orientation in a section taken from a uniaxially compressed sample of 316L stainless steel. Top surface on left of map, centre of sample on right.

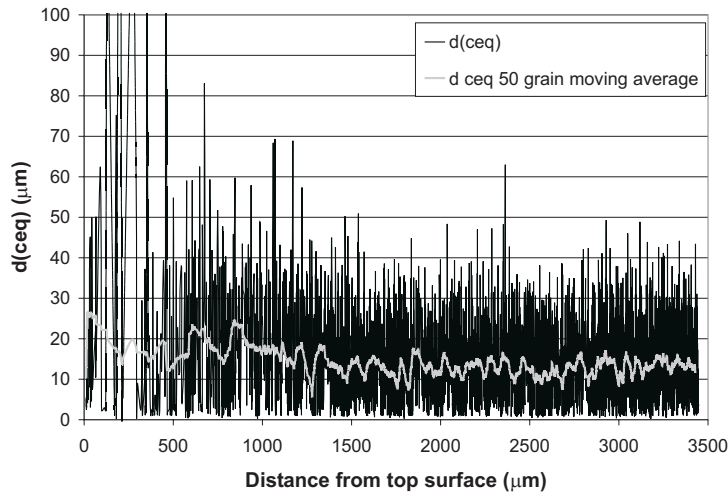


Figure B23 d_{ceq} plotted against distance from the top surface for all grains in map in figure B22, with superimposed moving average of 50 grains (value for succeeding 50 further from top surface so that average starts at surface)

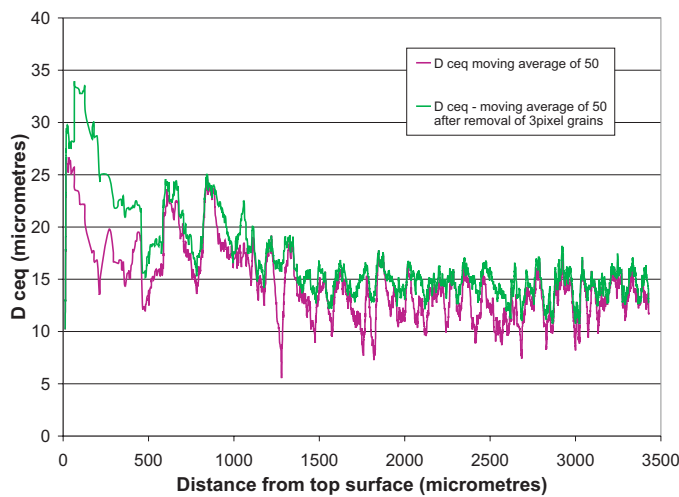


Figure B24 Moving average of 50 grains shown in Figure B23 together with the moving average line derived in the same way from the data in which all 3 pixel grains or less were removed

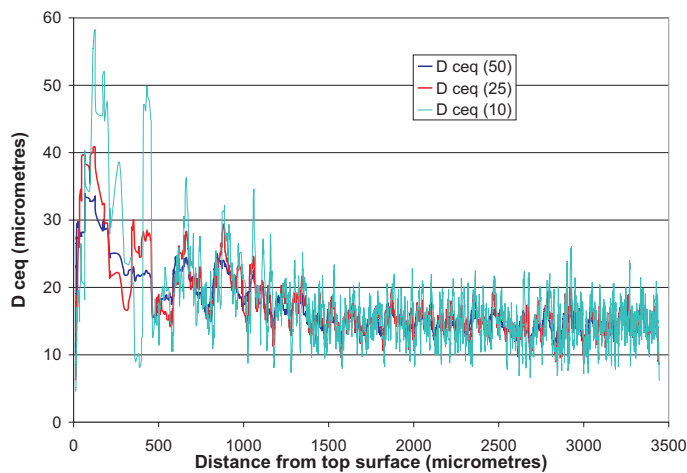


Figure B25 Moving averages of 50, 25 and 10 compared with data with 3 pixel grains removed.

B3.2 Effect of EBSD Step size

The preceding discussion used similar acquisition procedures and subsequent analysis with the principle parameters being a 1 μm or 2 μm step size with grain boundaries defined by a 10° misorientation. It was assumed that the data for the small grains was valid, even though there are a large number of small grains only 1 or 2 pixels in size, even after noise reduction routines have been applied. Mapping a small representative area of the Ni alloy at a much smaller step size of 0.075 μm , shown in Fig B26, confirms that the general necklace structure of fine grains seen with a 2 μm step size is correct and that almost all of the one- or two-pixel grains are likely to be real. In fact, many smaller grains are missed at the coarser step size. In Fig B26 grains as small as 0.25 μm in diameter can be seen in addition to a wealth of detail of the deformation of individual large grains and twin formation in newly recrystallised grains.

Thus, where recrystallisation has occurred in a microstructure, refining the step size can reveal increasing numbers of small grains and the standard EBSD procedure of ignoring grains of fewer than a specified number of pixels as poor indexing “noise” is difficult to justify. Treating grains in classes of size or misorientation as in the above example is one approach. Another possibility is illustrated by the area shown in Fig B27a-c, which shows EBSD maps acquired at 2, 1 and 0.5 μm step size of a fully recrystallised region of an AC Waspaloy sample. Figure B28 shows the mean misorientation of the area on the same scale as that of Fig B18; although the misorientations are at the higher end of the scale, 90% of the grains fall within the colour scale instead of <20%.

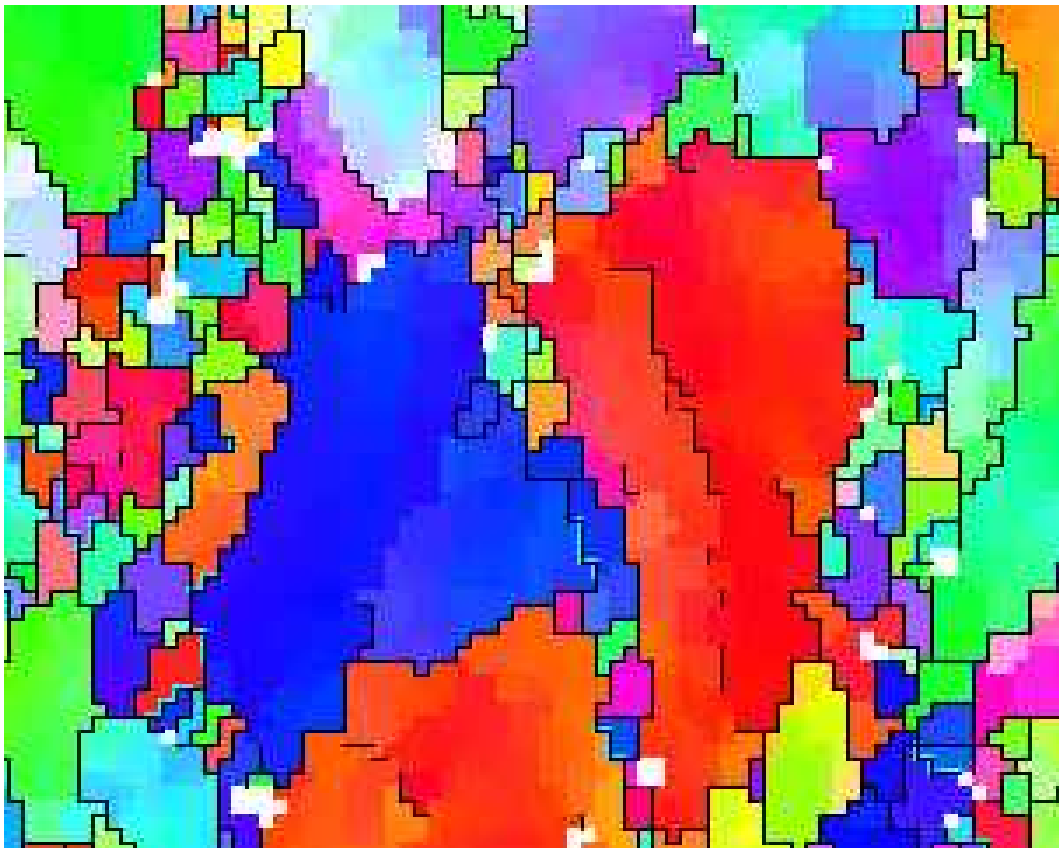
After a standard noise reduction procedure (remove single pixel wildspikes, index pixels with 5 near neighbours, apply a Kuwahara filter) and using a grain boundary definition of 10° , with extrapolation of incomplete boundaries to 5° , mean grain sizes were measured as:

Step size	2 μm	1 μm	0.5 μm
Number of grains	242	265	304
Mean grain size (μm)	16.9	15.7	13.7

There was a difference in calculated mean grain size of nearly 25% between the largest and smallest step size. This difference, given the limited number of grains, might for a normal uniform microstructure be considered as not statistically significant. However, these are measurements on identical areas and there is a need to know how to obtain consistent results if similar sized regions of heterogeneous structures are to be compared with large size gradients, perhaps using different step sizes for the different regions. The preferred option to remove “noise” arising from the small step size is, instead of removing all grains less than a given diameter, to eliminate all grains with 2 or less neighbours, which results in mean grain sizes within 10% of each other:

Step size	2 μm	1 μm	0.5 μm
Number of grains	231	248	250
Mean grain size (μm)	17.5	16.6	16.1

B26a



B26b

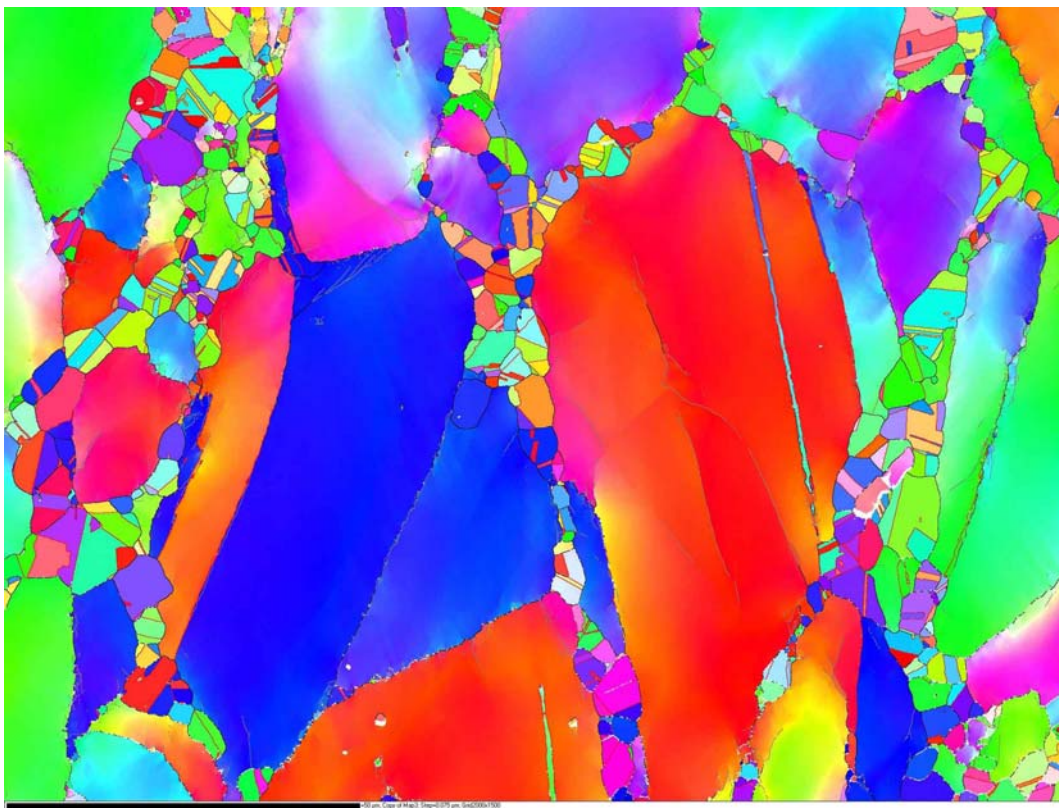
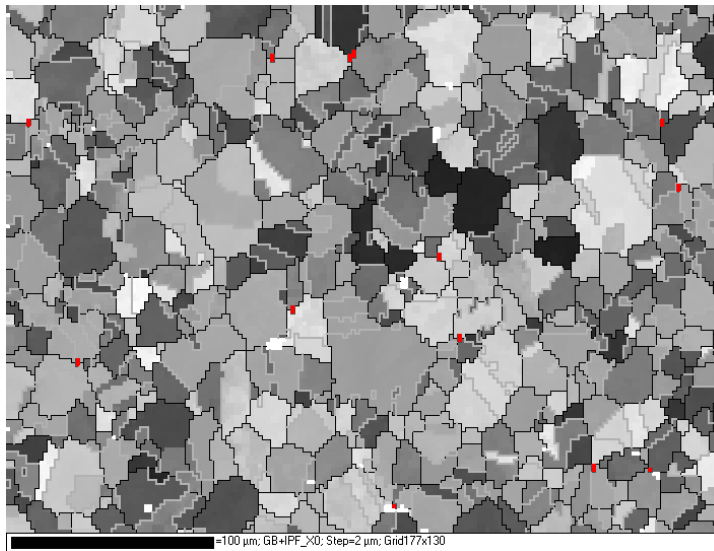
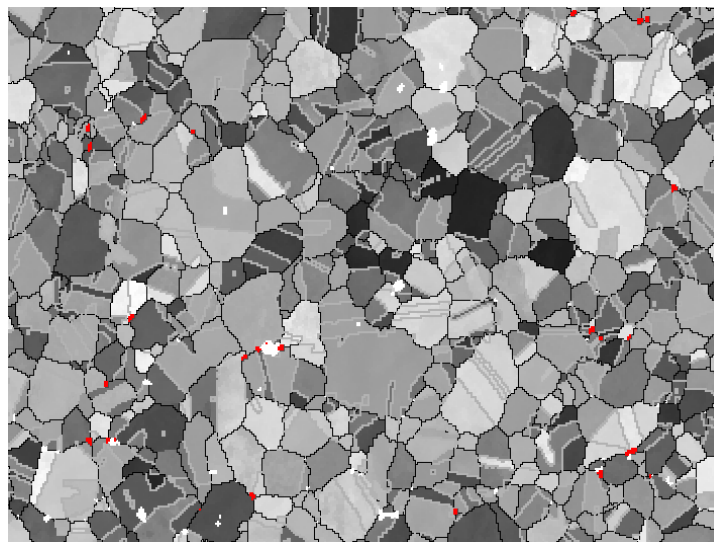


Figure B26 Effects of small step size. EBSD orientation maps (IPF colouring) of the same area at two different step sizes: a) 2 µm, the same as all maps previously shown, and b) 0.075 µm. The scale marker bar is 50 µm.

B27a



B27b



B27c

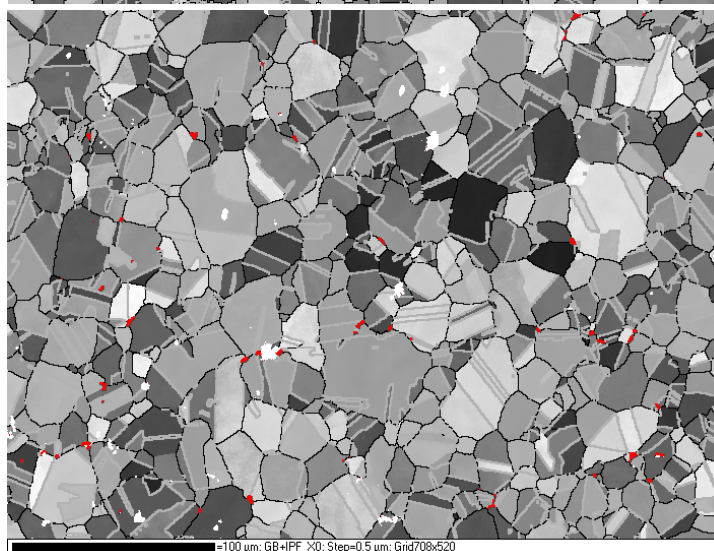


Figure B27 EBSD orientation maps of the same area of a fully recrystallised sample, acquired with 3 different step sizes: a) 2 µm, b) 1 µm, and c) 0.5 µm (inverse pole figure orientation colours converted to an arbitrary grey scale). Scale marker bar 100 µm

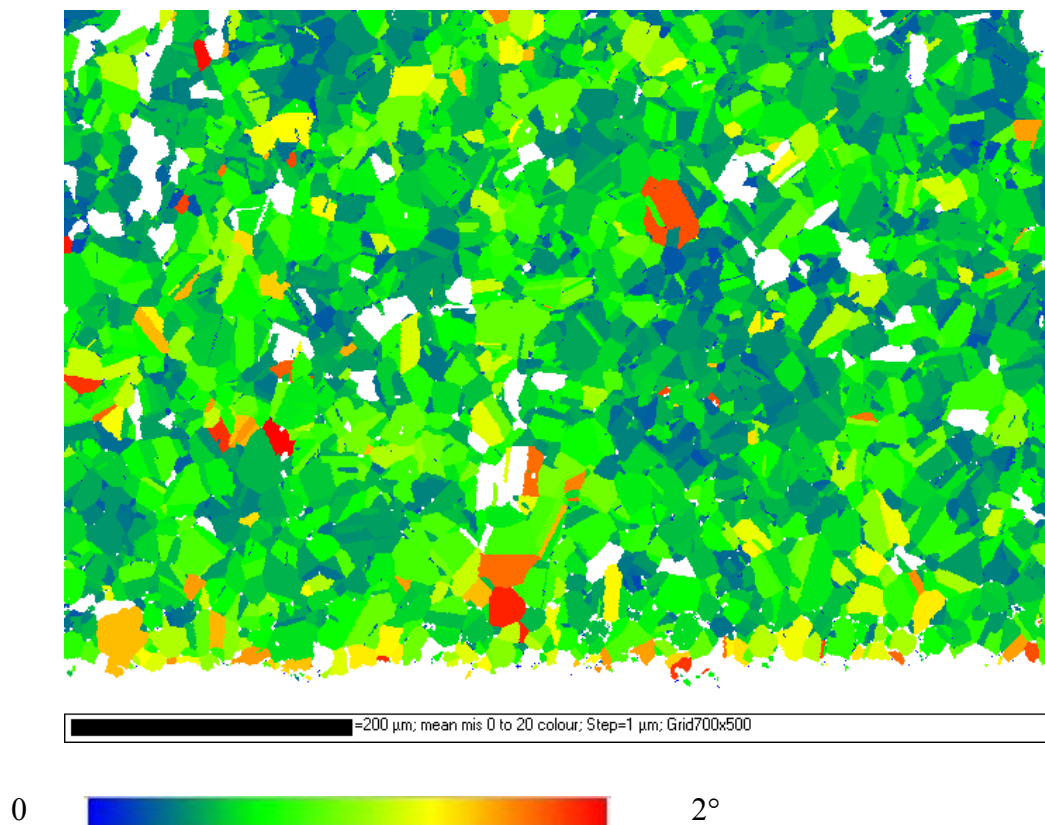


Figure B28 EBSD mean misorientation map of the fully recrystallised sample shown in Fig B27, with a scale to show misorientations between 0° to 2° . Scale marker bar $200\ \mu\text{m}$

Justification of this approach is based on the assumption that in a fully recrystallised structure, even small grains will occur at a triple point (in a 2-D cross section) and thus have a minimum 3 neighbours; 1 or 2 neighbours implies grains either completely surrounded by another grain or adjacent to a non-indexed region (caused perhaps by a precipitate or surface contamination) where by definition poor diffraction patterns that might be misindexed are likely to be produced. If number of neighbours is plotted against grain size for the 3 cases above, then a least squares fit yields an intercept on the ordinate (grain size $d = 0\ \mu\text{m}$) of between 2.0 and 2.2 neighbours (Fig B29). It does however result in some real small grains being lost at the coarsest step size, particularly where this step size had already resulted in other similar sized grains remaining undetected in the first place.

It is worth noting that in the $2\ \mu\text{m}$ step size map, grains of approximately 16 pixels in size or approximately $9\ \mu\text{m}$ diameter are within 5% of the area measured at the finer steps sizes, despite the pixelation of the grain shape. Below this size, the difference rapidly increases, and above, the difference reduces significantly. Figure B30 shows the percentage difference in circle equivalent grain sizes calculated from maps with step sizes of $2\ \mu\text{m}$ and $1\ \mu\text{m}$ with the $0.5\ \mu\text{m}$ step size map, which clearly shows the rapid divergence as the grain size falls below a factor of 4 to 5 times greater than the calculated diameter.

B3.3 Grain boundary definition

In the discussion above, grain boundaries were taken to be defined by a misorientation of $>10^\circ$. It is however, also common for the minimum misorientation to be set at 15° ; if this is applied to the map datasets used above, then as might be expected, there is a slight reduction in the total number of grains found and a small increase in mean grain size. The effect becomes less pronounced as the step size decreases because the angular changes at boundaries are more accurately defined as the spatial resolution increases (e.g. at the $2\ \mu\text{m}$ step size, the measured point in one grain may be $1.9\ \mu\text{m}$ from its boundary with another grain in which the boundary-measurement point distance is only $0.1\ \mu\text{m}$).

	2 μm		1 μm		0.5 μm	
Boundary Definition	10°	15°	10°	15°	10°	15°
No. of grains	242	216	265	227	304	259
Mean Grain size (μm)	16.9	17.3	15.7	16.0	13.7	13.8

In the case above of the almost fully recrystallised Ni alloy, the use of the facility in EBSD software to extrapolate partially defined grain boundaries has little effect on the grain size. However, for the Al alloy shown earlier, with large heavily deformed grains, the effect is significant. Comparing the graph in Fig B21 with that of Fig B31 shows that, by extrapolating grain boundaries down to 5° , mean grain size can be reduced by 20%, and the trend in mean misorientation is altered significantly.

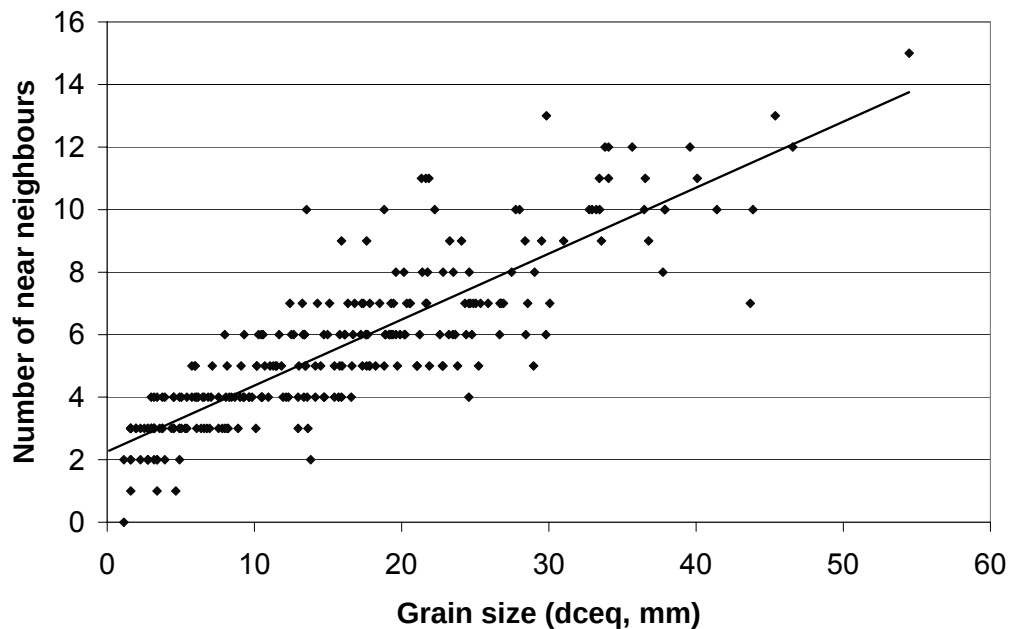


Figure B29 Graph of number of near neighbours plotted against grain size (circle equivalent diameter) for the EBSD map shown in Fig B23a.

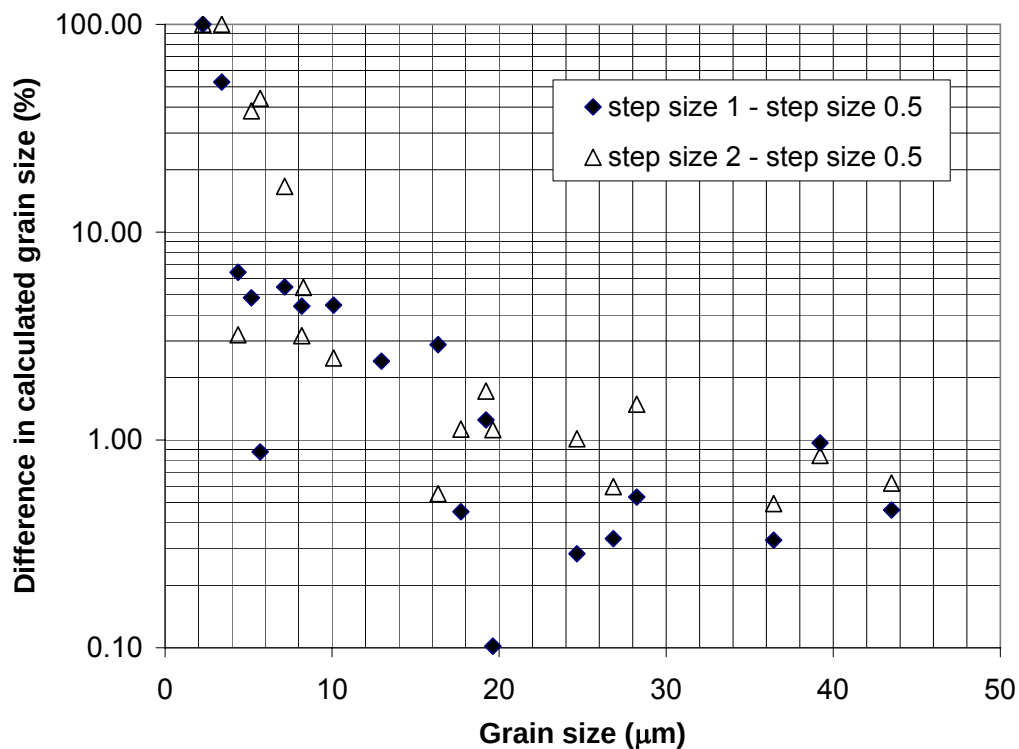


Figure B30 Graph plotting the difference in size of individual grains mapped in Fig B23, resulting from changing the map step size from 0.5 μm to 1 μm and from 0.5 μm to 2 μm

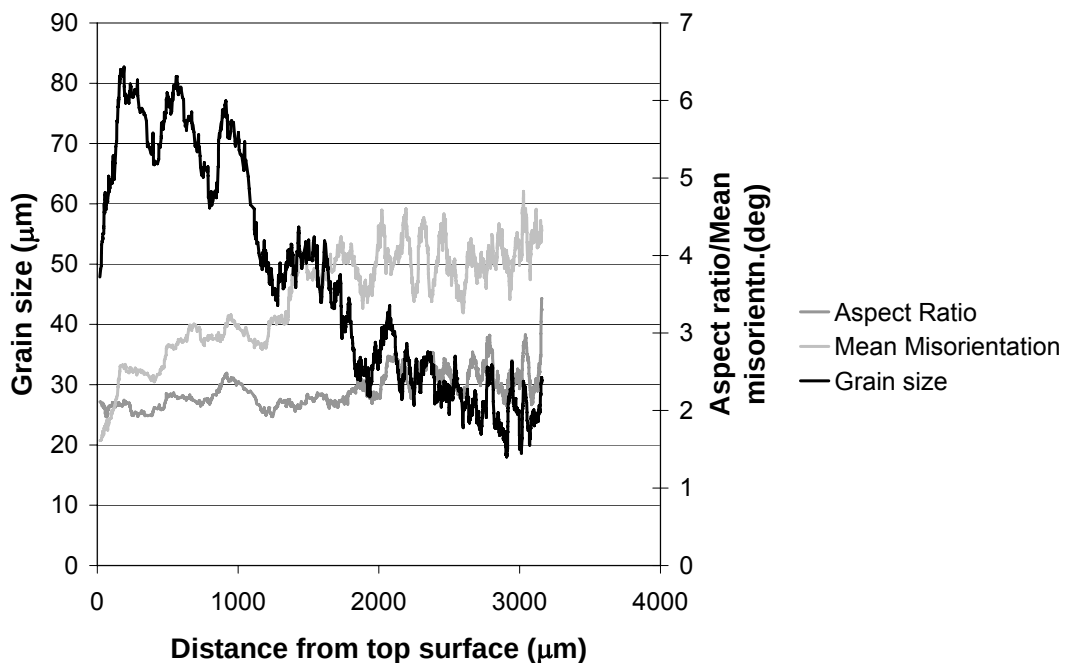


Figure B31 Graph plotting the same type of data in Fig B21, but with all grain data derived after extrapolating incompletely defined grain boundaries down to a 5° misorientation. Mean grain sizes are reduced by the extrapolation and mean misorientation is also significantly reduced.

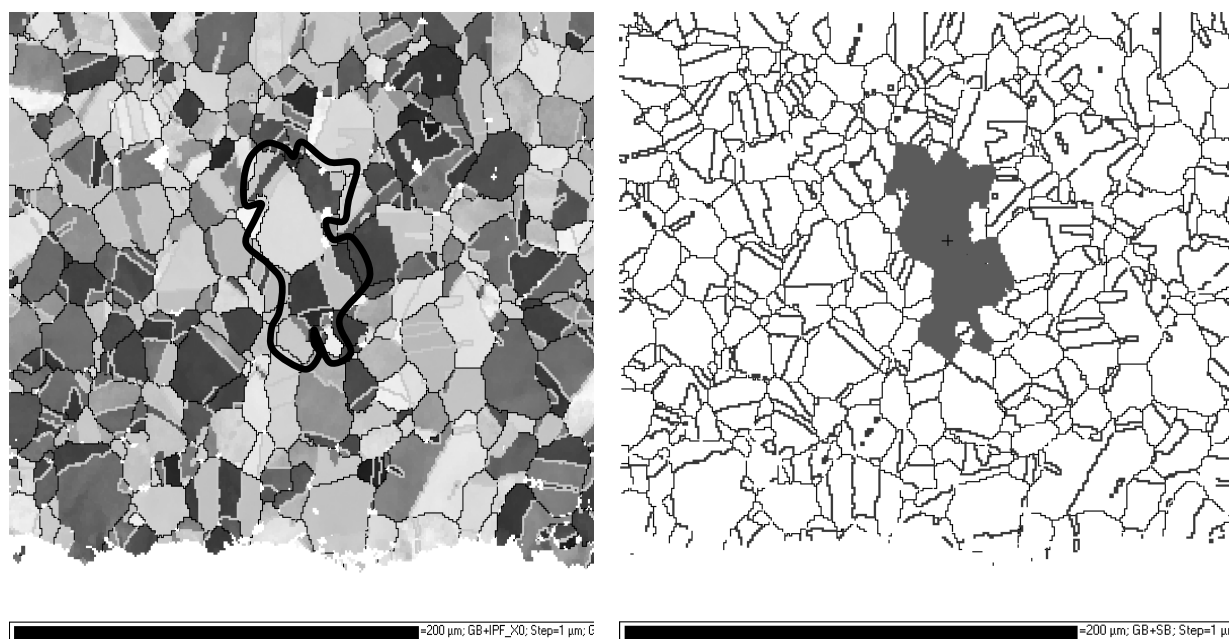


Figure B32a (left) and **B32b** (right)

a) EBSD orientation map (inverse pole figure colouring converted to arbitrary grey scale) of fully recrystallised Ni sample with region outlined showing a single grain as defined by software instructed to ignore grain boundaries of 60° about the $[111]$ direction, with a 5° tolerance. b) the same area with only grain boundaries shown.

B3.4 Twin definition

Any analysis of EBSD data must record in detail the parameters used to define boundaries between grains or twins. Different degrees of misorientation may be suitable for different materials or circumstances, but comparison of results should only be made if the same criteria are used. Results are unlikely to agree with conventional optical methods for measurement of grain size because of the enhanced resolution of EBSD and the more precise analysis of twin boundaries.

Twin boundaries are normally excluded in grain size measurement, and this is even more important with EBSD maps as the number of twins detected as step size decreases can be seen to increase sharply in Figs B26 and B27. The ability of EBSD to automatically detect which boundaries are twins also removes a potential source of uncertainty. However, care must be taken with the definition of a twin boundary, since allowance must be made for angular uncertainty in the EBSD measurement, i.e. no twin boundary in the Ni superalloy will be measured as having exactly 60° orientation change about the 111 axis. A common default twin definition is a boundary within 5° of a 60° misorientation about the 111 axis. Figure B32a shows grain boundaries and twin boundaries defined this way and Fig B32b shows how this can lead to unexpectedly large grains because the software groups regions together which share only a small fraction of a boundary that falls within the definition of a twin, even though the majority of the boundary is not a twin. Twin boundaries which do not have the classic banded appearance are also identified by EBSD where conventional optical/SEM examination might not classify them as twinned grains.

B3.5 Linear Intercept Measurements and Comparison with Light Microscopy Measurements

Conventional light microscopy measurements of microstructure are discussed in detail in appendix C but it is worth discussing here the differences in results that are likely to arise between optical measurements and those made by EBSD on the same or similar structures. However, in the preceding sections on EBSD circle equivalent diameter measurements were used almost exclusively, whereas for light microscopy the linear intercept measurement is the only simple technique available for deformed microstructures. Therefore, before comparing light microscopy with EBSD, an example will be given comparing circle equivalent diameter and linear intercept measurements, both by EBSD.

B3.5.1 Comparison of circle equivalent diameter and linear intercept measurement of grain size by EBSD

Circle equivalent diameter d_{ceq} and linear intercept measurements d_{lin} were made by EBSD on maps acquired at three step sizes of 0.5, 1.0 and 2.0 μm in a region close to the centre on the axis of an axi-symmetric compression sample of 316 stainless steel. The software determines the positions of the intercepts in the same way that it defines grain boundaries for the d_{ceq} measurements. Typically 350 values were obtained by linear intercept, compared with an average of 850 using the same dataset for area measurement. Results of both d_{lin} and d_{ceq} measurements for the three step sizes are shown in Table B5 and Figure B33 shows examples of the cumulative frequency curves for these measurements. The different step sizes produce some divergence in linear intercept data at small values, but the reduced number of these small values means the results for d_{lin} are less sensitive to step size and 95% confidence intervals are very slightly smaller at $\pm 7\%$. The ratio $d_{ceq}:d_{lin}$ is < 1 , in agreement with the findings Valcke et al [B14] when measuring sub-grain sizes in a geological specimen; they comment that calculations which show $d_{ceq}:d_{lin} \geq 1.22$ [B15] apply to particular grain shapes and to narrow grain size distributions. The linear intercept method is less sensitive to step size because only a fraction of the smallest grains are sampled. For example the percentage increase in arithmetic mean grain size d_{lin} between the 0.5 μm and 1 μm step maps is half that of the increase in arithmetic mean d_{ceq} for the same maps. Valcke et al also conclude that the linear intercept method is less sensitive to noise and non-indexing, when applied to sub-grain measurements in samples with many small sub grains and relatively few large grains without sub structure.

Table B5 EBSD comparison of linear intercept and circle equivalent grain sizes

	Linear intercept length, d_{lin} , μm			Circle equivalent diameter d_{ceq} μm		
	0.5	1.0	2.0	0.5	1.0	2.0
Map step size						
Arithmetic mean	7.8	8.1	8.7	7.2	7.8	8.4
Geometric mean	6.1	6.6	7.3	4.6	5.8	6.8
Median	6.9	7.1	7.6	5.9	6.6	7.0

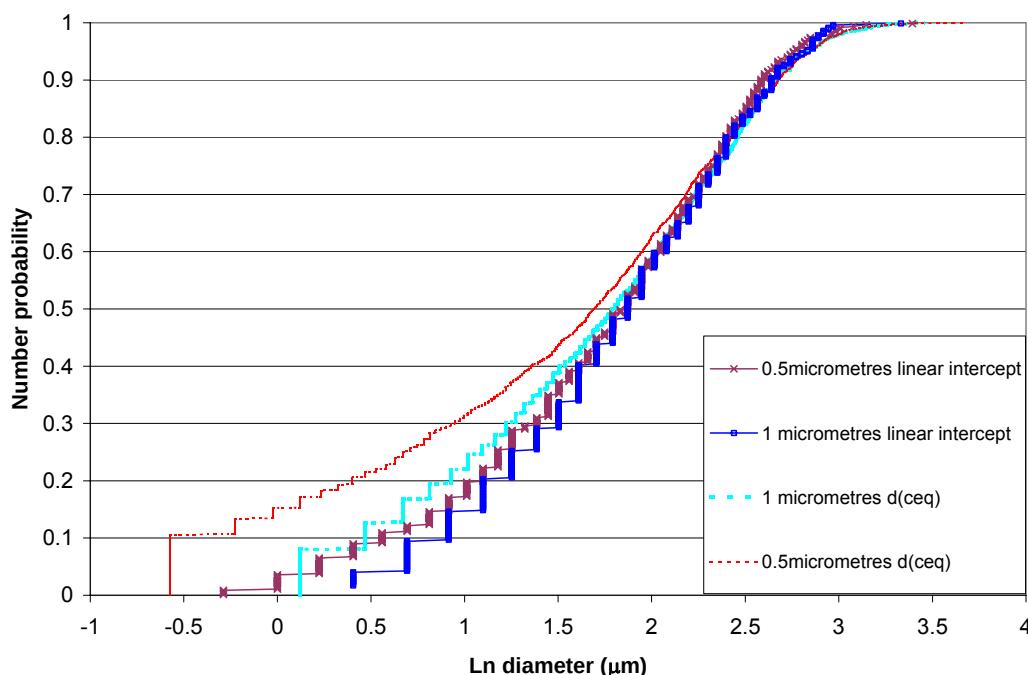


Figure B33 Cumulative number frequency plot of grain size distribution measured by linear intercept, d_{lin} , compared with circle equivalent diameter size d_{ceq} data for two step sizes of 0.5 μm and 1.0 μm

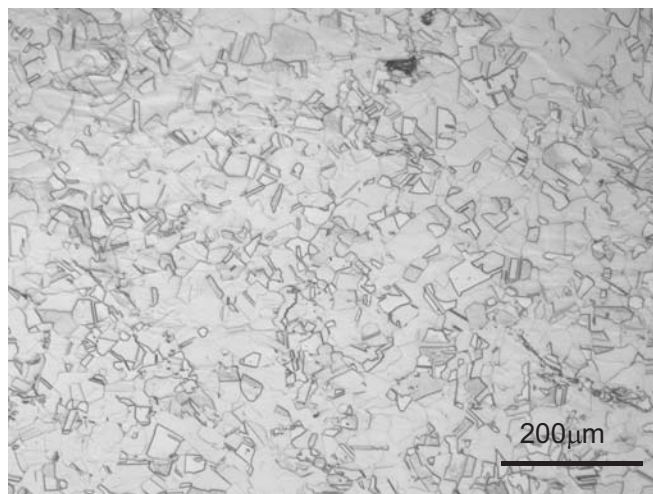
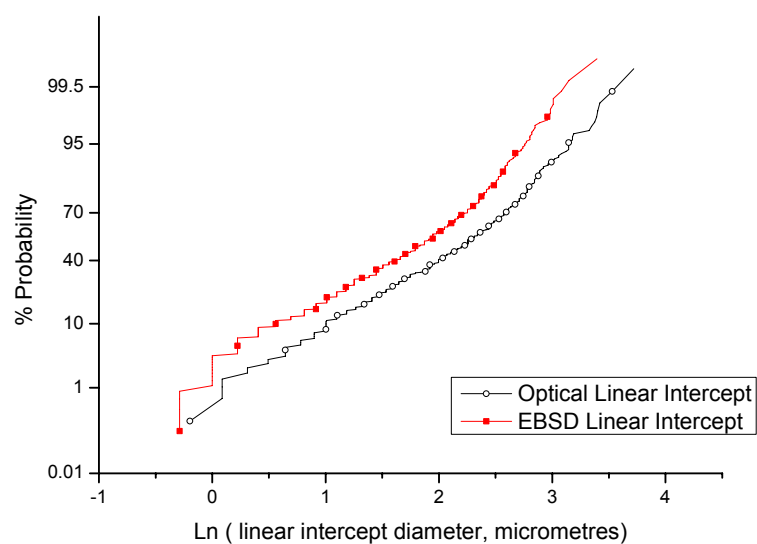
B3.5.2 Comparison of EBSD and Optical Measurements.

Linear intercept measurements were made by light optical microscope in the same region of the 316L sample described in the previous section, after etching. They were also compared with measurements by Swansea University [B16] on a separate but identically processed sample. Digital images of the etched samples (Figure B34) were acquired with a 20 $\times$ objective lens and two operators then measured intercepts on sets of lines 25 μm apart, giving approximately 250 measurements. Twin boundaries were ignored. Operator variability is clearly seen in the results shown in Table B6 but with 95% confidence intervals of $> \pm 10\%$ they are approximately equivalent. However there is a 25% increase over the EBSD measurements in the same orientation. Possible reasons for this include failure of the etch to reveal all grain boundaries, and a failure to resolve all the small grains identified by EBSD. These effects would both tend to cause overestimates of grain size by the optical method, although they would to some extent be counteracted by the mistaken inclusion of some twin boundaries.

Figure B35 is a probability plot showing that, relative to the EBSD results, a) the light optical microscope linear intercept data follows a very similar distribution but offset by the difference in mean values, and b) that the linear intercept data show a slightly closer correspondence to a lognormal distribution, but there is still a deviation away from a straight line at larger values.

Table B6 Optical measurements of linear intercept grain size

	Optical Linear intercept length, d_{lin} , μm		
Operator	1	2	Swansea
Arithmetic mean	12.7	11.8	13.8
Geometric mean	10.0	9.3	
Median	10.9	10.5	

**Figure B34** Optical micrograph of stainless steel sample in same region as that used for EBSD maps, and from which optical linear intercept measurements taken.**Figure B35** Probability plot of d_{lin} grain size distribution measured by linear intercept (optical and EBSD compared).

For a final comparison, Figure B36 compares the EBSD measurements on the ASC Waspaloy sample discussed in B3.1.3 with those obtained from two samples processed identically at the same time and assessed in a similar section by EBSD (at Sheffield University) with a linear intercept measurement on a 4 μm step map, and by optical linear intercept (at Swansea University). Despite the coarse 4 μm step size the correspondence of the average grain size by EBSD in the centre of the sample is very good. The coarser step size does not increase grain size towards the surface as much as the finer step size, reflecting partly the sample variation but mostly a poor level of indexing in the region closest to the surface. The light microscope technique measured only a single line and thus fewer grain boundaries so that the moving average is a smoother line. The difference of approximately 50% in grain size between light microscope and EBSD measurements shown in Table B5 is repeated in Figure B36 if the average grain size over a 1mm distance is considered furthest from the surface.

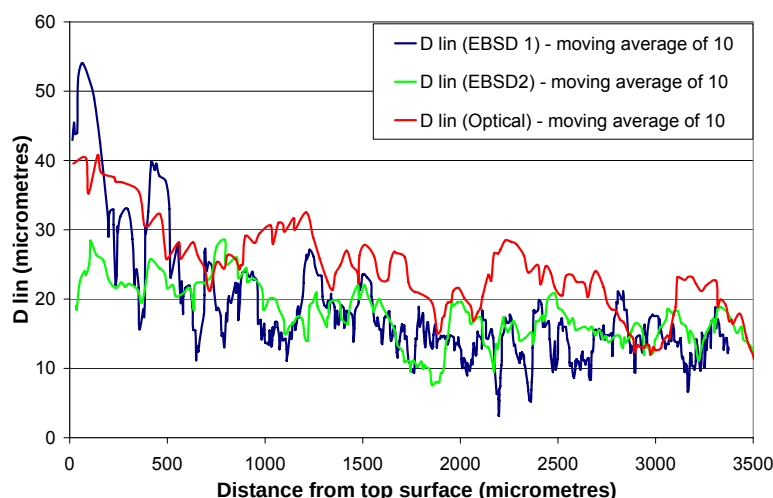


Figure B36 Comparison of linear intercept measurements on three equivalent samples.

B4 EBSD Acknowledgements

Much of the text in sections B1 and B2 is repeated from text provided by NPL (Drs P Quested and K P Mingard) and Aunt Daisy Scientific Ltd (Dr A Day) for the ISO standard on EBSD currently in development (ISO/CD 24173). Professor Val Randle is also acknowledged for promoting the science base in EBSD characterisation through the recent UK EPSRC network on EBSD. Many of the case study issues followed on from topics discussed at the network and parallel Royal Microscopical Society (RMS) meetings.

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Appendix C

15

IN THIS CHAPTER

- Optical Metallography – Case Studies
- Comparison with FE Model predictions

APPENDIX C Optical Metallography

C1 Case Studies – AC testpieces

Optical metallography was conducted at the IRC in Computer Aided Engineering (University of Wales, Swansea) on AC and PSC testpieces that had been deformed in the matrix of tests described previously (Section 9). Full details of the studies can be obtained if required from NPL or the IRC at the University of Wales, Swansea. This appendix contains summaries of key measurements from these systematic investigations.

The test matrices shown in Tables C1-C4 were conducted to generate samples for study.

Table C1 Axisymmetric compression testing conditions for Al 5052

Specimen Number	Temperature °C	Strain Rate s ⁻¹	Friction Condition	Strain
A1	400	0.01	lub (m=.13)	0.5
A2	400	0.01	dry (m=.39)	0.5
A3	400	1	lub (m=.15)	0.5
A4	400	1	dry (m=.31)	0.5

Table C2 Axisymmetric compression testing conditions for 316 stainless steel

Specimen Number	Temperature °C	Strain Rate s ⁻¹	Friction Condition	Strain
S1	950	0.01	lub (m=.13)	0.5
S2	950	0.01	dry (m=.36)	0.5
S3	950	1	lub (m=.11)	0.5
S4	950	1	dry (m=.36)	0.5

Table C3 Axisymmetric compression testing conditions for Waspaloy

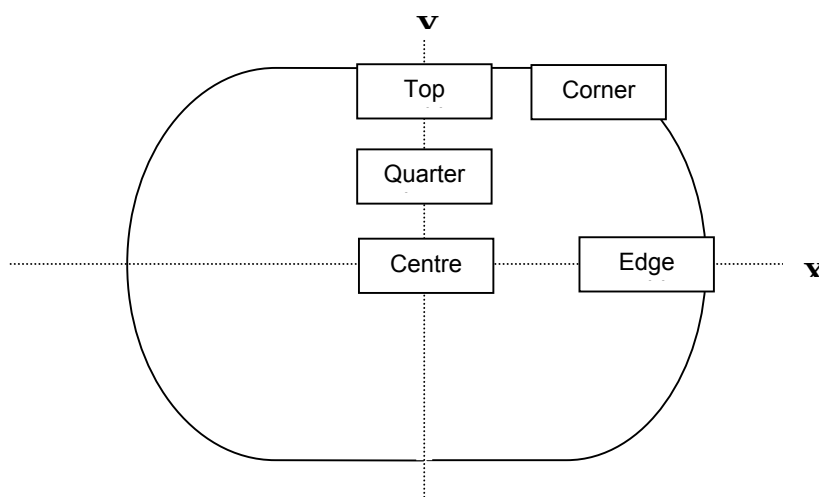
Specimen Number	Temperature °C	Strain Rate s ⁻¹	Friction Condition	Strain
W1	1040	0.01	lub (m=.13)	0.5
W2	1040	0.01	dry (m=.37)	0.5
W3	1040	1	lub (m=.11)	0.5
W4	1040	1	dry (m=.38)	0.5

Table C4 Axisymmetric compression testing conditions for Ti6Al4V

Specimen Number	Temperature °C	Strain Rate s ⁻¹	Friction Condition	Strain
T1	950	0.01	lub (m = .20)	0.5
T2	950	0.01	dry (m = .37)	0.5
T3	950	1	lub (m = .19)	0.5
T4	950	1	dry (m = .38)	0.5

C1.1 Al 5052

Axisymmetric compression specimens had an initial height of 12 mm and an initial diameter of 9 mm. They were cut from a rolled strip of Al 5052 with the specimen axes along the thickness direction. They were heated to testing temperature within 5 minutes and water quenched within 3 seconds at the end of the compression test. Tests were carried at true constant strain rate. High friction conditions were attained by using dry specimens and low friction by lubricating with PTFE. Following deformation, specimens were sectioned across a diameter and by a plane parallel to the cylinder axis. The specimens were then prepared for optical examination. In order to investigate the initial microstructure, specimens were cut from the rolled strip in directions perpendicular to the rolling direction, cross direction and thickness direction. These specimens were also prepared for optical metallography. A series of images were taken, so that a montage of the whole specimen could be constructed. From this, detailed measurements of grain size and shape were made in five distinct areas as shown in Fig C1. The height of the sampling area was approximately 1/7 of the total height of the specimen, and the width was 1.35 times the height. This equates to an area of 1600×1200 pixels, where 975 pixels=1mm.

**Figure C1.** Sampling areas for the Al 5052 compression specimens.

In each area, every grain that could be distinguished was measured individually. For each grain, the maximum tangent diameter in the x and y direction was noted. Grains that were not complete within the marked area were not measured. This method resulted in a different number of grains being measured in each sampling area, but ensured that any differences in the structure in the different regions of the specimen would be distinguished. All

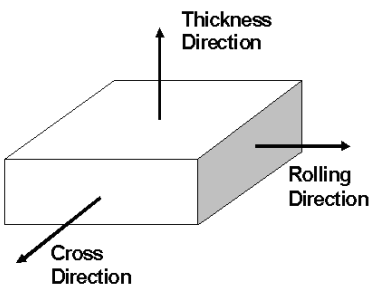
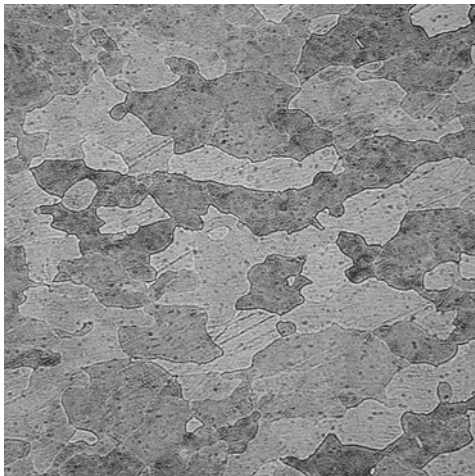
measurements were made directly from the digital image on the computer. Calculations were made of the mean values, standard deviations and 95% confidence intervals for the tangent diameters. In all cases, sufficient observations were made to ensure that the confidence intervals were less than 10% of the means.

Tested samples were prepared through graded emery papers followed by polishing using coarse and fine diamond wheels. A further final polish was then employed, using a commercially available SiO₂ suspension in water. Etching was carried out electrolytically using Barker's reagent: 5 g HBF₄; 200 ml distilled water. Etching was for 3.5 min at 20V. This etch deposited an anodised layer on the surface. The specimens were examined and photographed under polarised light. Figures C2 and C3 show the grain structure for as received material as well as histograms depicting the grain distributions. The grains show elongation in the rolling direction as well as some evidence of sub-grains. The distributions of grain diameters in all figures and in each of the two appropriate directions was skewed with a considerable tail to the right. The overall results for each section are given in Table C5.

Table C5 Grain sizes for as received Al 5052

Section Normal Direction	Measurement Direction	Mean diameter µm	Standard deviation µm	Sample Size	95% Confidence Interval (µm)
Rolling Rolling	Thickness	86.2	49.1	311	5.5
	Cross	136.1	87.1	311	9.7
Cross Cross	Rolling	164.9	103.9	236	13.3
	Thickness	82.7	46.2	236	5.9
Thickness Thickness	Rolling	67.4	45.5	359	4.7
	Cross	67.5	47.4	359	4.9

A selected number of the detailed grain size measurements obtained from all fields are shown in Figs C4 to C7. In each case the micrograph shows the area and structure on which the measurements were made. The two histograms illustrate the distribution of the tangent grain diameters in the directions of the coordinate axes. The values of average tangent grain diameters and confidence intervals are shown in Table C6. For specimens A1 and A3 (low friction), the grain diameters in the y direction are reasonably constant but less than that for the as received material (Table C5). For specimens A2 and A4 (high friction) the grain diameters decrease from the top edge to the centre of the specimens. In all cases the distributions of grain diameters are skewed at the right.



Magnification x 50.

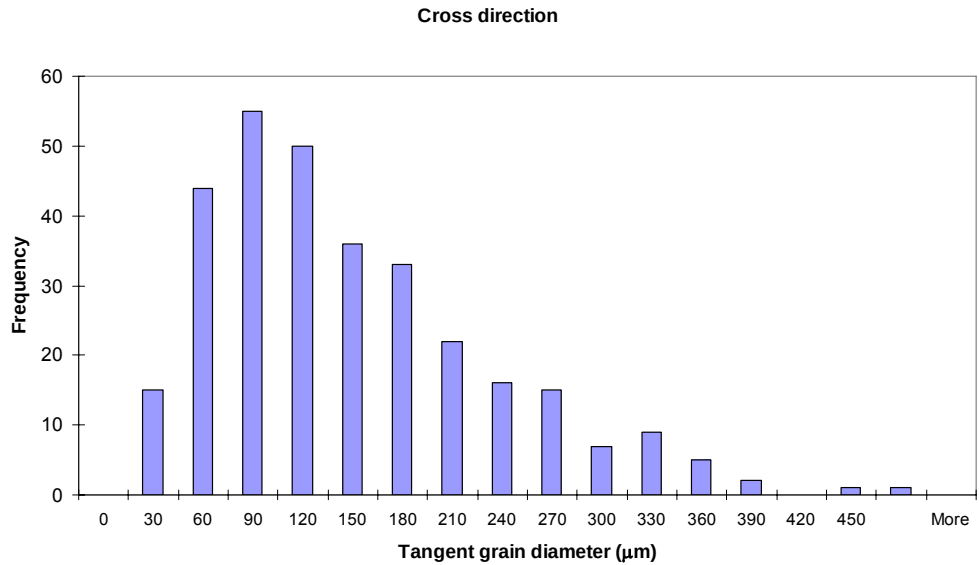
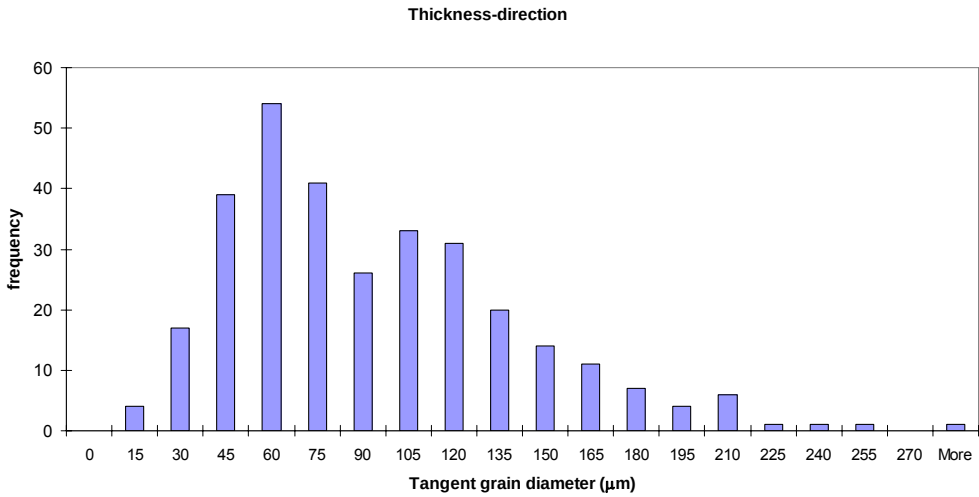
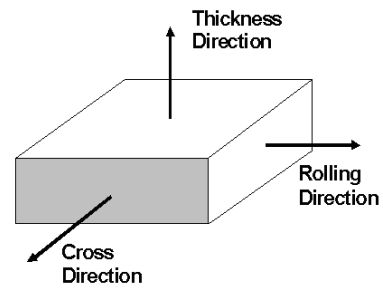
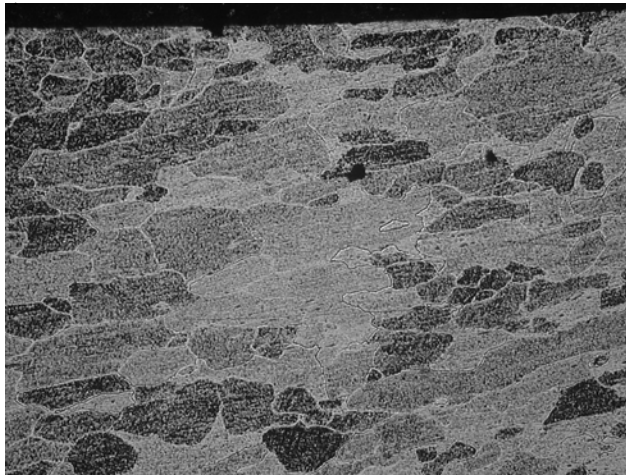


Figure C2. As-received material, rolling direction.



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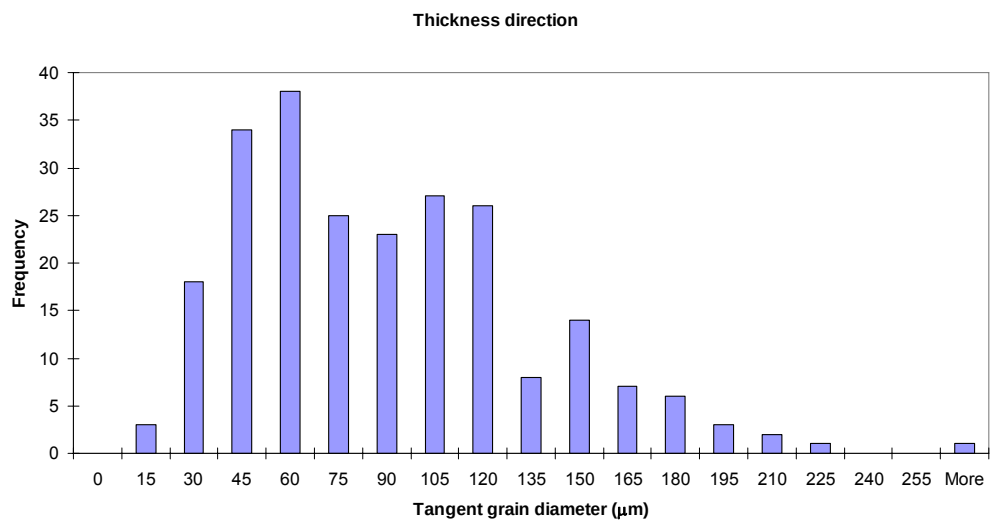
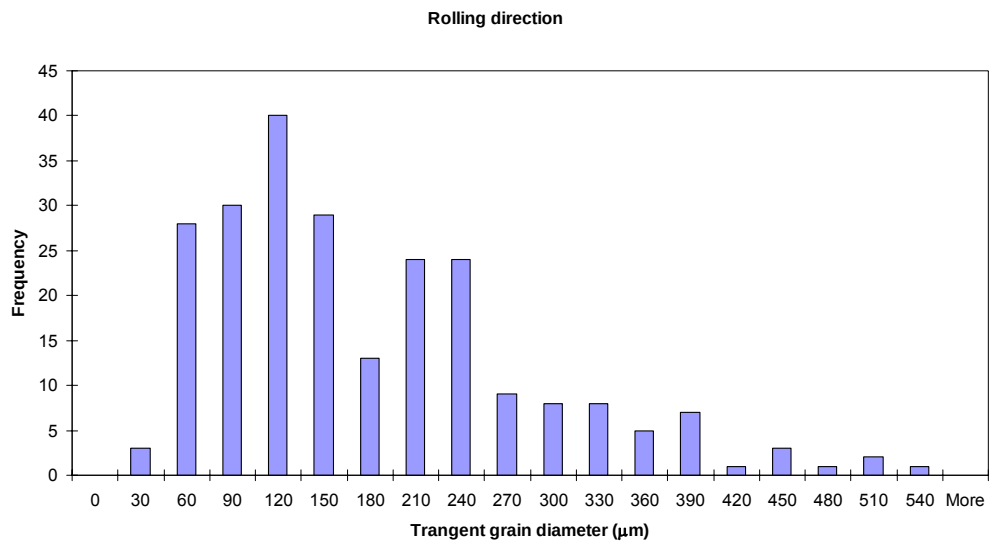
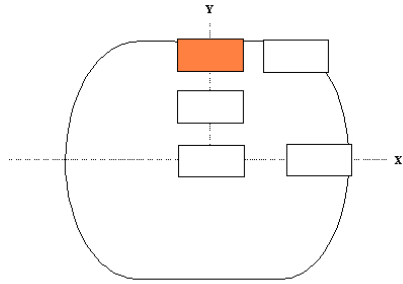
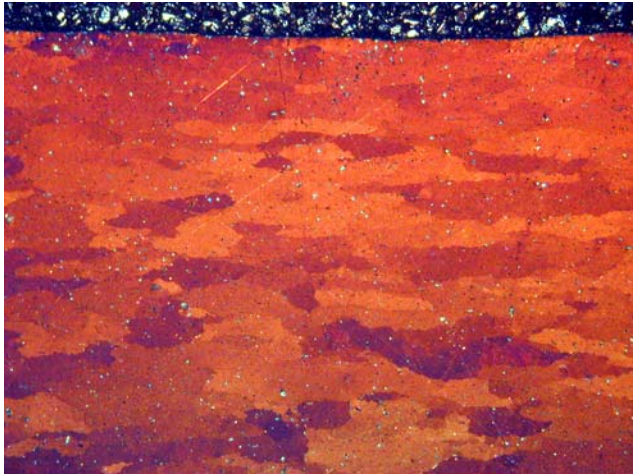


Figure C3. As-received material, cross direction.



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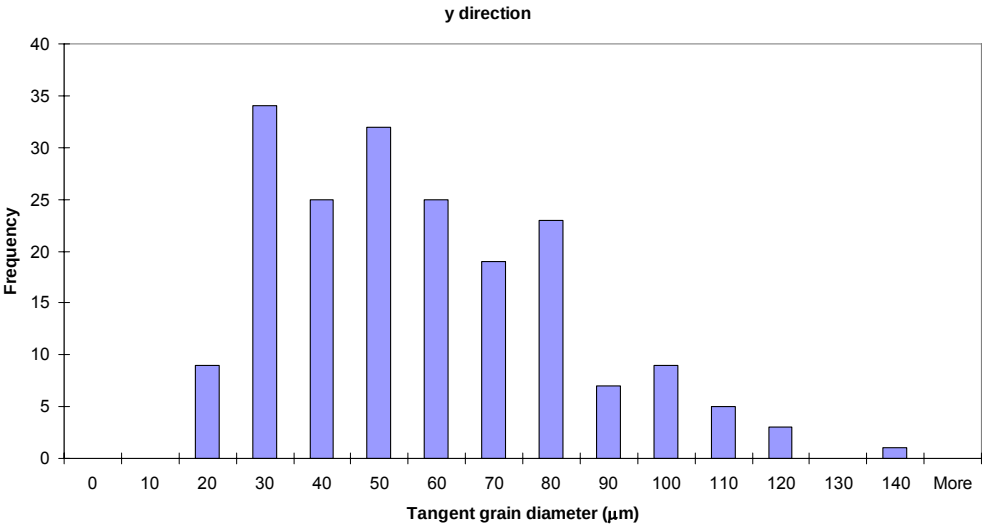
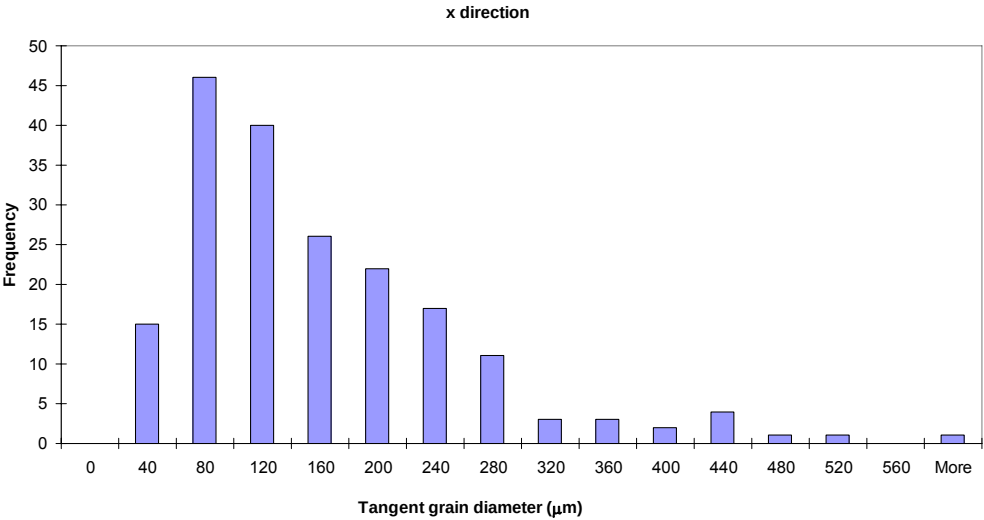
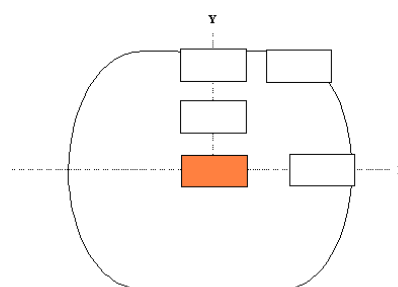
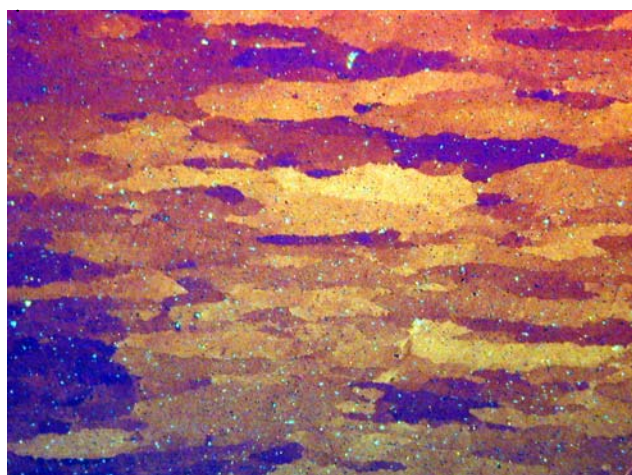


Figure C4 Specimen A1, top-mid.



Magnification x 50.

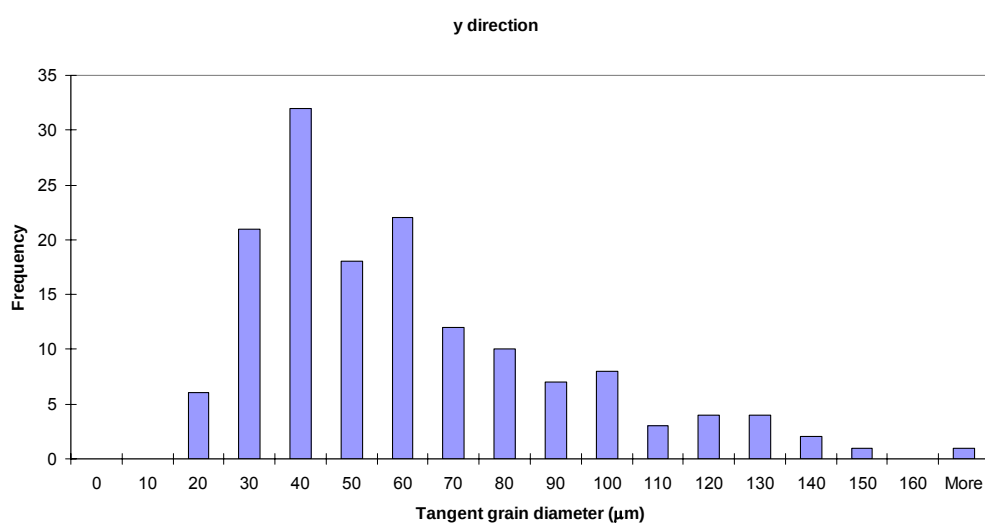
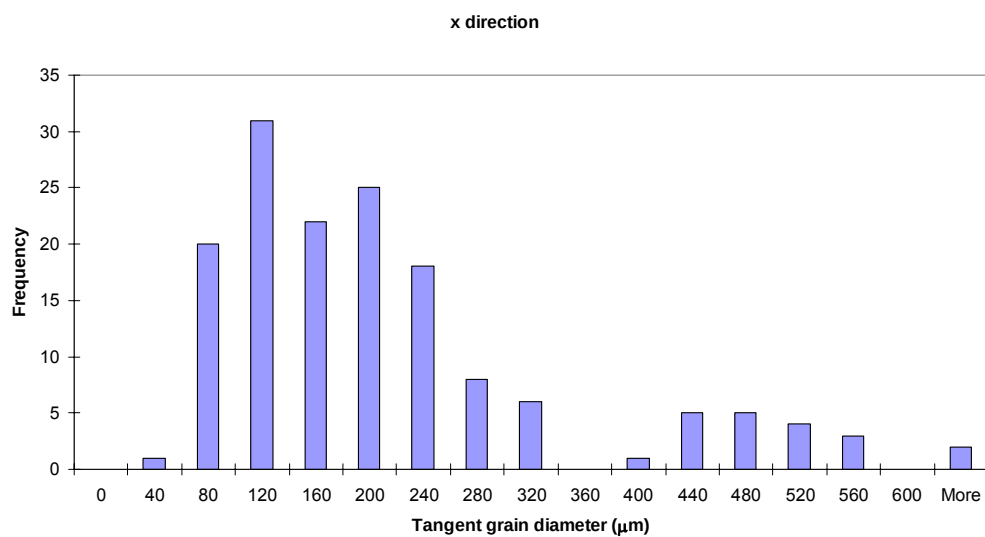
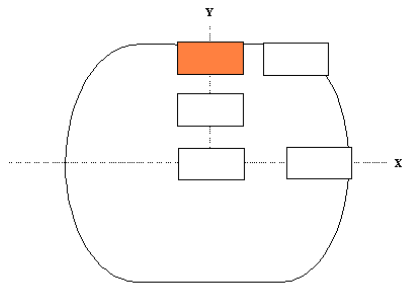
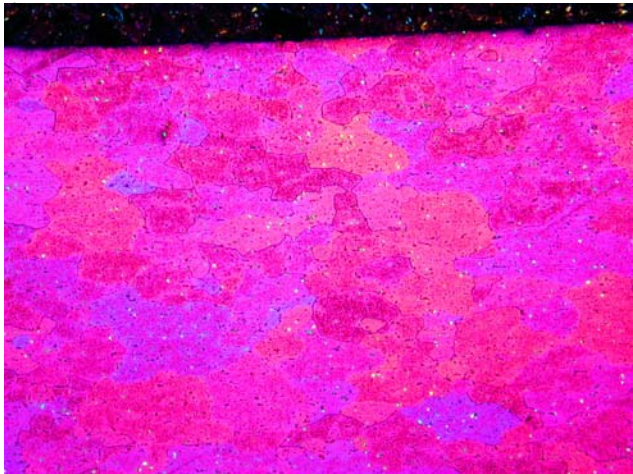


Figure C5 Specimen A1, centre.



Magnification x 50.

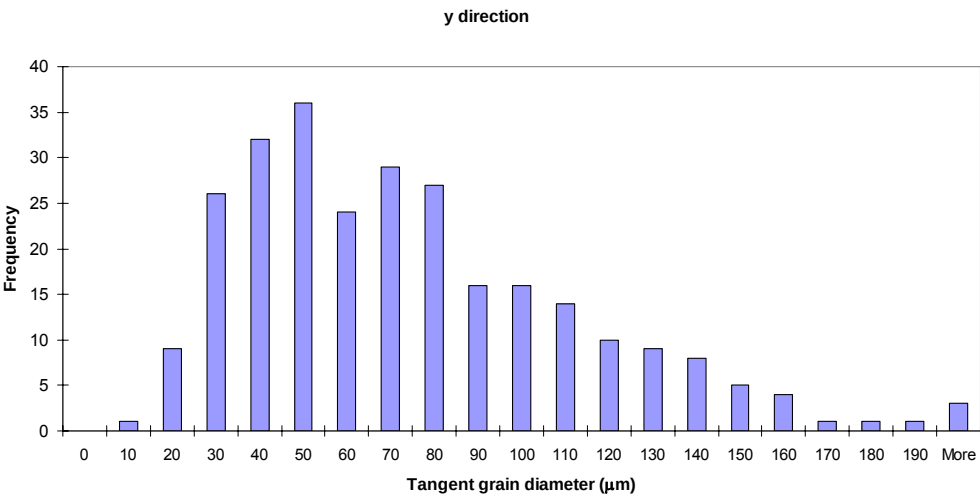
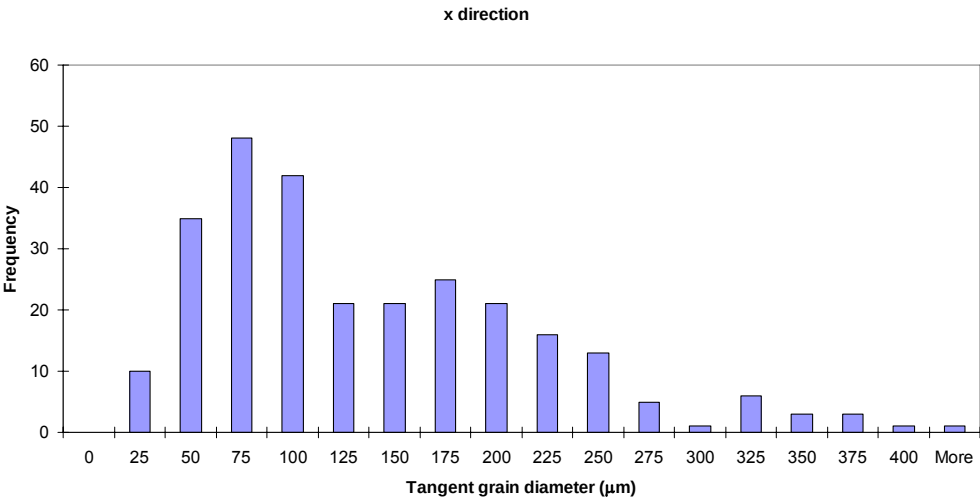
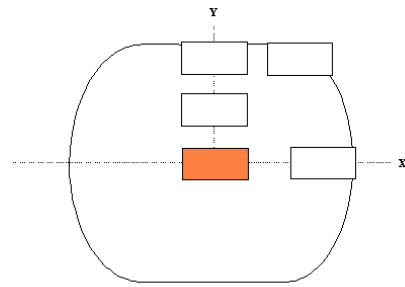
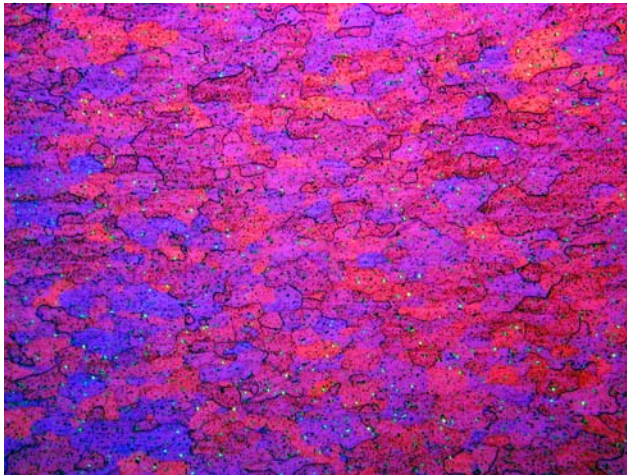


Figure C6 Specimen A4, top-mid.



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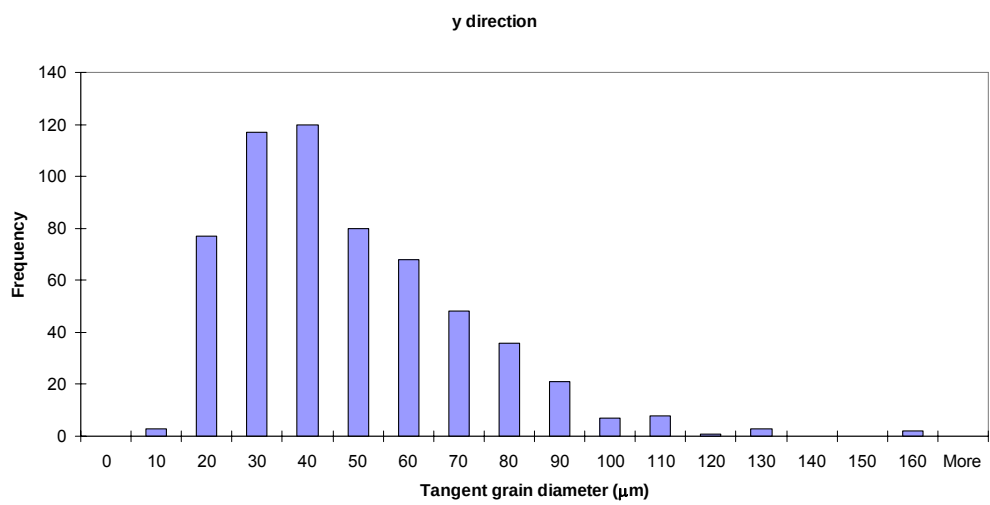
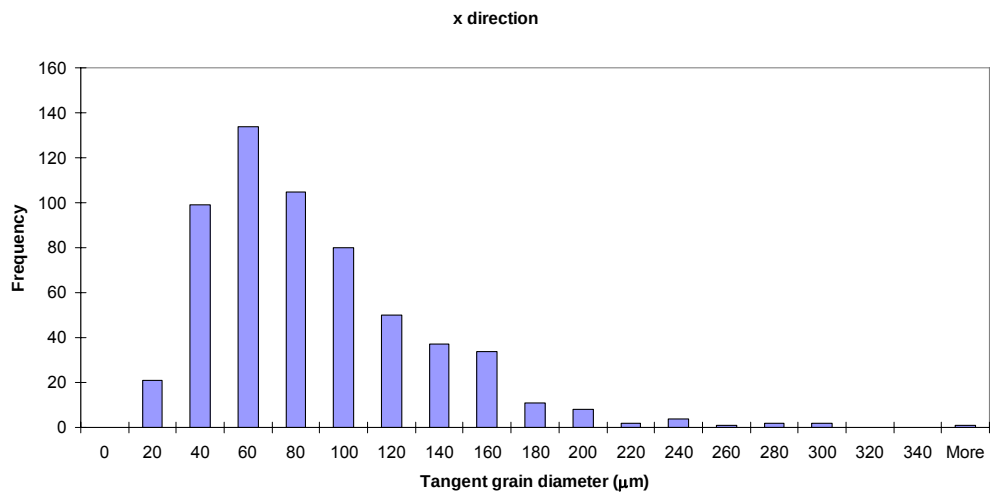


Figure C7 Specimen A4, centre.

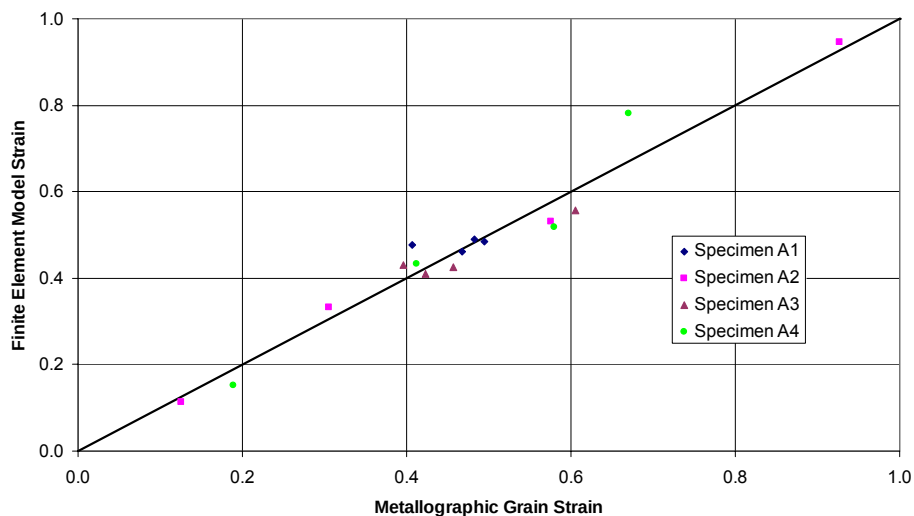
Table C6 Tangent grain diameters for the Al 5052 deformed specimens.

Specimen Number	Sample Position	Sample Size	x-direction			y-direction		
			mean μm	std. dev. μm	95% limit μm	mean μm	std. dev. μm	95% limit μm
A1	Corner	192	143.4	98.5	13.8	52.9	24.6	3.4
A1	Top-Mid	192	143.4	98.5	13.8	52.9	24.6	3.4
A1	Quarter	171	194.4	127.8	19.0	51.5	28.4	4.2
A1	Centre	151	196.9	136.2	21.5	56.2	30.4	4.8
A1	Edge-Mid	139	188.5	127.6	21.0	52.1	30.1	5.0
A2	Corner	220	130.2	118.1	15.5	54.0	41.7	6.0
A2	Top-Mid	184	124.5	83.9	12.1	74.5	44.4	8.1
A2	Quarter	196	175.7	128.4	18.0	62.2	31.1	9.5
A2	Centre	315	152.1	132.6	14.6	33.4	20.7	8.3
A2	Edge-Mid	233	145.9	101.8	13.1	47.5	25.6	4.1
A3	Corner	295	113.1	75.2	8.6	52.2	30.0	3.4
A3	Top-Mid	255	125.8	85.4	10.5	55.3	30.0	3.7
A3	Quarter	305	148.2	100.3	11.3	56.8	29.6	3.3
A3	Centre	253	143.7	105.2	13.0	53.5	24.6	3.0
A3	Edge-Mid	234	161.2	111.7	14.3	46.1	22.9	2.9
A4	Corner	398	99.2	72.7	7.1	62.5	37.4	3.7
A4	Top-Mid	272	126.6	82.3	9.8	69.9	38.3	4.6
A4	Quarter	349	109.4	74.1	7.8	47.3	23.5	3.0
A4	Centre	591	78.4	48.0	3.9	43.4	23.2	1.9
A4	Edge-Mid	262	137.3	92.8	11.2	55.9	31.4	3.8

Table C7 Calculated metallographic grain strain and finite element model strain.

Specimen	Sample Position	Grain Strain	95% Confidence	Model Strain
A1	top-mid	0.47	0.053	0.46
A1	quarter	0.50	0.058	0.48
A1	centre	0.41	0.058	0.48
A1	edge	0.48	0.053	0.49
A2	top-mid	0.13	0.059	0.11
A2	quarter	0.31	0.054	0.33
A2	centre	0.93	0.056	0.95
A2	edge	0.58	0.055	0.53
A3	top-mid	0.42	0.054	0.41
A3	quarter	0.40	0.052	0.43
A3	centre	0.46	0.050	0.42
A3	edge	0.61	0.053	0.56
A4	top-mid	0.19	0.054	0.15
A4	quarter	0.58	0.049	0.52
A4	centre	0.67	0.045	0.78
A4	edge	0.41	0.055	0.43

The agreement between the FE calculation (Section 9) and the optical measure of strain is good. This is illustrated in Fig C8. The 95% confidence intervals for the grain strain are quite large so that the model predictions fall within these intervals. These uncertainties may be a difficulty with metallographic measurements and they will not be much improved with larger samples except at great effort. However, the reasonable correlation suggests that the assumption of no change in grain shape is a reasonable one for this alloy.

**Figure C8** Relationship between metallographic grain strain and predicted model strain. Correlation coefficient 0.974.

C1.1.1 Conclusions – Axisymmetric Compression Al 5052

1. Quantitative metallographic measurements of tangent grain diameter were made for hot deformed Al 5052. The statistics of the data suggests that around 200 observations are required to give 95% confidence intervals less than 10% of the mean.
2. Grain diameters show a systematic variation with testing conditions and position within the deformed solid. This suggests that the values of diameter may quantitatively reflect changes occurring during hot working.
3. Comparisons between strains calculated from the metallographic measurements and those calculated from Finite Element models suggest that, for this alloy, the correlations are good.

C1.2 316 Stainless Steel

Following deformation, specimens were sectioned across a diameter and by a plane parallel to the cylinder axis (Fig C9). All were prepared through graded emery papers followed by polishing using coarse and fine diamond wheels. Etching was carried out by swabbing the sample for 2 min using a Glyceregia etch: 2 parts hydrochloric acid; 2 parts glycerol; 1 part nitric acid. Before etching, the samples were warmed by immersion in hot water for at least 5 min. The specimens were examined under normal reflected light. A series of images were taken, so that a montage of the whole specimen could be constructed. From this, detailed measurements of grain size were made in four distinct areas as shown below. The sampling area was a square having sides 1150 pixels long, where 975 pixels represented 0.5mm on the sample.

Grain size measurements were made using the linear intercept method. Within the sampling area, 11 guide lines were constructed in both the horizontal and vertical directions (i.e. in the specimen radial direction and specimen axial direction respectively). Each line was 1150 pixels long. The number of grain boundaries intercepting each line was counted. Twin boundaries were not counted. All measurements were made directly from the digital image on the computer. Calculations of average linear intercept grain diameters in both directions and grain aspect ratio (radial diameter/axial diameter) were made together with variances and 95% confidence intervals.

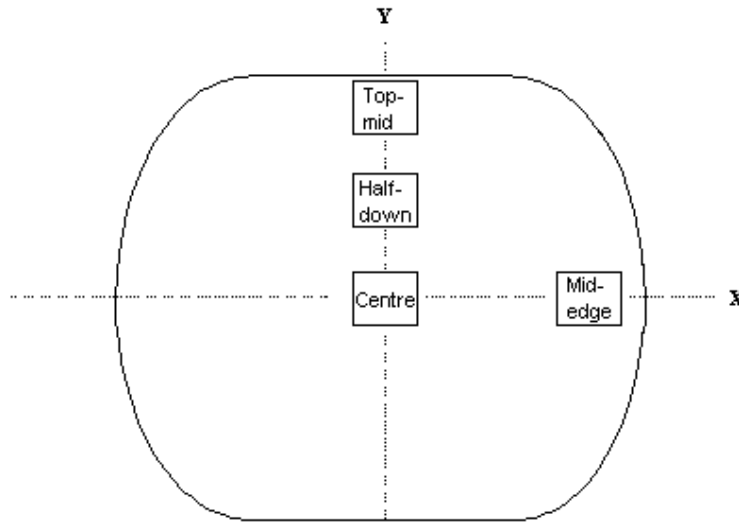
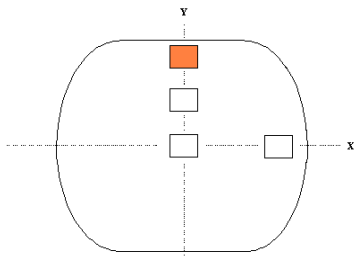
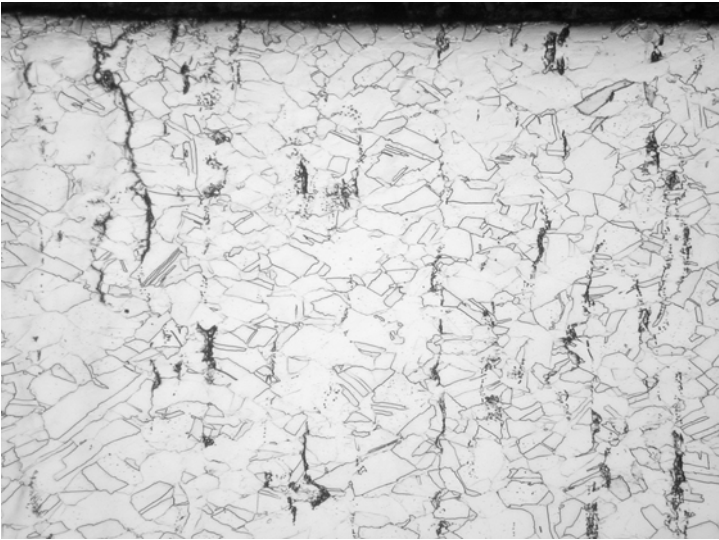


Figure C9 Schematic diagram showing areas of the 316 stainless steel sample which were measured. Not to scale.

The grain structures of the deformed samples were complex with different areas of the sections showing structures varying from un-deformed and un-recrystallised grains through recrystallised grains which had not subsequently deformed to grains which had both recrystallised and subsequently deformed. All specimens showed small regions of ferrite and the volume fraction of ferrite was larger near the surfaces of the specimens where oxidation during testing had altered the matrix oxygen concentrations.

The measured radial grain diameters, axial grain diameters and aspect ratio of all specimens and all sampling areas are shown in Table C8. The microstructures of sampled areas together with distributions of grain sizes in radial and axial directions are shown in Figs C10 to C13. All specimens showed a region of small deformation immediately under the platens but the extent of this zone increased with increasing strain rate and friction.



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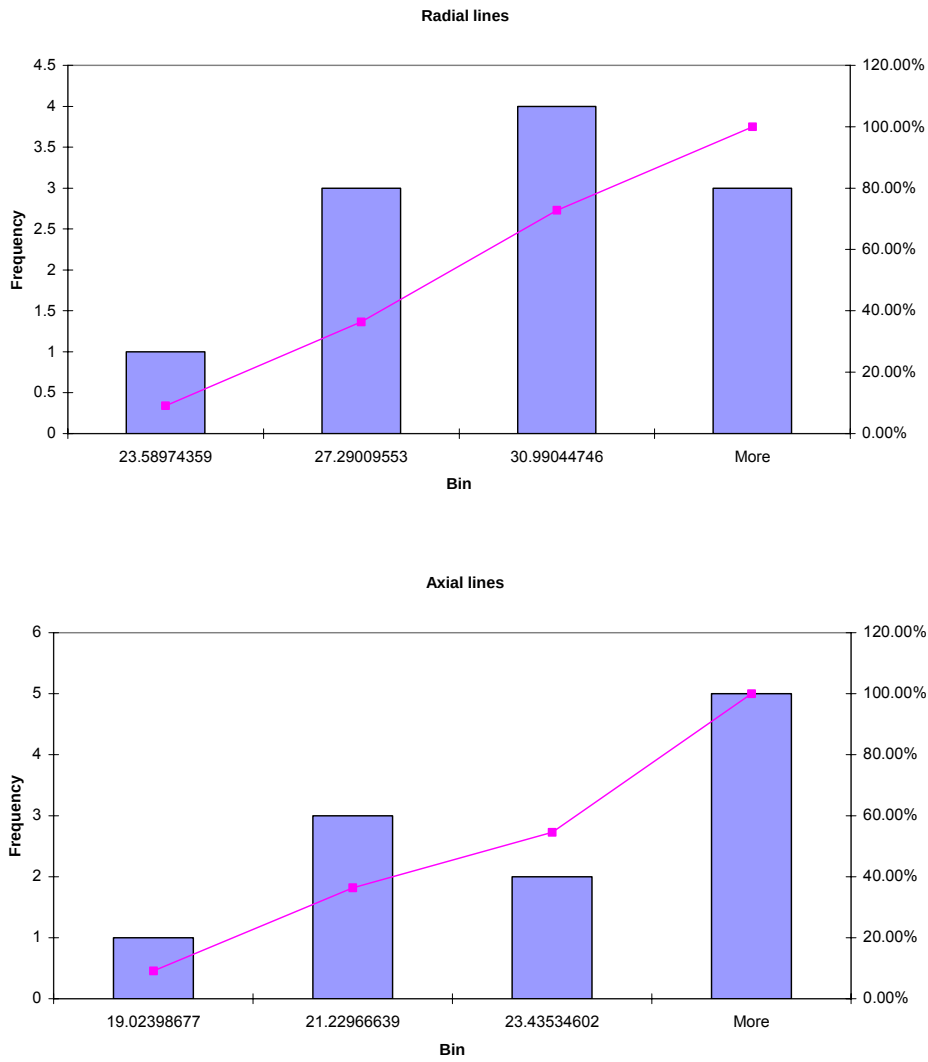
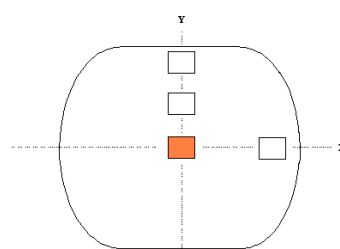


Figure C10 Microstructure and grain diameter distributions for specimen S1 and sampling area Top-mid. Radial direction horizontal.



Magnification x 116

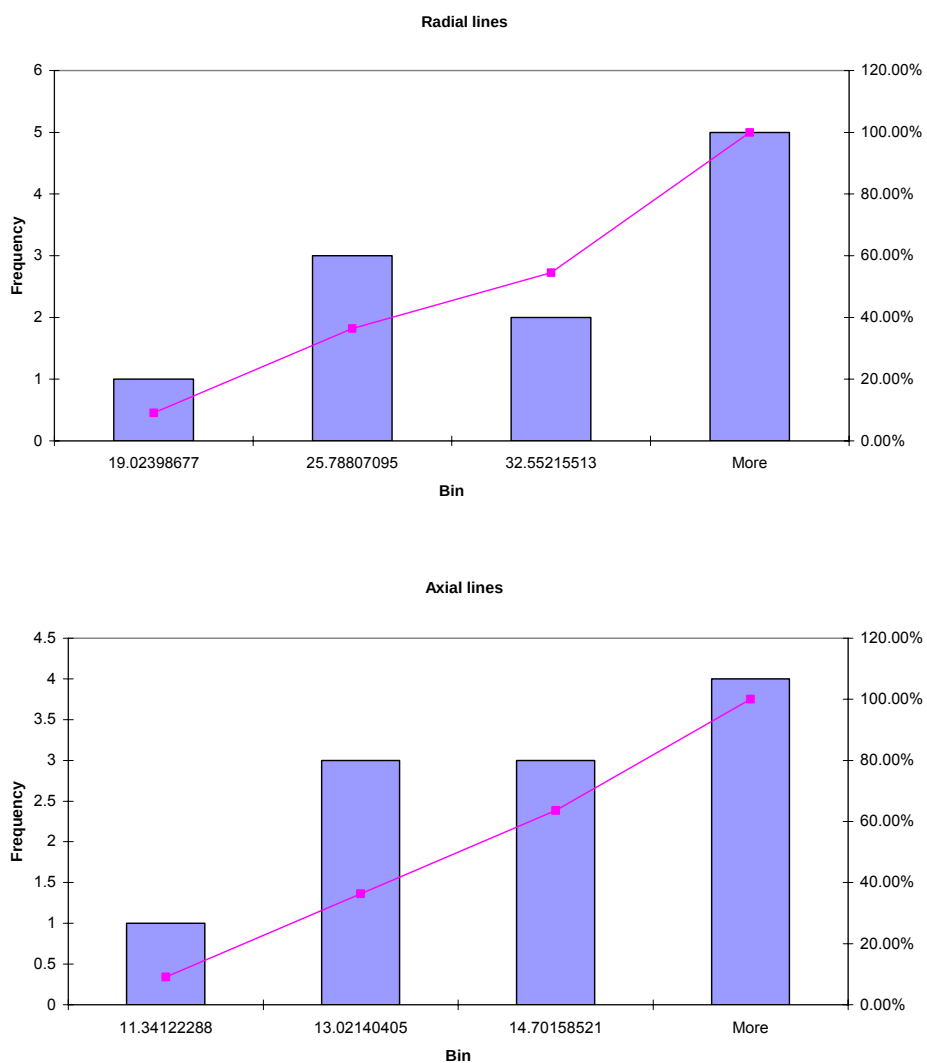


Figure C11 Microstructure and grain diameter distributions for specimen S1 and sampling area Centre. Radial direction horizontal.

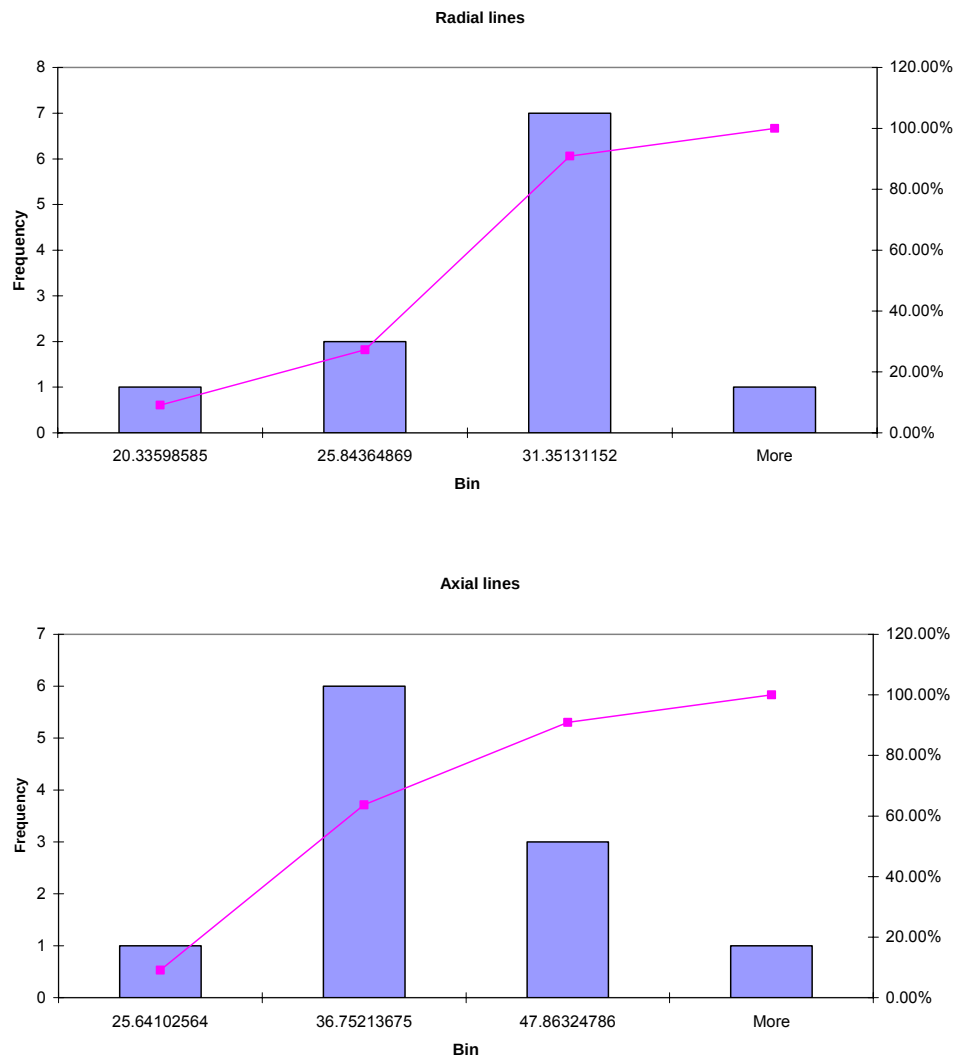
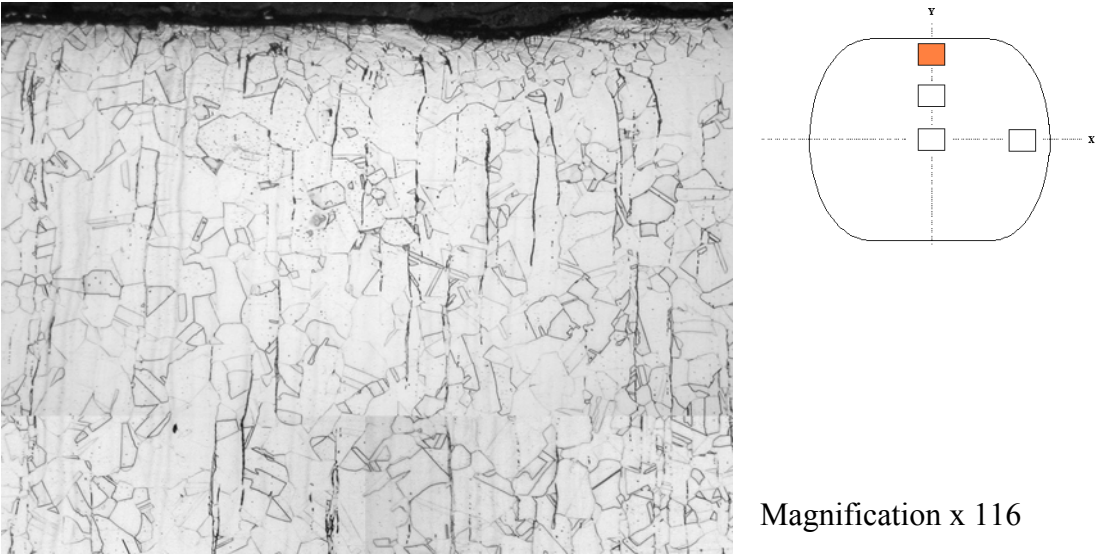
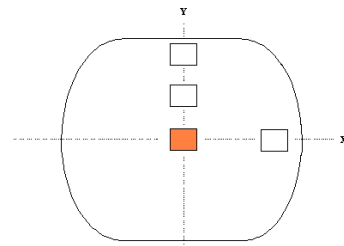


Figure C12 Microstructure and grain diameter distributions for specimen S4 and sampling area Top-mid. Radial direction horizontal.



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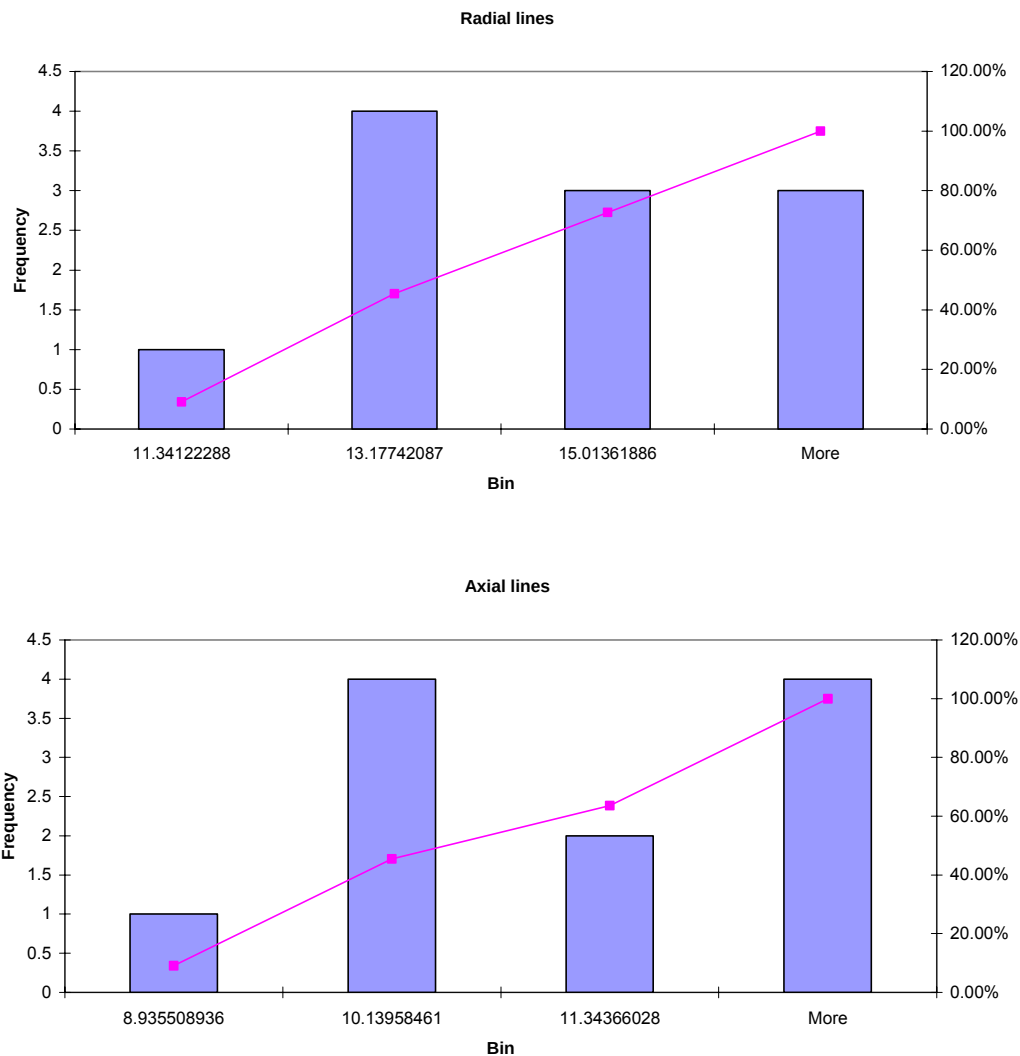


Figure C13 Microstructure and grain diameter distributions for specimen S4 and sampling area Centre. Radial direction horizontal.

Table C8 Measured diameters and statistical analysis for 316 stainless steel testpieces

Specimen Number	Sample Area	Sample Size 590 µm line	Radial Linear Intercept Diameter µm			Axial Linear Intercept Diameter µm			Aspect Ratio		
			Average	Std. dev.	95% conf. int.	Average	Std. dev.	95% conf. int.	Average	Std. dev.	95% conf. int.
S1	Top-mid	11	28.6	2.5	1.7	22.7	2.9	1.9	1.3	0.2	0.1
S1	Half-down	11	31.5	4.2	2.8	19.5	3.8	2.6	1.6	0.4	0.2
S1	Centre	11	29.8	5.6	3.7	13.8	5.5	3.7	2.2	1.0	0.4
S1	Mid-edge	11	26.1	2.8	1.9	16.8	5.5	3.7	1.6	0.5	0.2
S2	Top-mid	11	23.0	2.5	1.7	22.0	3.5	2.3	1.0	0.2	0.1
S2	Half-down	11	21.9	3.7	2.5	21.3	3.6	2.4	1.0	0.2	0.1
S2	Centre	11	20.8	3.7	2.5	12.7	6.0	4.0	1.6	0.8	0.4
S2	Mid-edge	11	20.5	2.6	1.8	17.9	2.5	1.7	1.1	0.2	0.1
S3	Top-mid	11	22.9	6.4	4.3	15.9	4.6	3.1	1.4	0.6	0.2
S3	Half-down	11	23.0	6.2	4.2	15.8	6.8	4.5	1.4	0.7	0.3
S3	Centre	11	18.0	4.9	3.3	13.1	6.6	4.4	1.4	0.8	0.3
S3	Mid-edge	11	20.4	3.6	2.4	18.3	5.9	4.0	1.1	0.4	0.2
S4	Top-mid	11	28.4	4.2	2.8	34.7	4.4	2.9	0.8	0.2	0.1
S4	Half-down	11	25.0	3.3	2.2	19.5	3.2	2.2	1.3	0.3	0.1
S4	Centre	11	13.8	5.9	4.0	10.6	6.5	4.4	1.3	1.0	0.4
S4	Mid-edge	11	24.3	4.1	2.8	20.8	3.1	2.1	1.2	0.3	0.1

Table C9 Model calculations (Section 9) of grain aspect ratios assuming no recrystallisation. Shaded boxes indicate values not statistically different from measured specimen values.

Specimen	Sampling Point			
	Top-mid	Half-down	Centre	Mid-edge
S1	1.4	2.1	2.3	1.8
S2	1.1	2.4	3.0	1.7
S3	1.5	2.2	2.4	1.9
S4	1.1	2.3	3.2	1.7

C1.2.21 Conclusions – Axisymmetric Compression 316 Stainless Steel

- 1) Quantitative metallographic measurements on 316-stainless steel specimens were made using linear intercept methods. eleven linear scans each containing about 20 grain boundary intersections were found to yield 95% confidence intervals less than 10% of the mean grain diameters.
- 2) Specimens deformed with low friction showed reasonably uniform structures. Those deformed with high friction showed non-uniform recrystallisation.
- 3) The degree of recrystallisation corresponded well with the degree of non-uniform deformation determined by fully coupled finite element calculations.
- 4) Measurements of grain aspect ratios and their comparison with model calculated values provided a good test for the presence of recrystallisation.

C1.3 Waspaloy

All testpieces were prepared through graded emery papers followed by polishing using coarse and fine diamond wheels. Etching was carried out by swabbing the sample for 3 minutes using the following etch: 1 ml nitric acid, 2 ml sulphuric acid, 97 ml hydrochloric acid. The specimens were examined and photographed under normal reflected light. A series of images were taken, so that a montage of the whole specimen could be constructed. From this, detailed measurements of grain size and shape were made in six distinct areas as shown in Fig C14. The height of the sampling area was 550 pixels and the width 600 pixels, where 1230 pixels represents 1mm on the sample.

Grain size measurements were made using the linear intercept method. Within the sampling area, 12 guide lines were constructed, each of 600 pixels length. The number of grain boundaries intercepting each line was counted. Twin boundaries were not counted. All measurements were made directly from the digital image on the computer. In addition, the grain widths at the point of intersection of a line drawn down the centre of the specimen were measured, from the top surface to the middle, and again on a line drawn mid-way between the centre-line and the end of the top surface. For all measurements, the mean values, standard deviations and 95% confidence intervals for the mean were calculated.

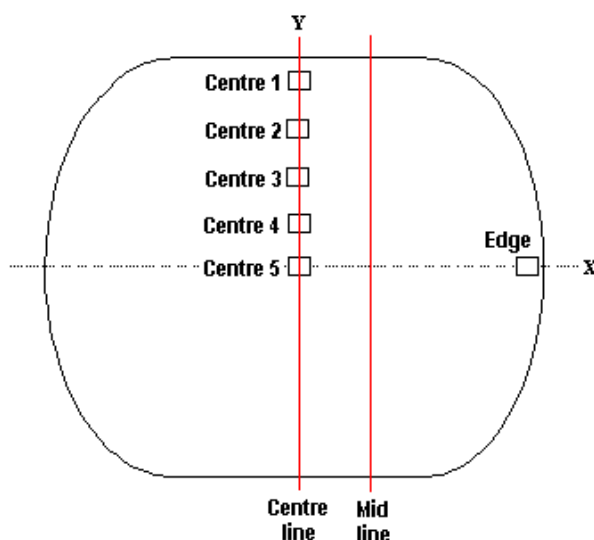


Figure C14 Schematic diagram showing areas of the sample which were measured. Not to scale.

The linear intercept grain sizes for as received and deformed specimens are shown in Table C10. The microstructure of the initial stock is shown in Fig C15 and microstructures of deformed specimens at specific sampling areas are shown in Figs C16-C19.

Specimens deformed with low friction (W1 and W3) showed fully recrystallised structures with average grain sizes about half the as received material. The lower strain rate specimen (W1) had a grain size slightly smaller than for high strain rate (W3) but for both specimens there was a decrease in grain size from specimen edge to centre (from sampling position Centre-1 to Centre-5).

High friction specimens were not fully recrystallised everywhere. Some parts (e.g. Centre-1) were hardly recrystallised at all and others (e.g. Centre-5) were largely fully recrystallised. Because the recrystallised grain size was not very different from the initial grain size, it was not possible to measure volume fraction recrystallised directly. However, the variation in fraction recrystallised exhibited itself in lower mean values as the fraction increased. The grain diameter surveys down representative centre-lines are shown in Figs C20 and C21. In all cases, the point at which uniform small grain size is observed (i.e. largely recrystallised material) corresponds well with the strain distributions in the models.

Table C10 Linear intercept grain sizes for as received and deformed Waspaloy.

Specimen Number	Sample Area	Sample Size 600 μm line	grain size μm		
			Mean	Std. dev.	95% conf. interval
As Received		55	48.5	6.6	1.7
W1	Centre 1	12	22.3	4.0	2.5
W1	Centre 2	12	22.3	4.0	2.5
W1	Centre 3	12	21.9	2.5	1.6
W1	Centre 4	12	19.0	2.0	1.3
W1	Centre 5	12	19.3	2.6	1.7
W1	Edge	12	22.1	2.1	1.3
W2	Centre 1	12	36.0	9.8	6.2
W2	Centre 2	12	28.6	5.4	3.5
W2	Centre 3	12	23.7	3.9	2.5
W2	Centre 4	12	22.6	3.4	2.2
W2	Centre 5	12	19.5	2.0	1.3
W2	Edge	12	26.7	3.6	2.3
W3	Centre 1	12	26.4	3.6	2.3
W3	Centre 2	12	25.5	4.3	2.7
W3	Centre 3	12	22.1	4.8	3.0
W3	Centre 4	12	21.1	2.1	1.3
W3	Centre 5	12	21.8	3.6	2.3
W3	Edge	12	24.1	2.6	1.7
W4	Centre 1	12	42.0	6.1	3.9
W4	Centre 2	12	38.5	7.3	4.7
W4	Centre 3	12	23.6	3.5	2.2
W4	Centre 4	12	20.6	2.4	1.6
W4	Centre 5	12	20.3	2.7	1.7
W4	Edge	12	22.5	2.3	1.5

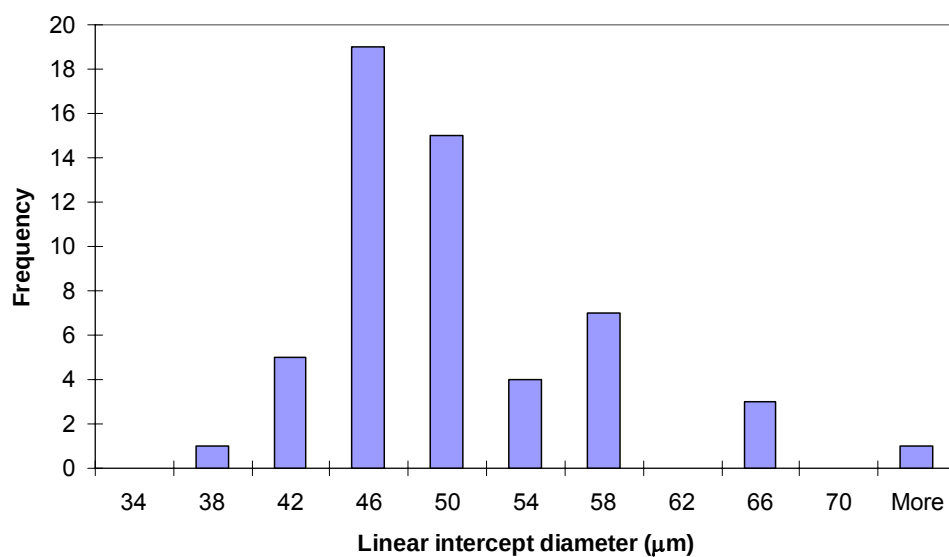


Figure C15 Microstructure (x105) and linear intercept grain size histogram for as-received material.

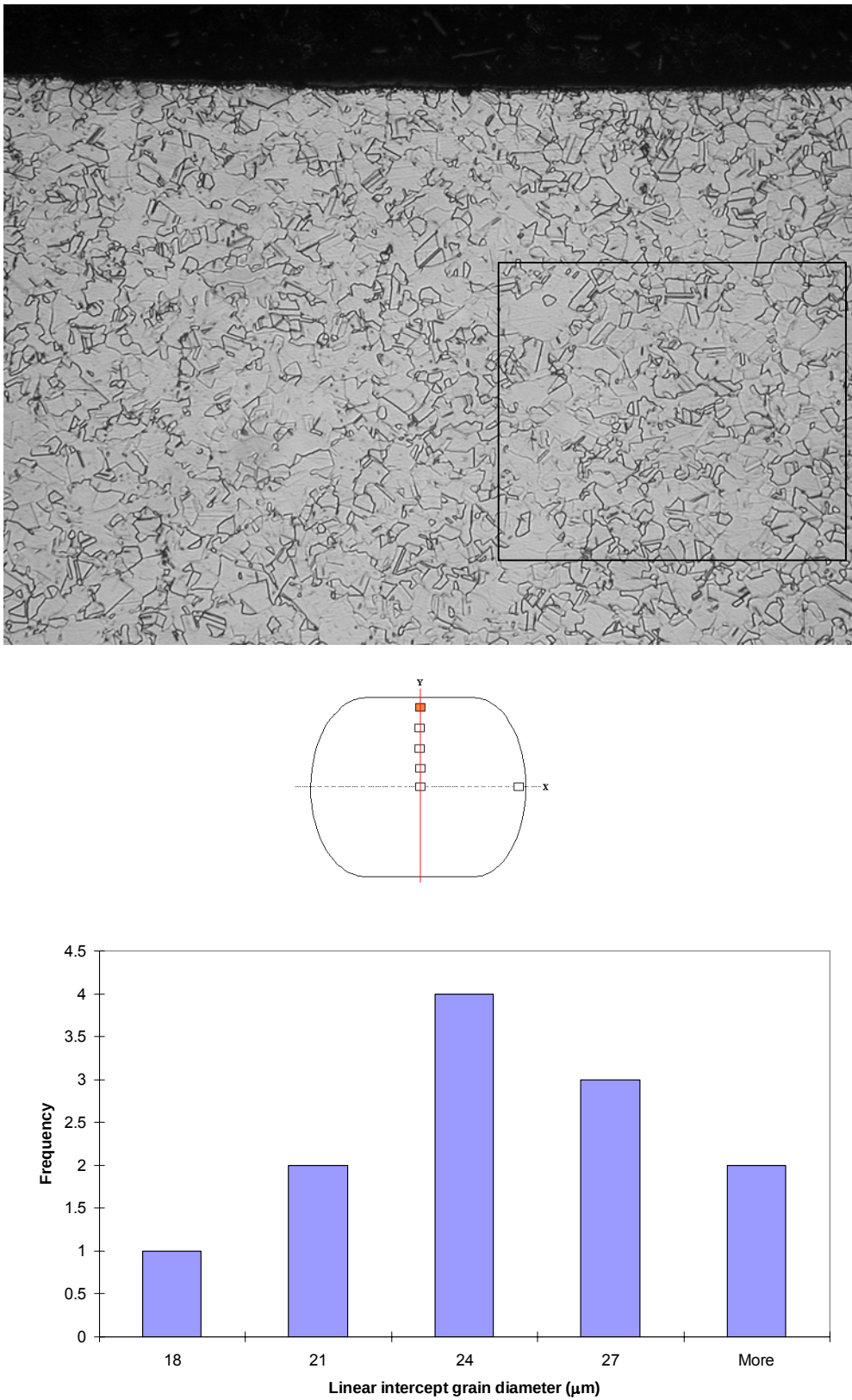


Figure C16 Microstructure (x105) and linear intercept grain size histogram for sample W1, position Centre 1.

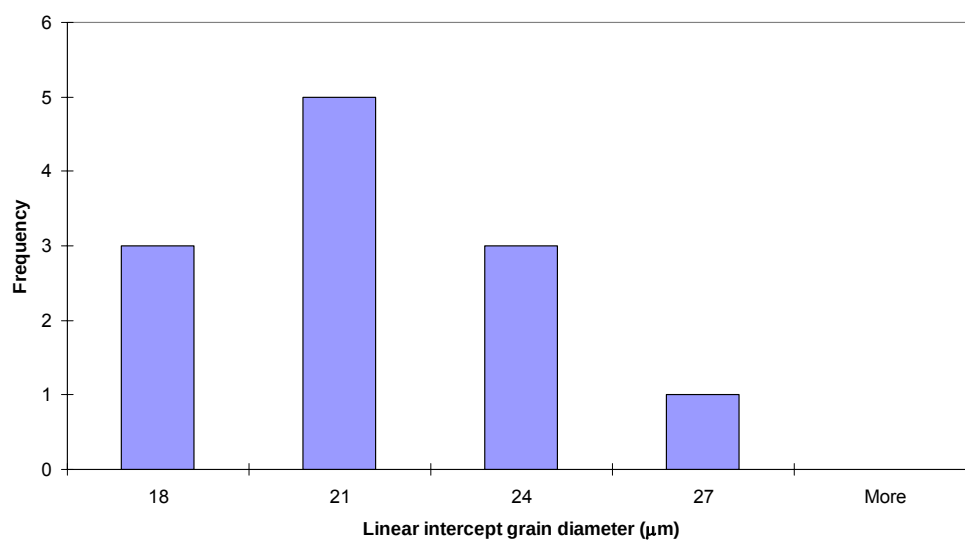
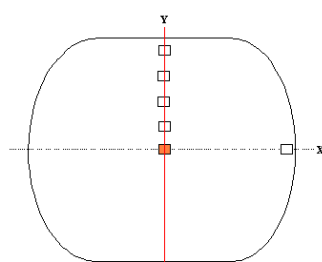
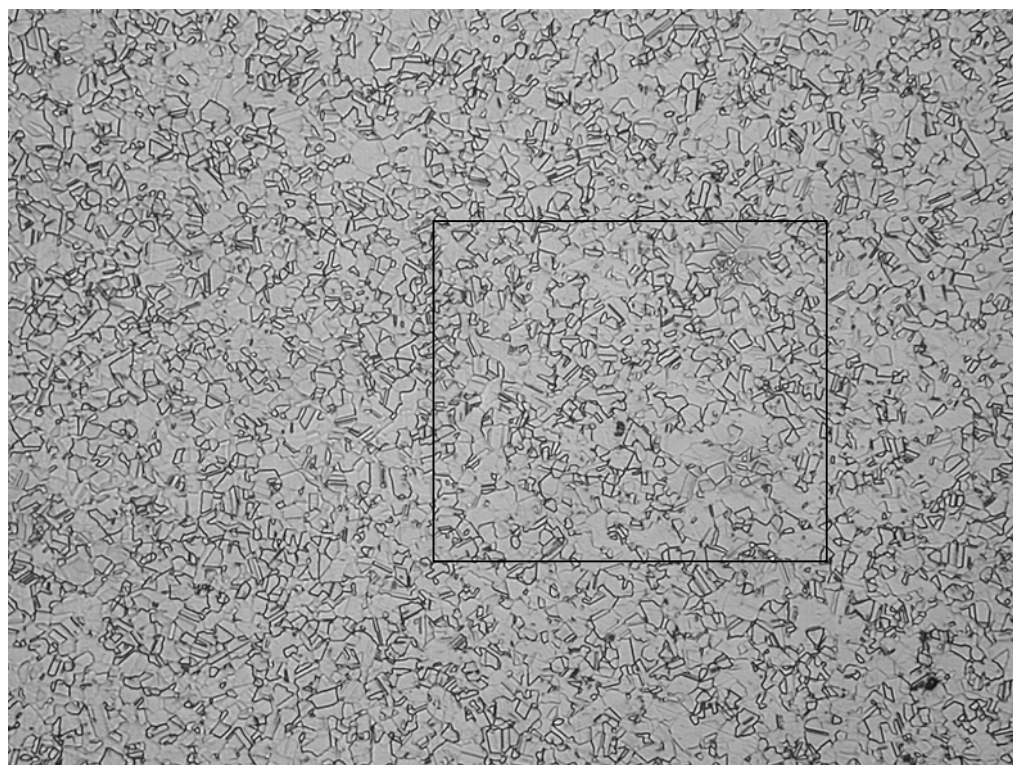


Figure C17 Microstructure (x105) and linear intercept grain size histogram for sample W1, position Centre 5.

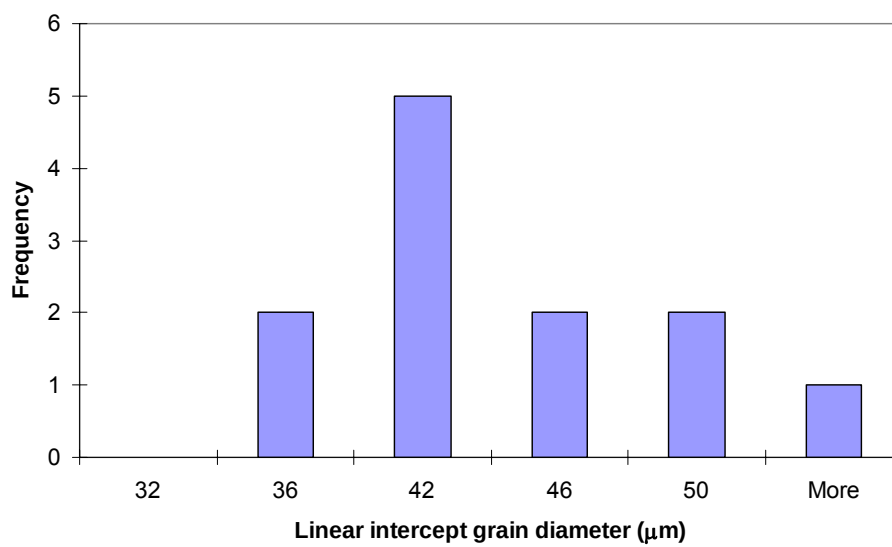
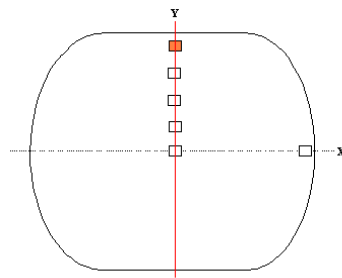
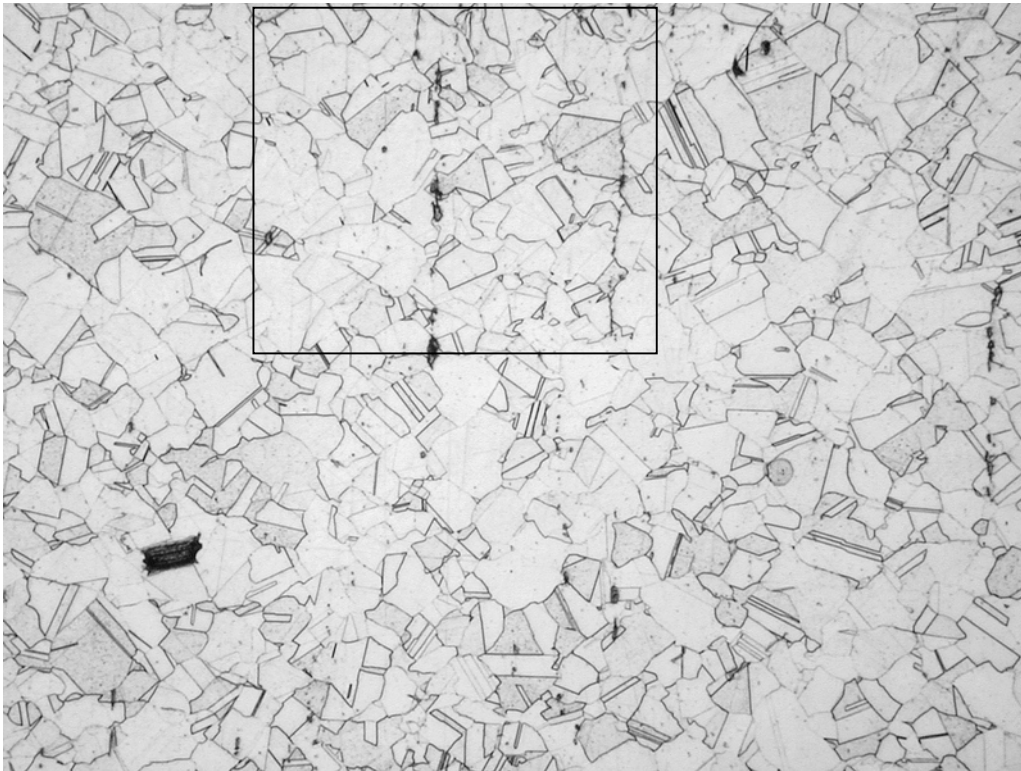


Figure C18 Microstructure (x105) and linear intercept grain size histogram for sample W4, position Centre 1.

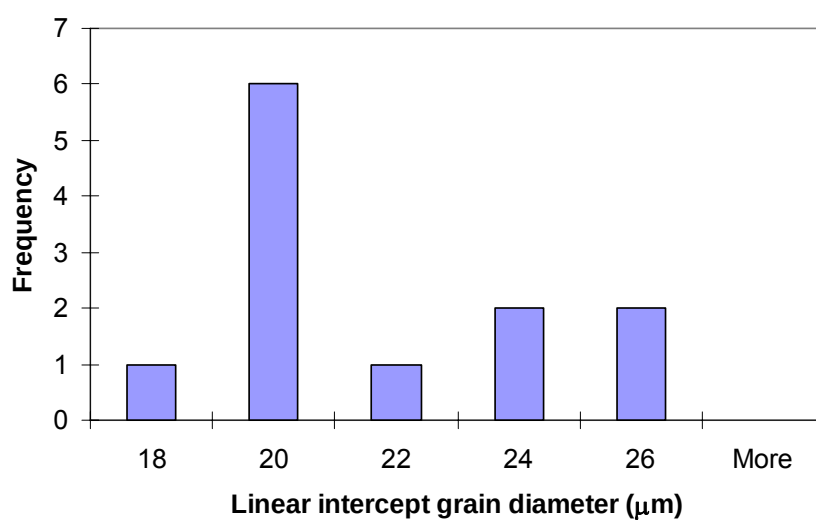
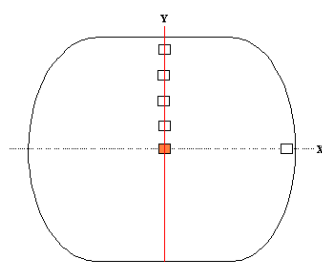
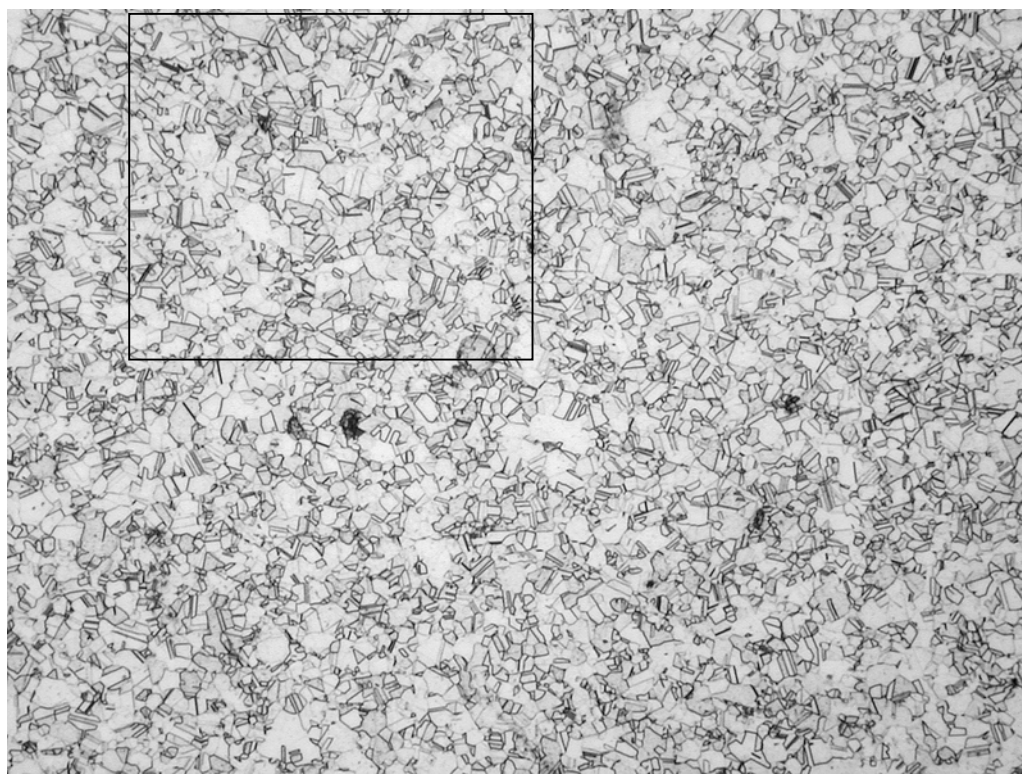


Figure C19 Microstructure (x105) and linear intercept grain size histogram for sample W4, position Centre 5.

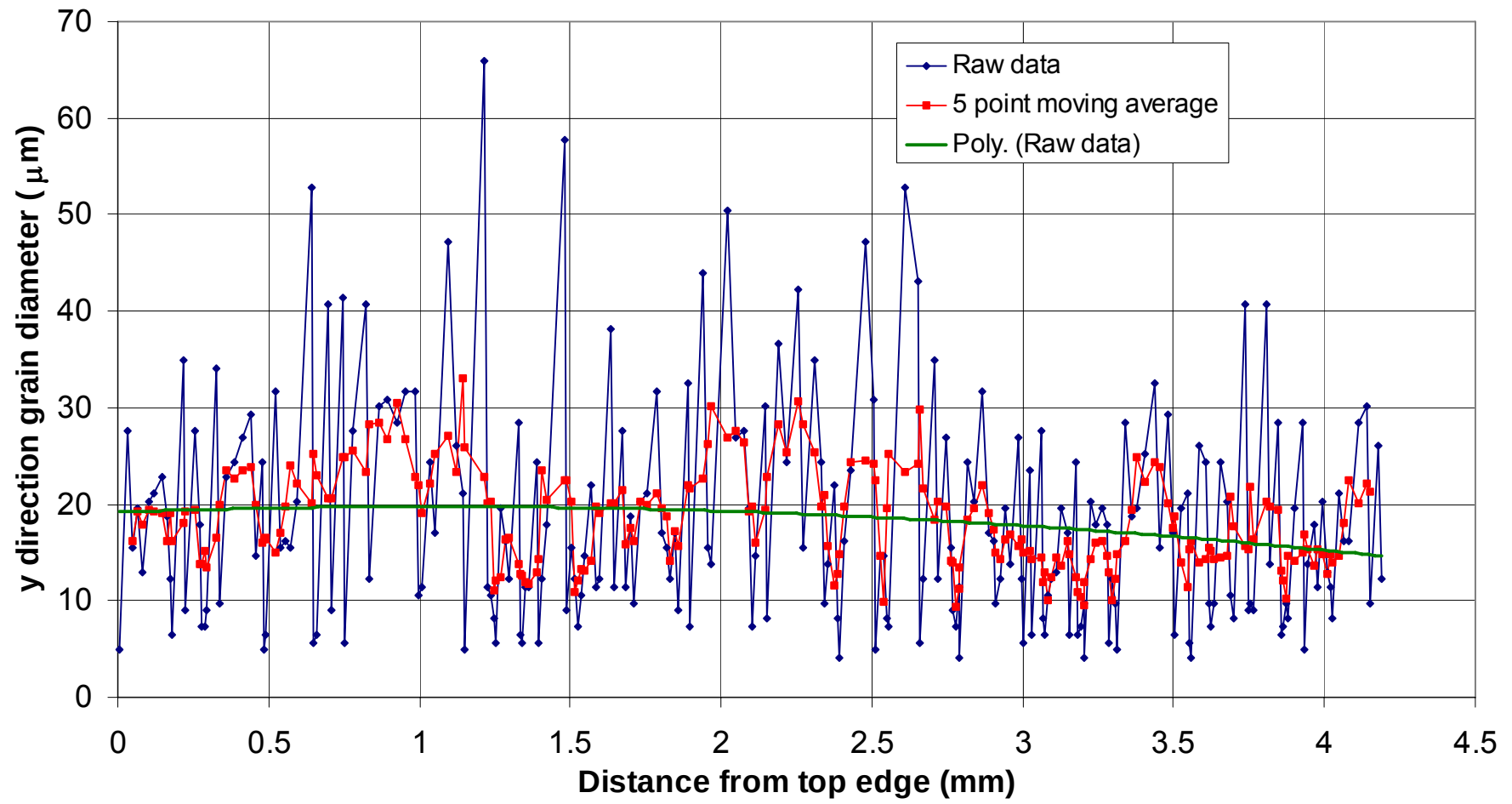


Figure C20 Centre line grain diameters for specimen W1.

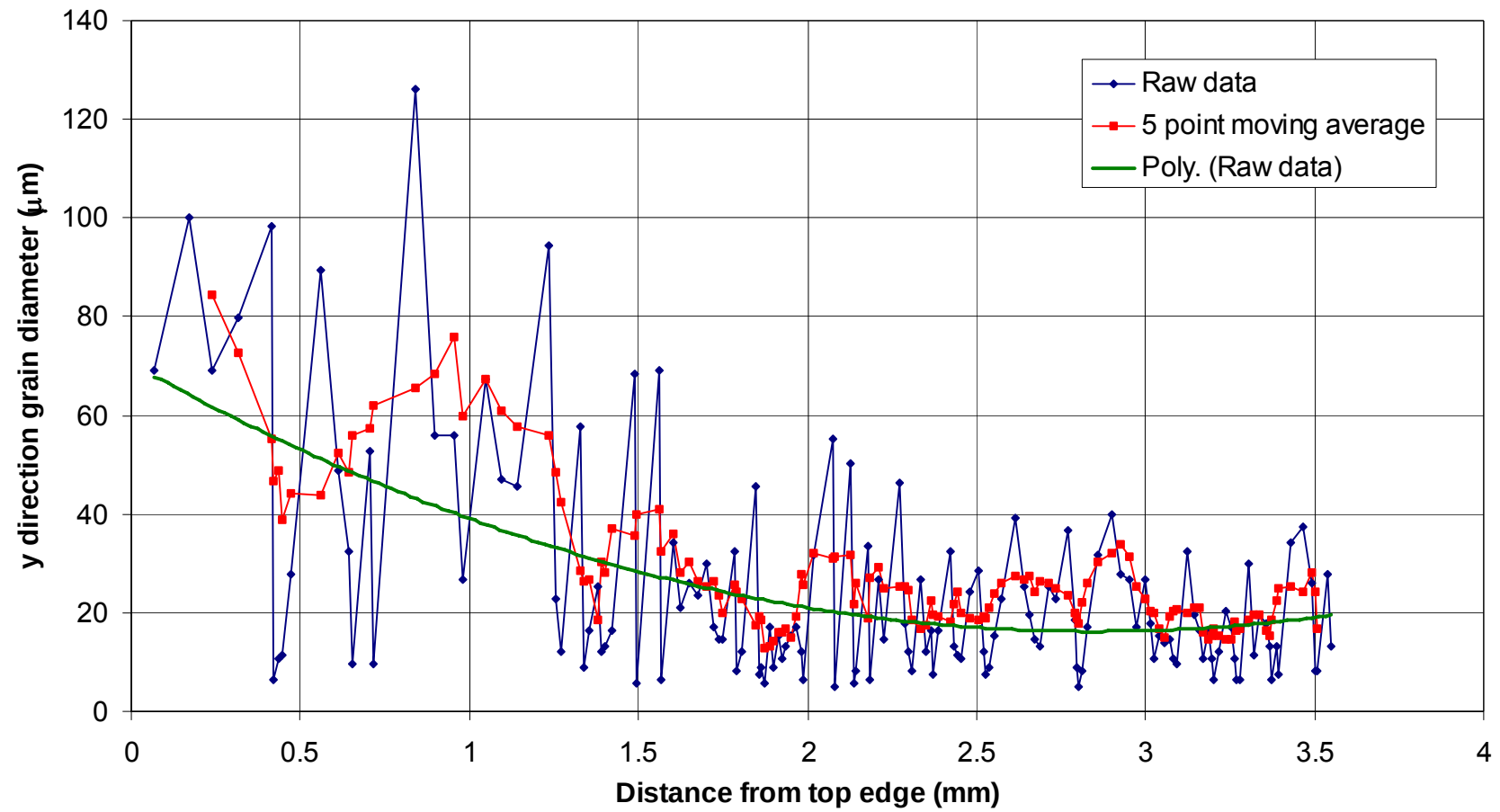


Figure C21 Centre line grain diameters for specimen W4

C1.3.1 Conclusions – Axisymmetric Compression Waspaloy

- 5) Quantitative metallographic measurements on Waspaloy specimens have been made using linear intercept methods. Twelve linear scans each containing about 15 grain boundary intersections were found to yield 95% confidence intervals less than 10% of the mean grain diameters.
- 6) Specimens deformed with low friction showed reasonably uniform fully recrystallised structures. Those deformed with high friction showed non-uniform recrystallisation.

The degree of recrystallisation corresponded well with the degree of non-uniform deformation determined by fully coupled finite element calculations.

C1.4 Ti6Al4V

All testpieces were prepared through graded emery papers followed by polishing using coarse and fine diamond wheels. Etching was carried out using a variant of Kroll's reagent: 1 ml HF, 2 ml nitric acid, 97 ml distilled water. The samples were etched for 2 minutes.

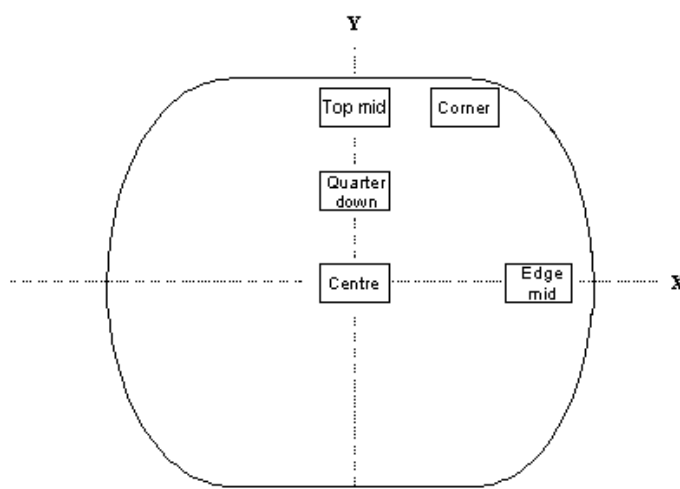


Figure C22 Schematic diagram showing areas of the sample which were measured. Not to scale.

The specimens were examined and photographed under normal reflected light. All samples showed primary alpha grains in a matrix of transformed beta. A full photographic montage was not carried out for these samples since there was very little difference in the structure over the cross-section. Detailed measurements of volume fraction of the alpha phase were made in five distinct areas as shown in Fig C22. Care was taken to avoid measurements at the very edge of the samples, due to the formation of an alpha-case. The width of the sampling area was approximately 0.64 mm, and the height was 0.48 mm. This equates to an area of 1600×1200 pixels, where $2500 \text{ pixels} = 1 \text{ mm}$.

For each area, a 10 point line was superimposed on the image and the volume fraction alpha measured by counting the number of points which fell on the alpha particles. The line was repositioned 10 times to give 100 point counts in all. All measurements were made directly from the digital image on the computer. For all measurements, the mean values, standard deviations and 95% confidence intervals for the mean were calculated.

The point counted volume fractions of primary alpha for as-received and deformed specimens are shown in Table C11. The microstructure of the initial stock is shown in Fig C23 and the structures of all deformed specimens and at all sampling areas are shown in Figs C24-C27.

Table C11 Point counted alpha volume fractions for as received and deformed Ti6Al4V.

Specimen Number	Sample Area	Sample Size 100 points	Volume fraction α		
			Mean	Std. dev.	95% conf. interval
As Received		100	0.60	0.14	0.10
T1	Centre	100	0.50	0.07	0.05
T1	Quarter Down	100	0.50	0.13	0.09
T1	Top Mid	100	0.50	0.10	0.07
T1	Corner	100	0.32	0.14	0.10
T1	Edge Mid	100	0.36	0.15	0.10
T2	Centre	100	0.53	0.13	0.09
T2	Quarter Down	100	0.48	0.12	0.09
T2	Top Mid	100	0.50	0.12	0.08
T2	Corner	100	0.51	0.12	0.09
T2	Edge Mid	100	0.55	0.10	0.07
T3	Centre	100	0.29	0.12	0.08
T3	Quarter Down	100	0.26	0.10	0.07
T3	Top Mid	100	0.28	0.11	0.08
T3	Corner	100	0.25	0.12	0.08
T3	Edge Mid	100	0.30	0.13	0.09
T4	Centre	100	0.20	0.12	0.08
T4	Quarter Down	100	0.20	0.13	0.09
T4	Top Mid	100	0.21	0.15	0.09
T4	Corner	100	0.21	0.10	0.07
T4	Edge Mid	100	0.26	0.13	0.09

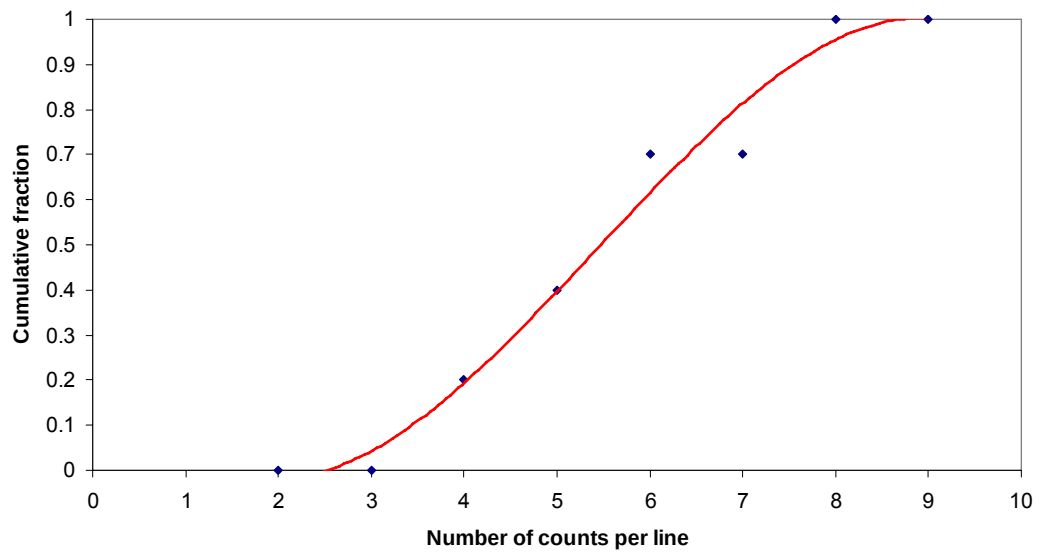
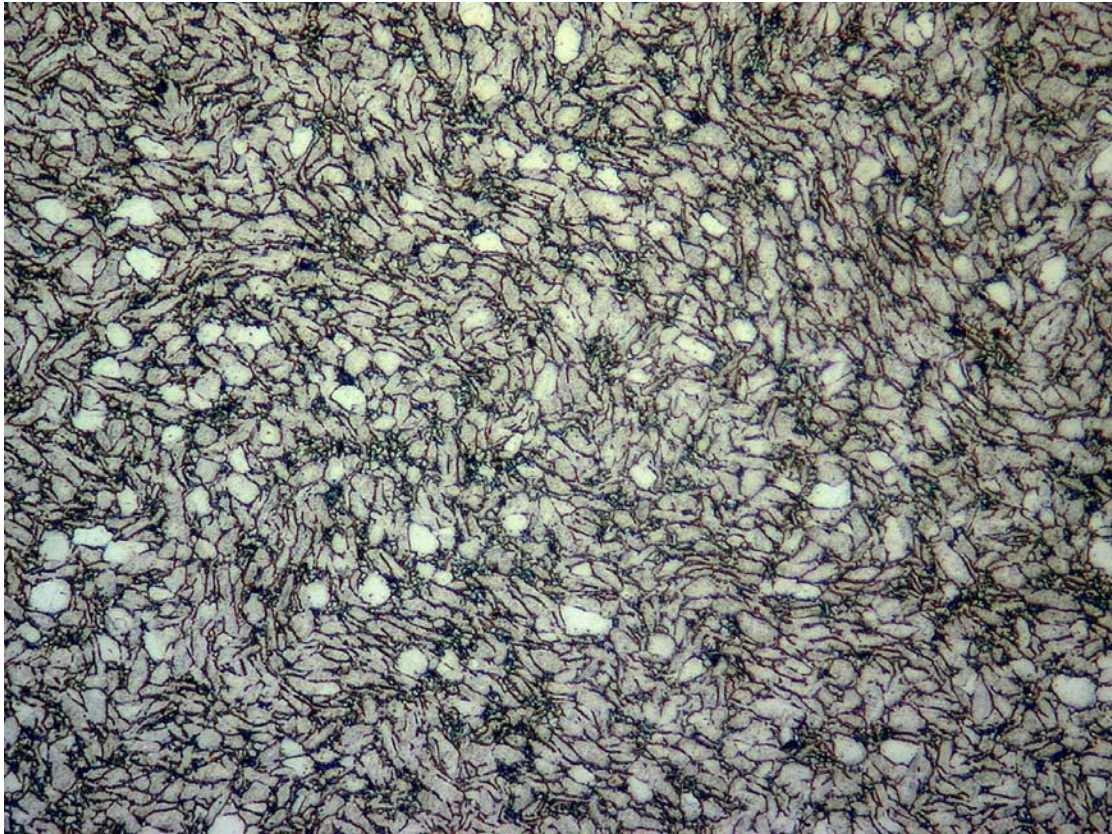


Figure C23 Microstructure ($\times 330$) and volume fraction α distribution for as-received Ti6Al4V.

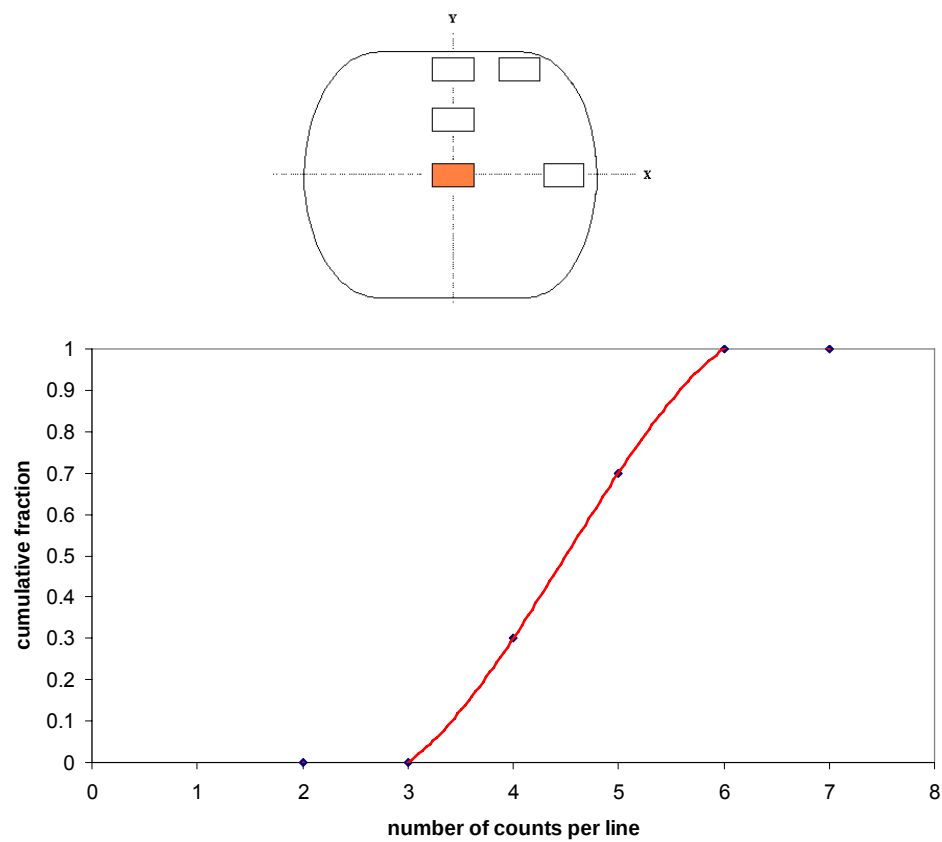


Figure C24 Microstructure (x 210) and volume fraction α distribution for specimen T1 at position Centre.

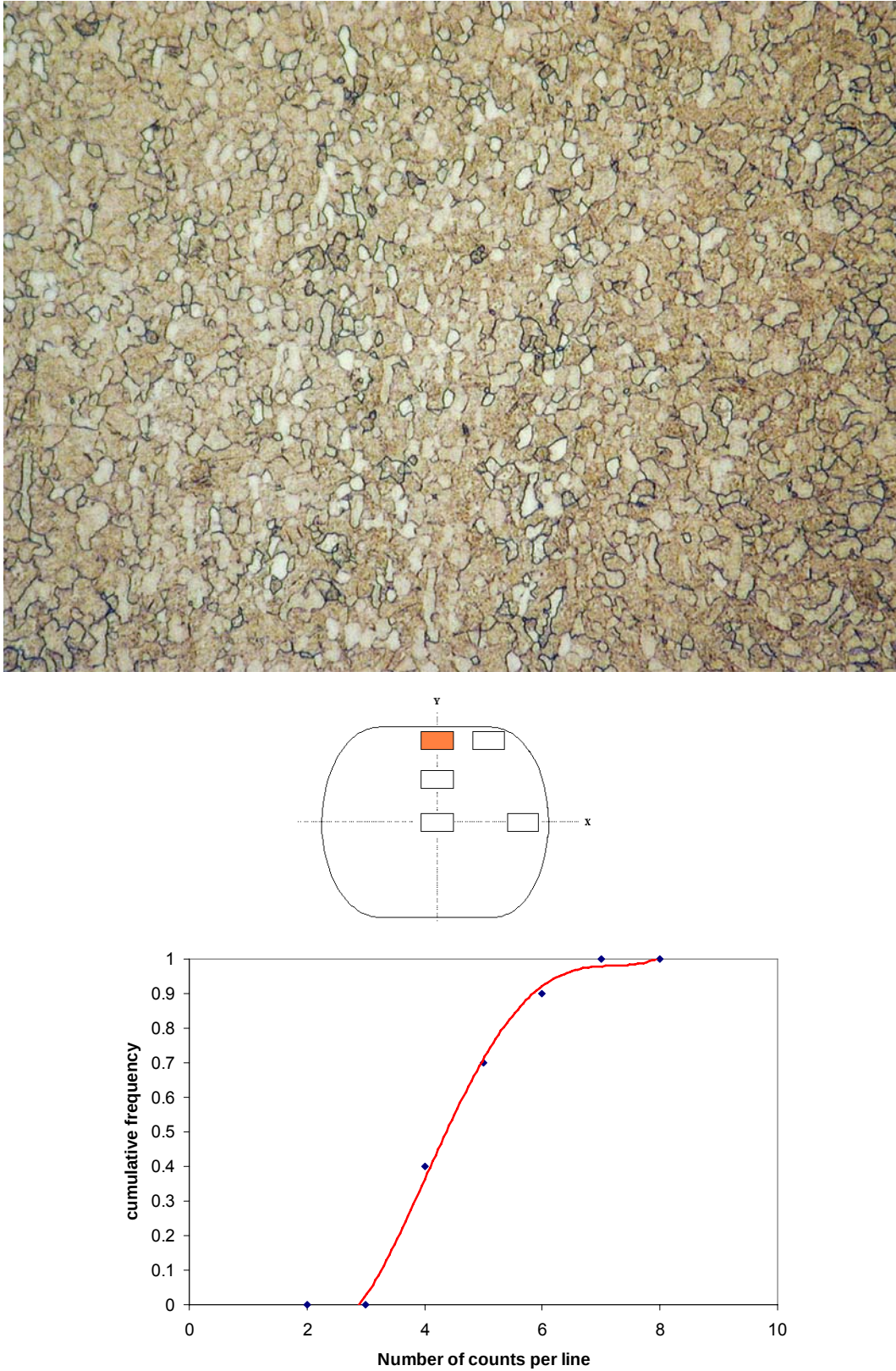


Figure C25 Microstructure ($\times 210$) and volume fraction α distribution for specimen T1 at position Top mid

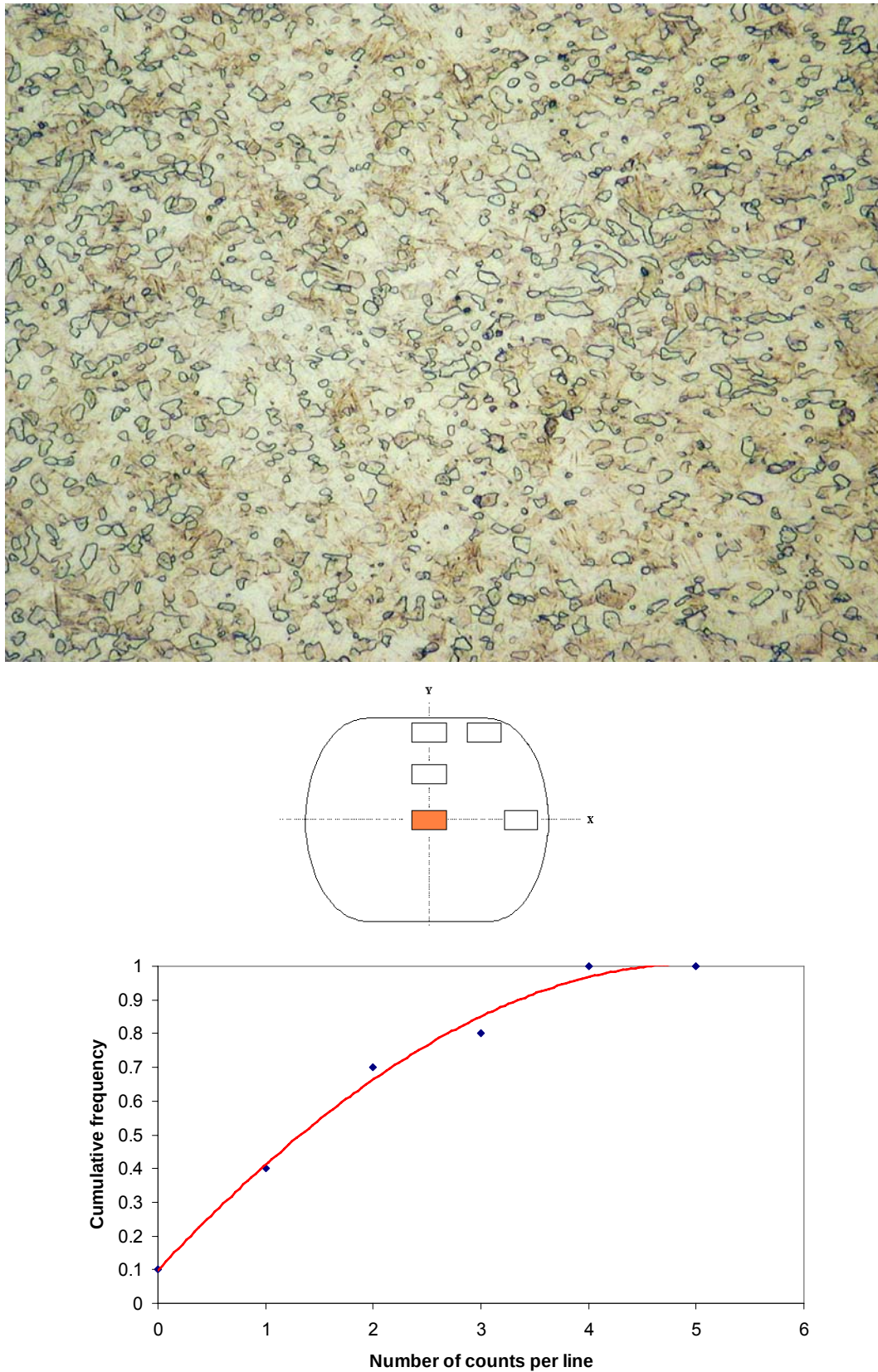


Figure C26 Microstructure (x 210) and volume fraction α distribution for specimen T4 at position Centre

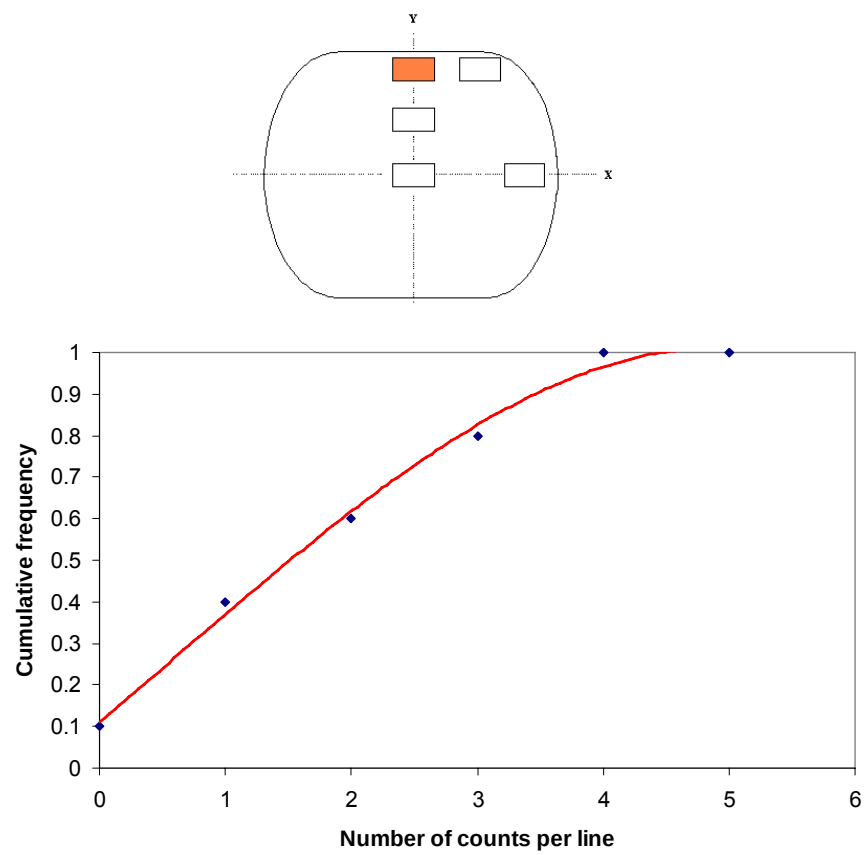
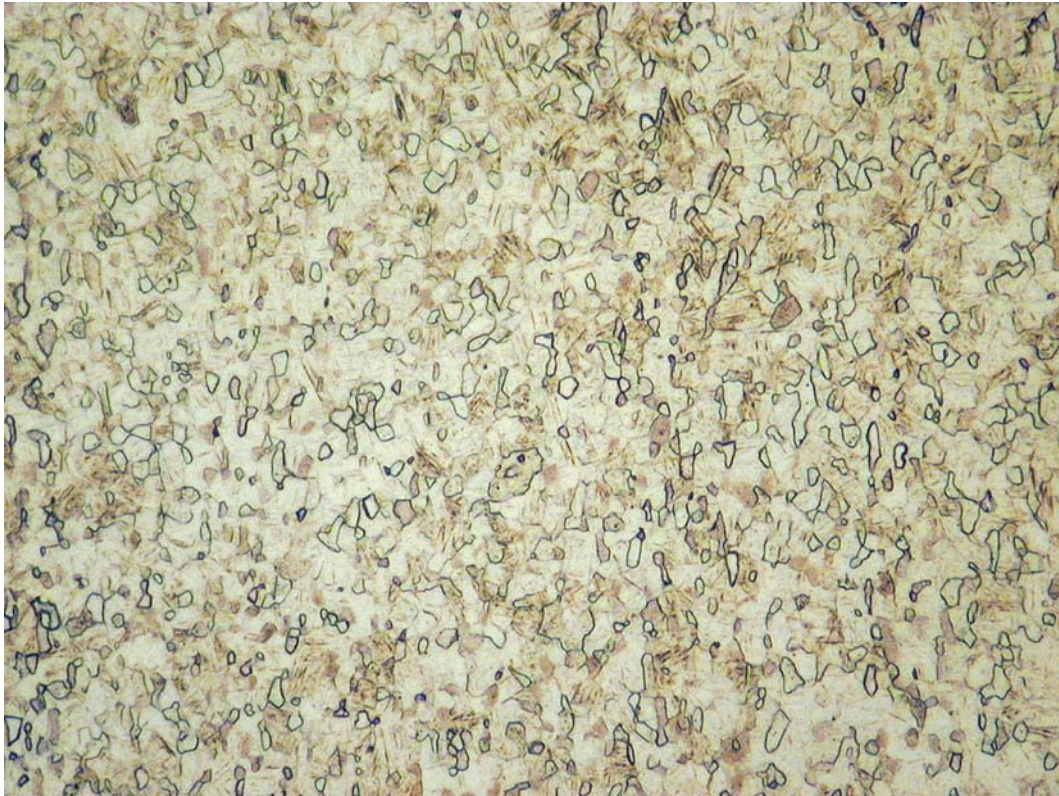


Figure C27 Microstructure ($\times 210$) and volume fraction α distribution for specimen T4 at position Top mid.

All specimens deformed at low strain rates (T1 and T2) exhibited a volume fraction α which was greater than the equilibrium volume fraction for 950 °C (approximately 0.25). However fast strain rate tests (T3 and T4) had volume fractions close to that for equilibrium. It is clear that the initial soaking time was insufficient to achieve equilibrium α at 950°C but some of the forging conditions were able to attain equilibrium condition.

C1.4.1 Conclusions – Axisymmetric Compression Ti6Al4V

- 7) Quantitative metallographic measurements of α volume fraction in deformed Ti6Al4V were made by point counting methods. Sampling by 100 random points produces 95% confidence intervals of about 0.07.
- 8) Specimens had not attained equilibrium volume fractions of α at the commencement of deformation at 950°C.
- 9) Rises in temperature during straining were a major driving force in reducing volume fractions of α to their equilibrium values.

C2 Case Studies – PSC testpieces

Optical metallographic analysis was conducted of plane strain deformed specimens of the four alloys, Waspaloy, Ti6Al4V, Al 5052 and 316 stainless steel. Specimens were deformed under varying temperature, strain rate and friction conditions, in order to assess the effect of these variables on the different alloy microstructures. The details of the testing were presented in Tables C1-C4.

Experimental Procedures

Plane strain compression specimens were heated to testing temperature within 10 minutes (5 minutes for Al 5052) and water quenched within 3 seconds at the end of the test. Tests were carried out at a true constant strain rate. High friction conditions were achieved by using dry specimens, while low friction conditions were attained through lubrication with a glass mixture. Following deformation, specimens were sectioned along their diameter parallel to the direction of loading. All specimens were prepared for metallographic examination through graded emery papers followed by polishing using coarse (6 μ m) and fine (1 μ m) diamond wheels. Al 5052 required a further final polish using a SiO₂ suspension in water. Specimens were then etched using the procedures shown in Table C12.

Table C12 Etching procedure for plane strain compression specimens.

Material	Etch	Method
Waspaloy	1 ml nitric acid, 2 ml sulphuric acid, 97 ml hydrochloric acid	Specimens swabbed for 3 minutes
316 stainless steel	2 parts hydrochloric acid, 2 parts glycerol, 1 part nitric acid	Specimens immersed in hot water for 5 minutes prior to etching by swabbing for 2 minutes
Al 5052	Barker's reagent (5 g HBF_4 , 200 ml distilled water)	Specimens etched electrolytically for 3.5 minutes at 20 V, depositing an anodised layer on the surface.
Ti6Al4V	Kroll's reagent (1 ml HF, 2 ml nitric acid, 97 ml distilled water)	Specimens immersed in etch for 2 minutes.

Two 316 stainless steel, Ti6Al4V and Waspaloy specimens were examined and photographed under normal reflected light, while the Al 5052 specimens were photographed under polarised light. Specimens were photographed at varying magnifications, depending on microstructure scale, shown in Table C13.

Table C13 PSC testpiece photograph magnifications.

Material	Image resolution (pixels)	Magnification
Al 5052	2560 × 1920	× 50
316 stainless steel	2560 × 1920	× 125
Ti6Al4V	2560 × 1920	× 100
Waspaloy	1600 × 1200	× 125 (lubricated) × 160 (dry)

Images were taken along the width of the specimen in 3 different 1 mm strips shown in Figure C28. The strips are located at the shoulder of the specimen, $\frac{1}{4}$ way through the specimen and $\frac{1}{2}$ way through the specimen.

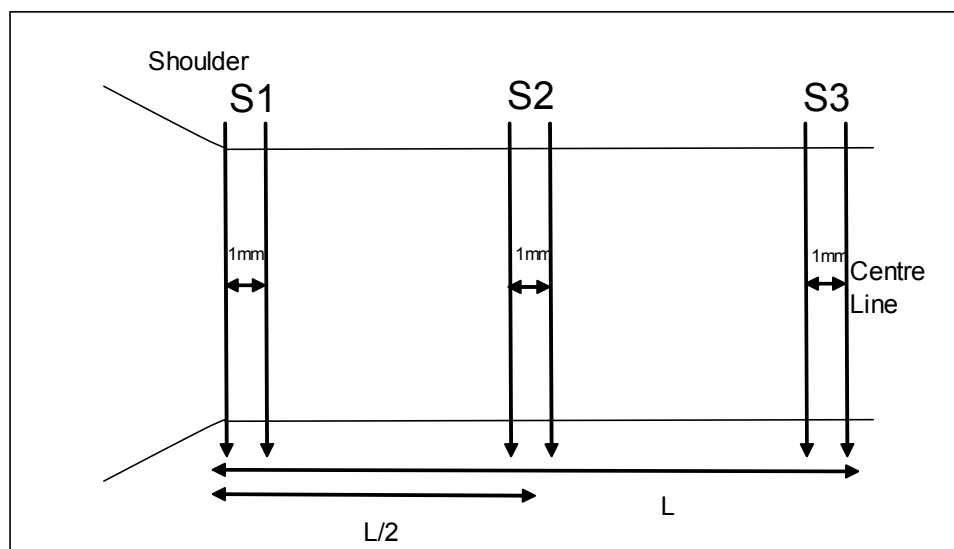


Figure C28: Schematic showing photographed areas of specimen. Not to scale.

Grain size measurements were made using the linear intercept method, with guide lines running through the centre of each 1mm strip for the entire width of the specimen. The number of grain boundaries intercepting each line was counted, with measurements made directly from the digital images using the program SigmaScan. For all measurements, the mean values, standard deviations and 95% confidence intervals for the mean were calculated.

Results

Al 5052 Al 5052 as-received and deformed grain sizes are shown in Table C14, while Figs C29-C32 show centreline grain diameters. A significant reduction in grain diameter (compared with the as-received material) in the loading direction is apparent, while grain diameters in the y direction are reasonably constant at all 3 measured locations (Table C14). Lubricated specimens (A1 and A3) show a similar grain distribution pattern with a larger grain diameters at the centre of the shoulder location compared to the surface grain sizes. At the L/2 location, the opposite was the case with larger surface grains giving way to smaller grain diameters at the centre of the strip, grain diameters along the length of the L strip were constant from surface to centre in both specimens. Unlubricated testpieces (A2 and A4) gave less predictable results through they tended towards larger grain diameter at the specimen surfaces, when compared with the centre of the specimens.

316 stainless steel Table C15 shows 316 stainless steel as-received and deformed grain sizes, while Figs C33-C36 show centreline grain diameters. Mean grain diameters of the tested specimens were approximately half that of the as received grain diameters, with all specimens having a mean grain diameter of roughly 10µm in all measured locations.

Although there was little variation in grain diameters in all four specimens, some underlying patterns were apparent. Lubricated specimens (S1 and S3) displayed similar grain distributions at all 3 measured locations, with a rise in grain diameter from the specimen surface towards the specimen centre at the shoulder location and the L location. At the location L/2 the opposite was the case with larger grain diameters at the surface leading to smaller grain diameters at the specimen centre. The dry specimens (S2 and S4) also exhibited

comparable results with an increase in grain size from specimen surface to centre at the shoulder location, with the opposite being the case at the L/2 and L locations for both specimens.

Ti6Al4V Alpha phase volume fraction was not measured in the Ti6Al4V PSC testpieces (compared with results from AC testpieces described in Appendix C1.4).

Waspaloy Table C16 shows Waspaloy as-received and deformed grain sizes, while Figs C37-C40 show Waspaloy centreline grain diameters. A substantial decrease in specimen grain diameter can be seen when compared with as-received grain sizes. It can also be seen that the mean grain diameter for lubricated specimens is greater than that of dry specimens. Lubricated testpieces exhibited a uniform grain diameter distribution across the 3 measured areas, whereas more variation was seen in the unlubricated specimens, which tended towards larger grain diameters at the specimen surfaces compared with the centres.

Al 5052

Table C14: Al 5052 as-received and deformed grain sizes.

Specimen Number	Sample Area	y direction sample size (µm)	y direction linear intercept diameter (µm)
A1	As-received	4711	63.7
A1	Shoulder	7375	49.8
A1	L/2	7315	49.4
A1	L	7432	43.7
A2	As-received	4703	52.3
A2	Shoulder	7367	47.2
A2	L/2	7314	47.2
A2	L	7367	45.2
A3	As-received	4821	70.9
A3	Shoulder	7340	45.9
A3	L/2	7594	37.2
A3	L	7410	39.6
A4	As-received	5225	60.1
A4	Shoulder	7378	37.5
A4	L/2	7394	38.5
A4	L	7439	41.6

316 Stainless Steel

Table C15 316 stainless steel as-received and deformed grain sizes

Specimen Number	Sample Area	y direction sample size (μm)	y direction linear intercept diameter (μm)
SS1	As-received	5392	19.7
SS1	Shoulder	7759	9.9
SS1	L/2	7281	10.4
SS1	L	7265	10.2
SS2	As-received	5392	19.7
SS2	Shoulder	7235	10.4
SS2	L/2	7243	10.0
SS2	L	7262	11.4
SS3	As-received	5967	21.4
SS3	Shoulder	7195	10.8
SS3	L/2	7300	9.9
SS3	L	7249	10.7
SS4	As-received	5971	19.8
SS4	Shoulder	7177	10.8
SS4	L/2	7230	10.4
SS4	L	7272	11.2

Waspaloy

Table C16: Waspaloy as-received and deformed grain sizes.

Specimen Number	Sample Area	y direction sample size (μm)	y direction linear intercept diameter (μm)
W1	As-received	3166	30.7
W1	Shoulder	7716	13.4
W1	L/2	7422	14.0
W1	L	7248	12.1
W2	As-received	2581	20.0
W2	Shoulder	7705	10.3
W2	L/2	7591	9.9
W2	L	7557	9.1
W3	As-received	3014	61.5
W3	Shoulder	7523	16.3
W3	L/2	7450	15.8
W3	L	7311	14.4
W4	As-received	2526	24.8
W4	Shoulder	7623	10.3
W4	L/2	7588	9.8
W4	L	7585	9.8

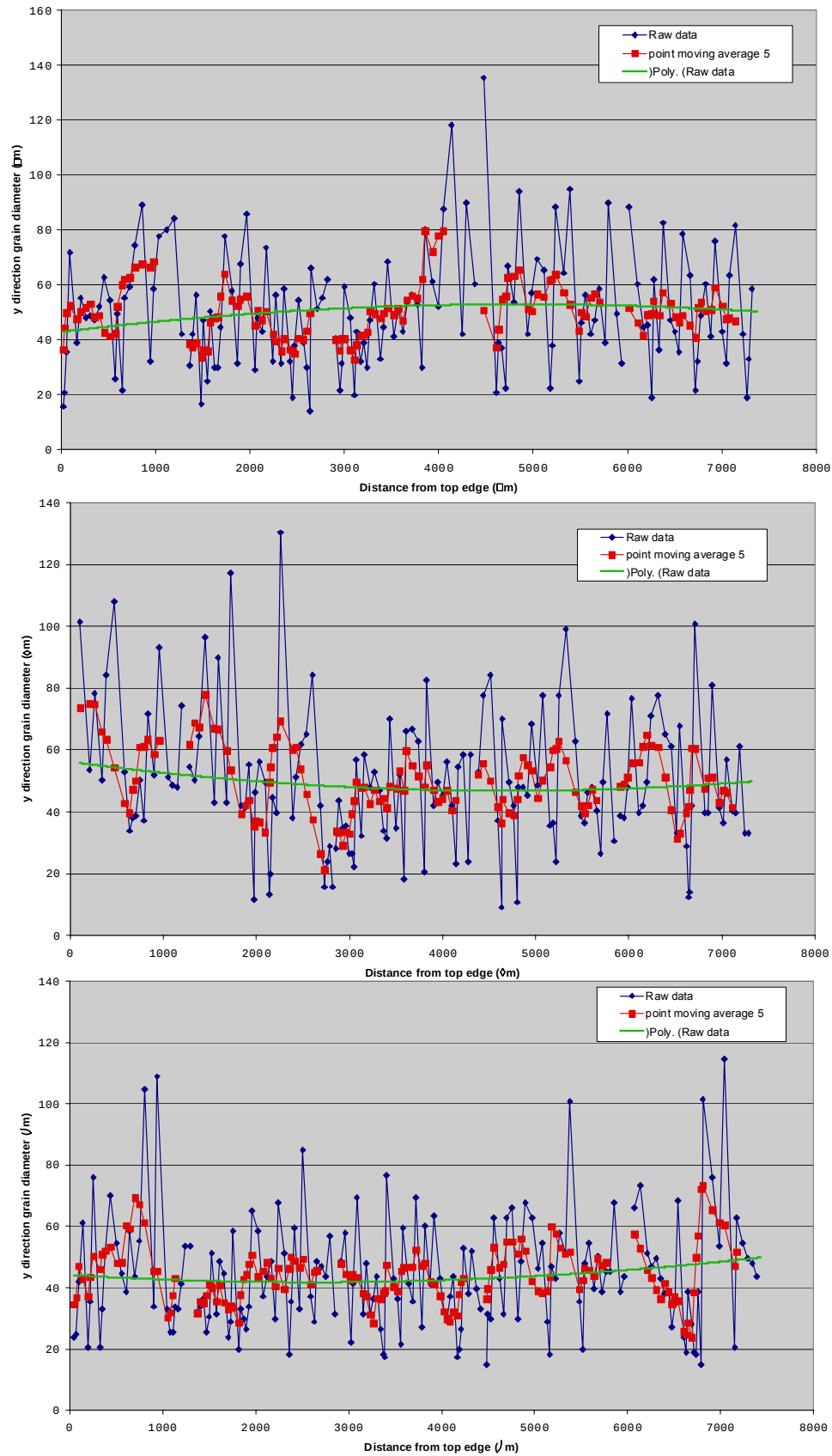


Figure C29 Centre line grain diameters for specimens A1: top to bottom (shoulder, L/2, L).

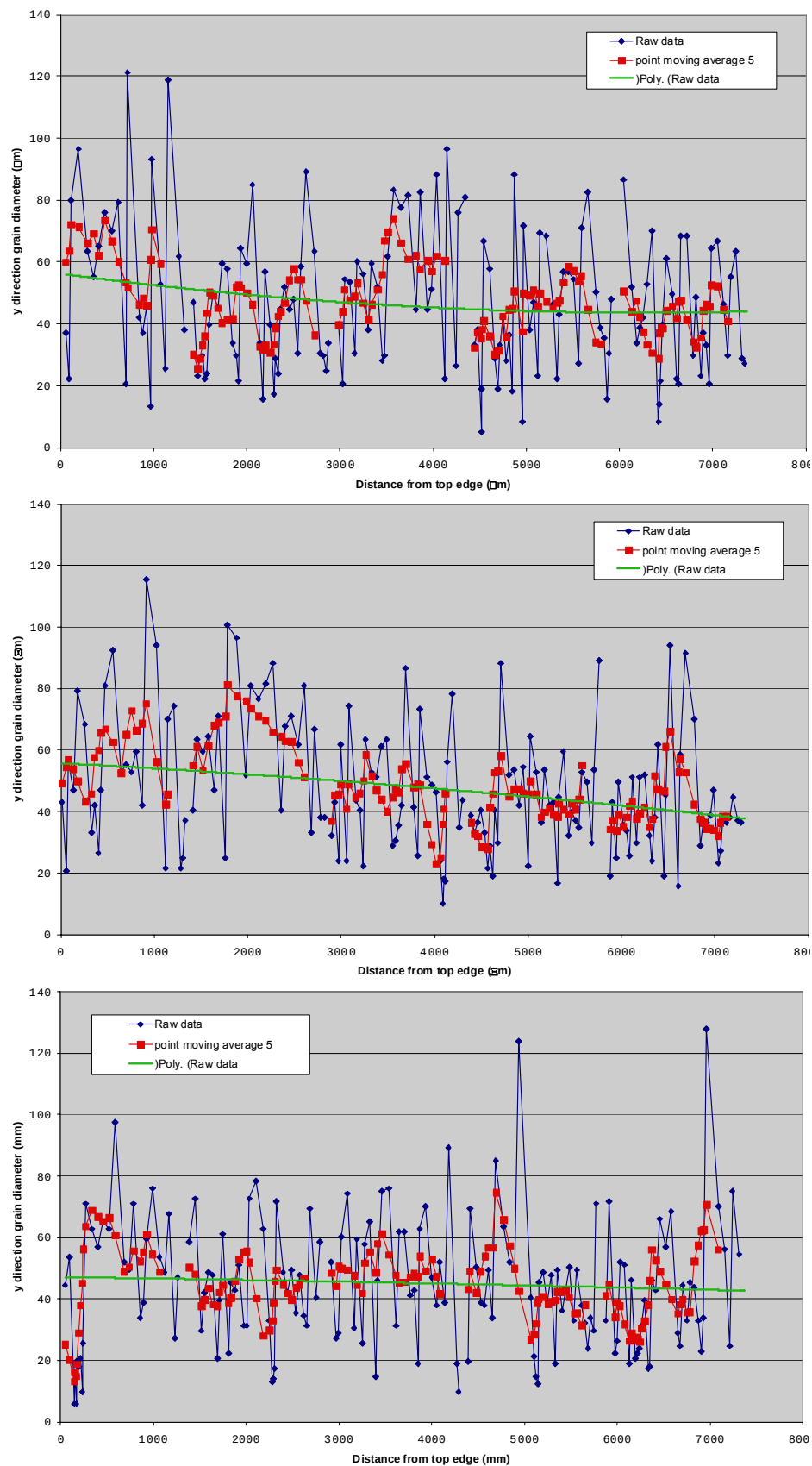


Figure C30 Centre line grain diameters for specimens A2: top to bottom (shoulder, L/2, L).

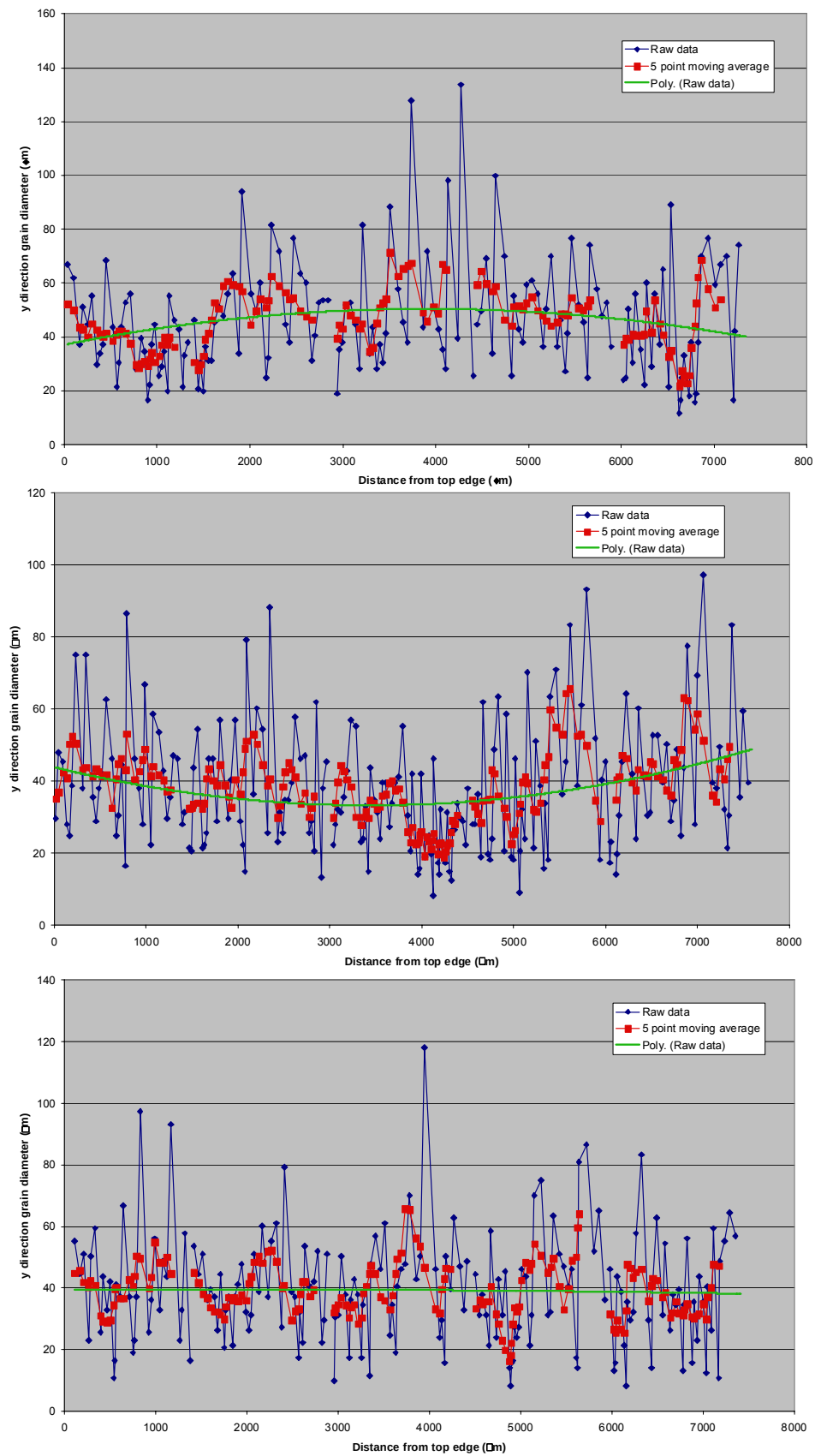


Figure C31 Centre line grain diameters for specimens A3: top to bottom (shoulder L/2, L).

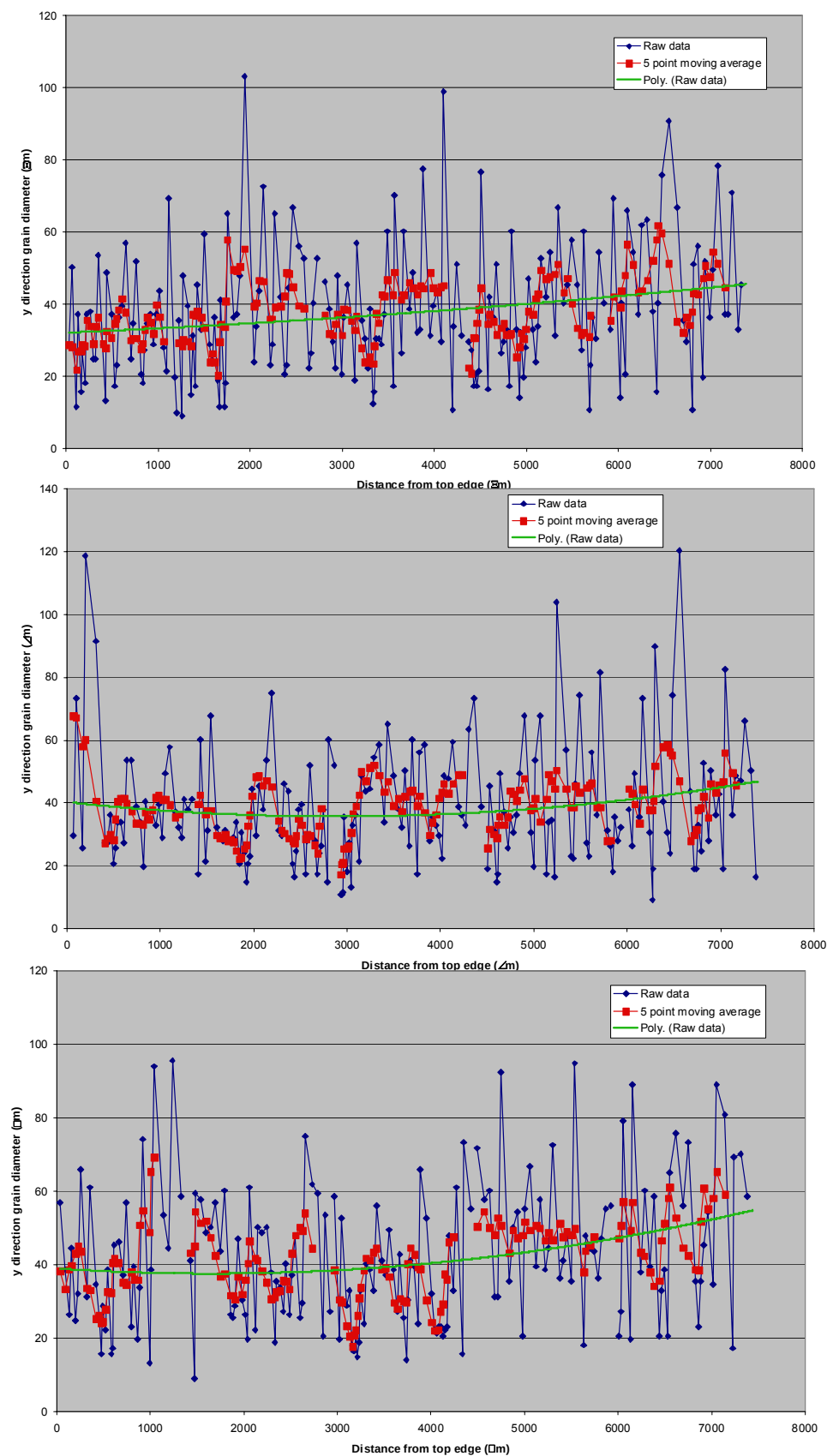


Figure C32 Centre line grain diameters for specimens A4: top to bottom (shoulder, L/2, L).

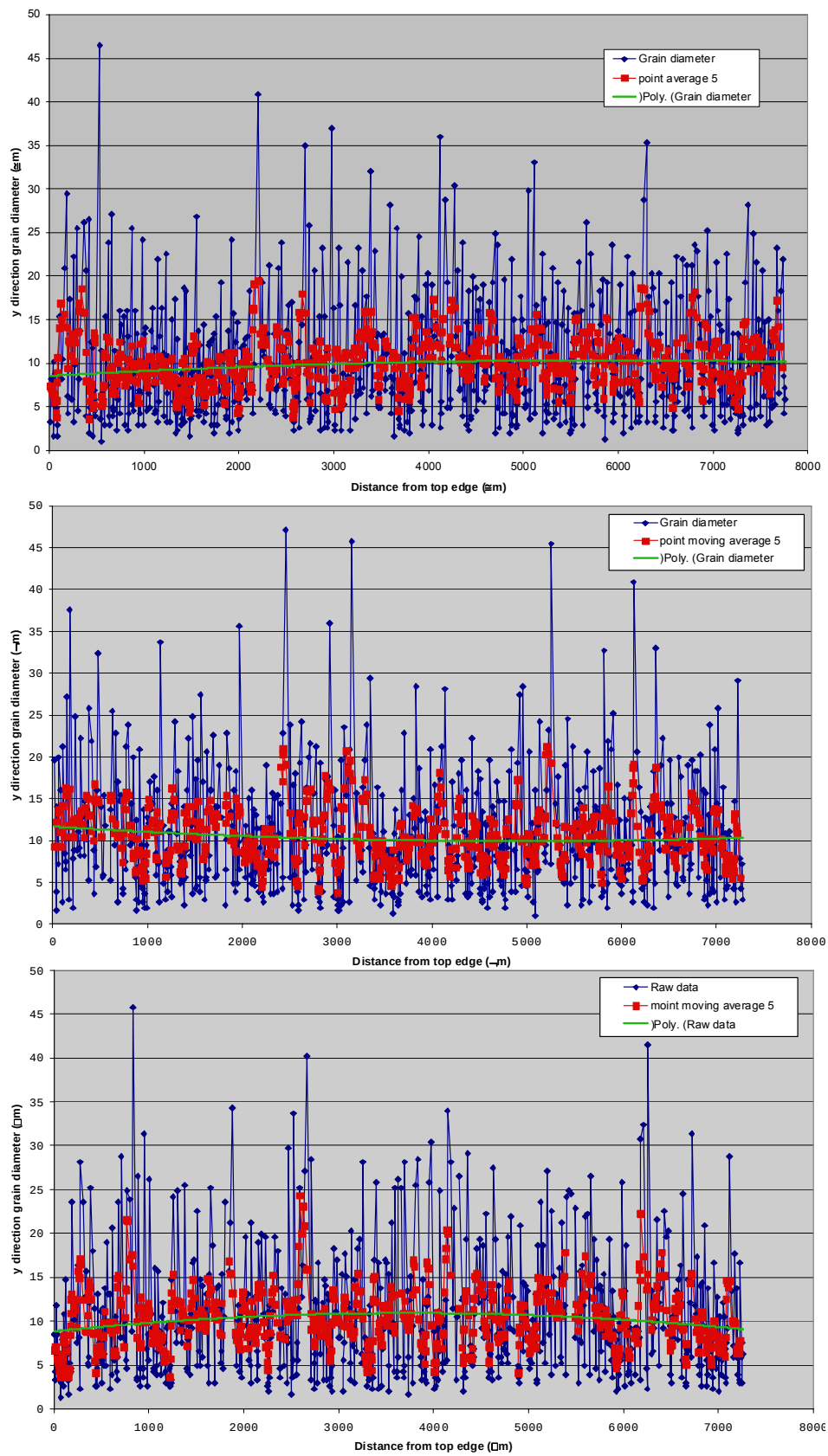


Figure C33 Centre line grain diameters for specimens S1: top to bottom (shoulder, L/2, L).

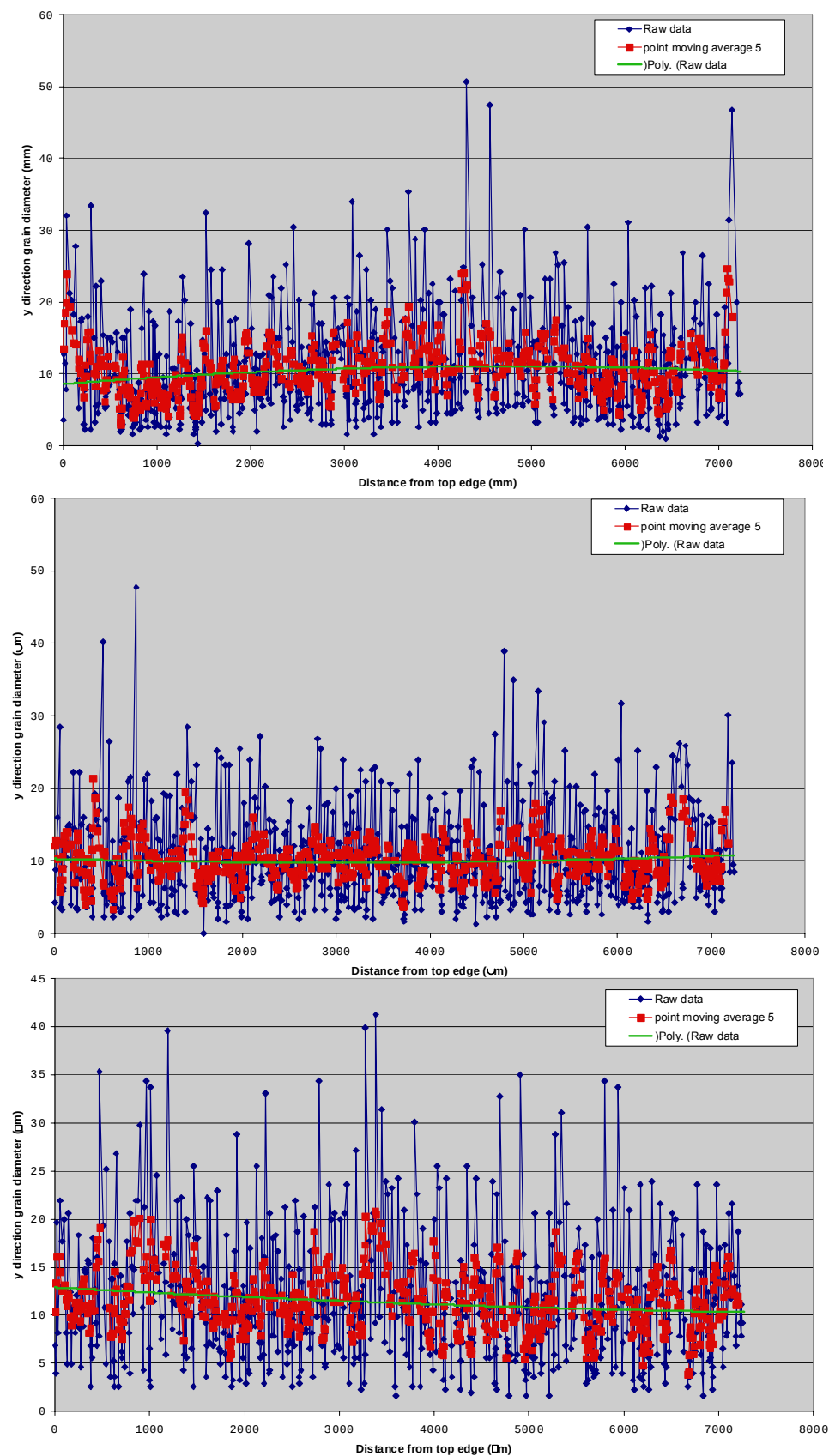


Figure C34 Centre line grain diameters for specimens S2: top to bottom (shoulder, L/2, L).

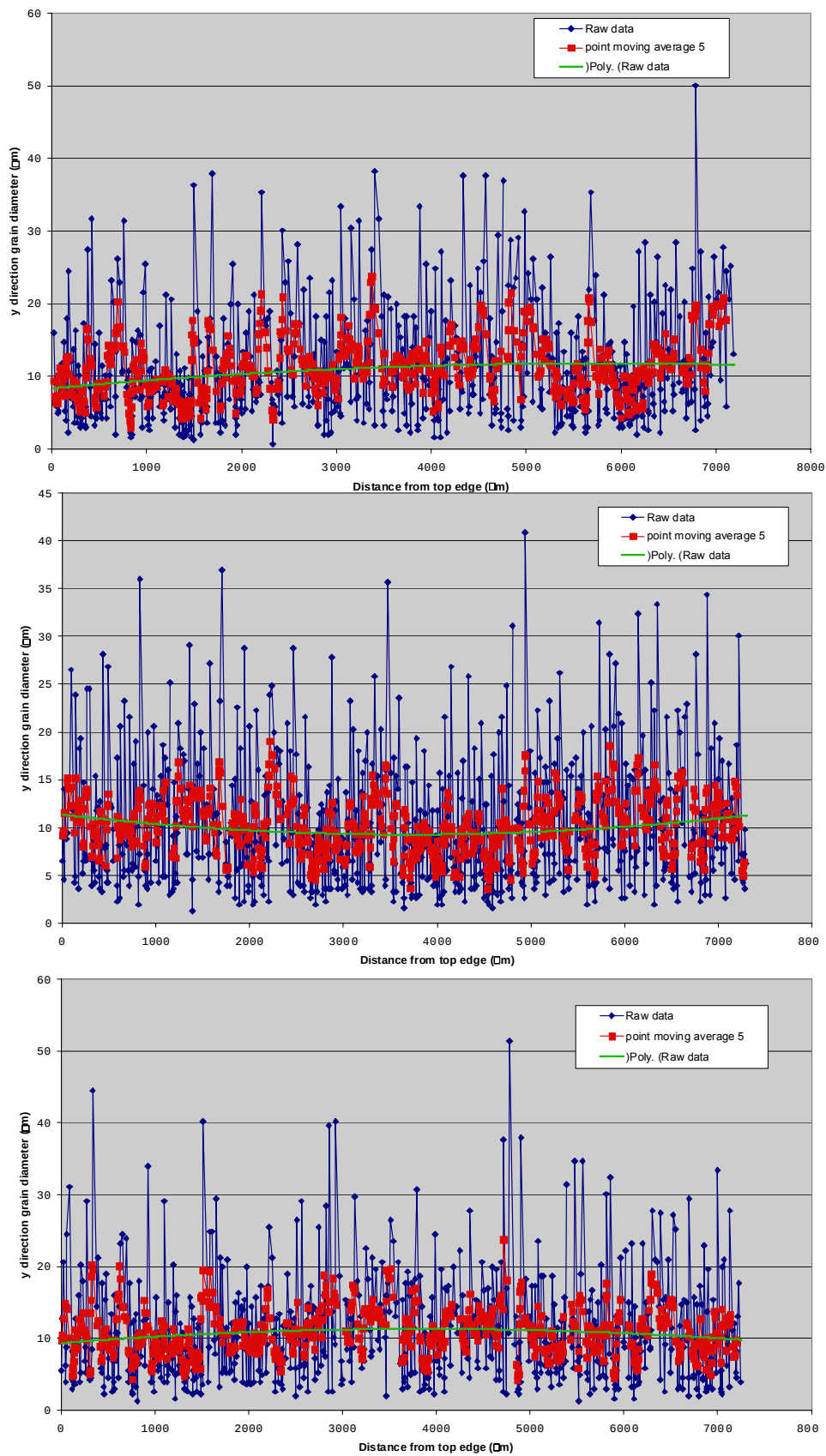


Figure C35 Centre line grain diameters for specimens S3: top to bottom (shoulder, L/2, L).

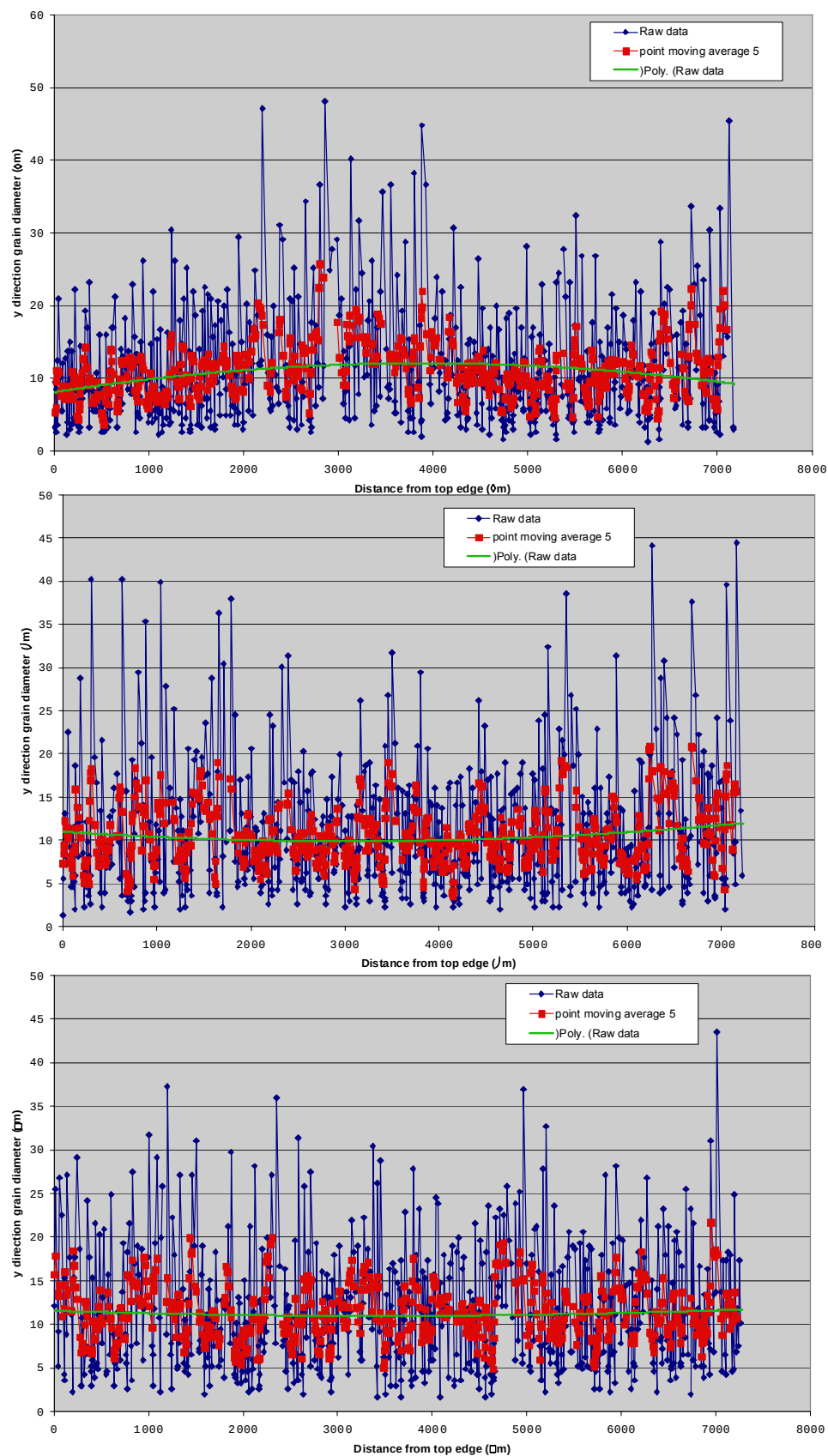


Figure C36 Centre line grain diameters for specimens S4: top to bottom (shoulder, L/2, L).

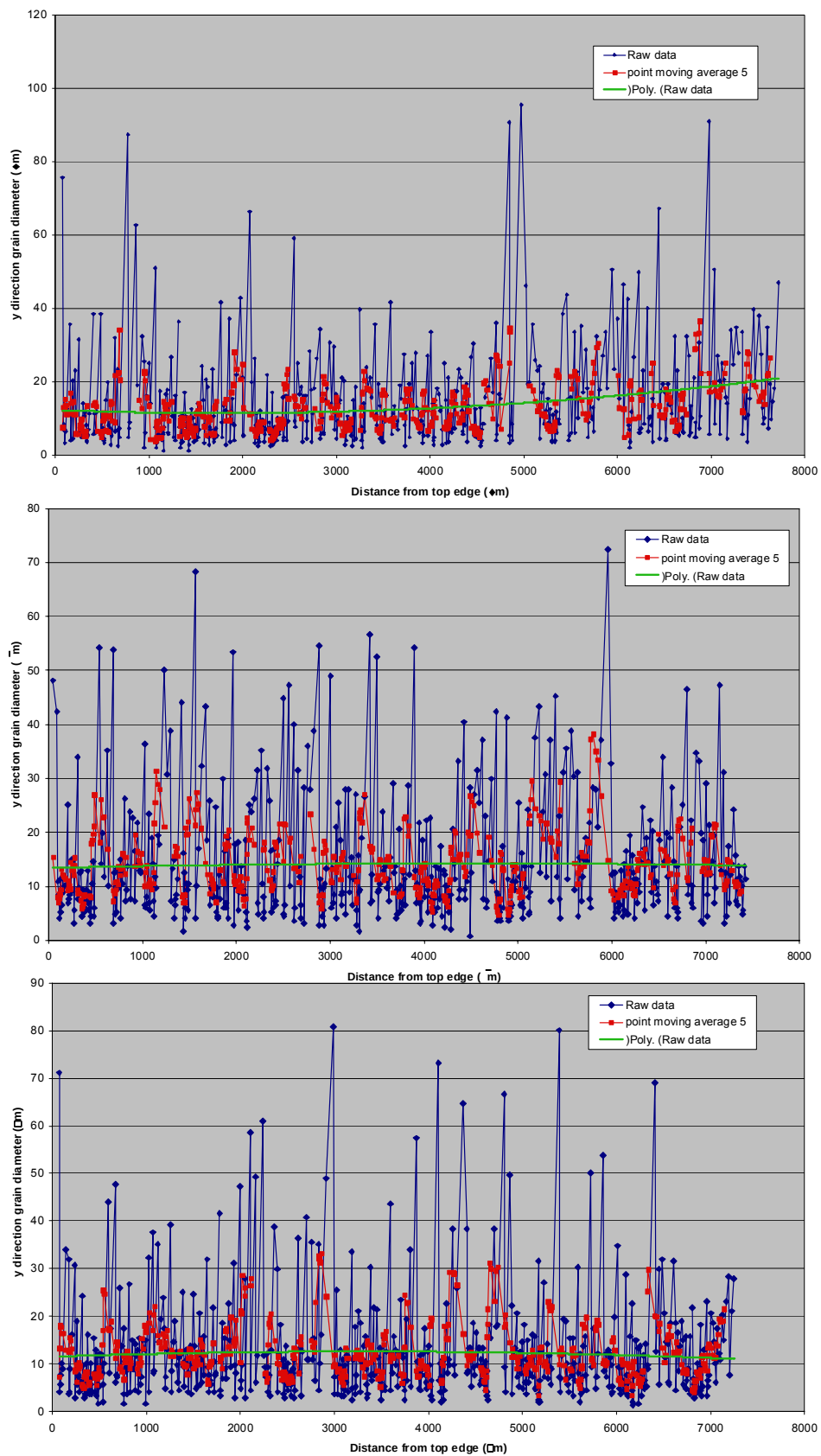


Figure C37 Centre line grain diameters for specimens W1: top to bottom (shoulder, L/2, L).

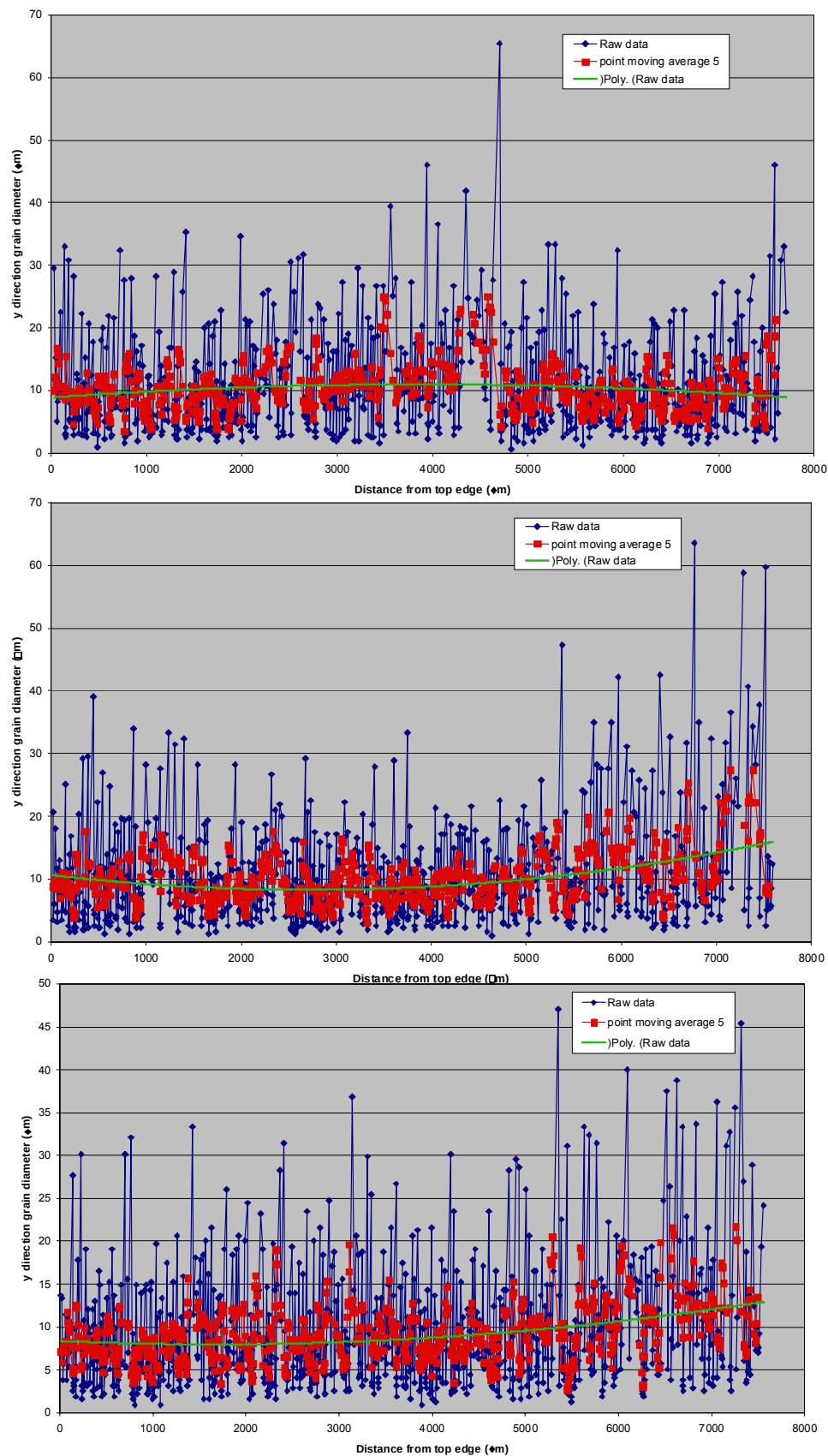


Figure C38 Centre line grain diameters for specimens W2: top to bottom (shoulder, L/2, L).

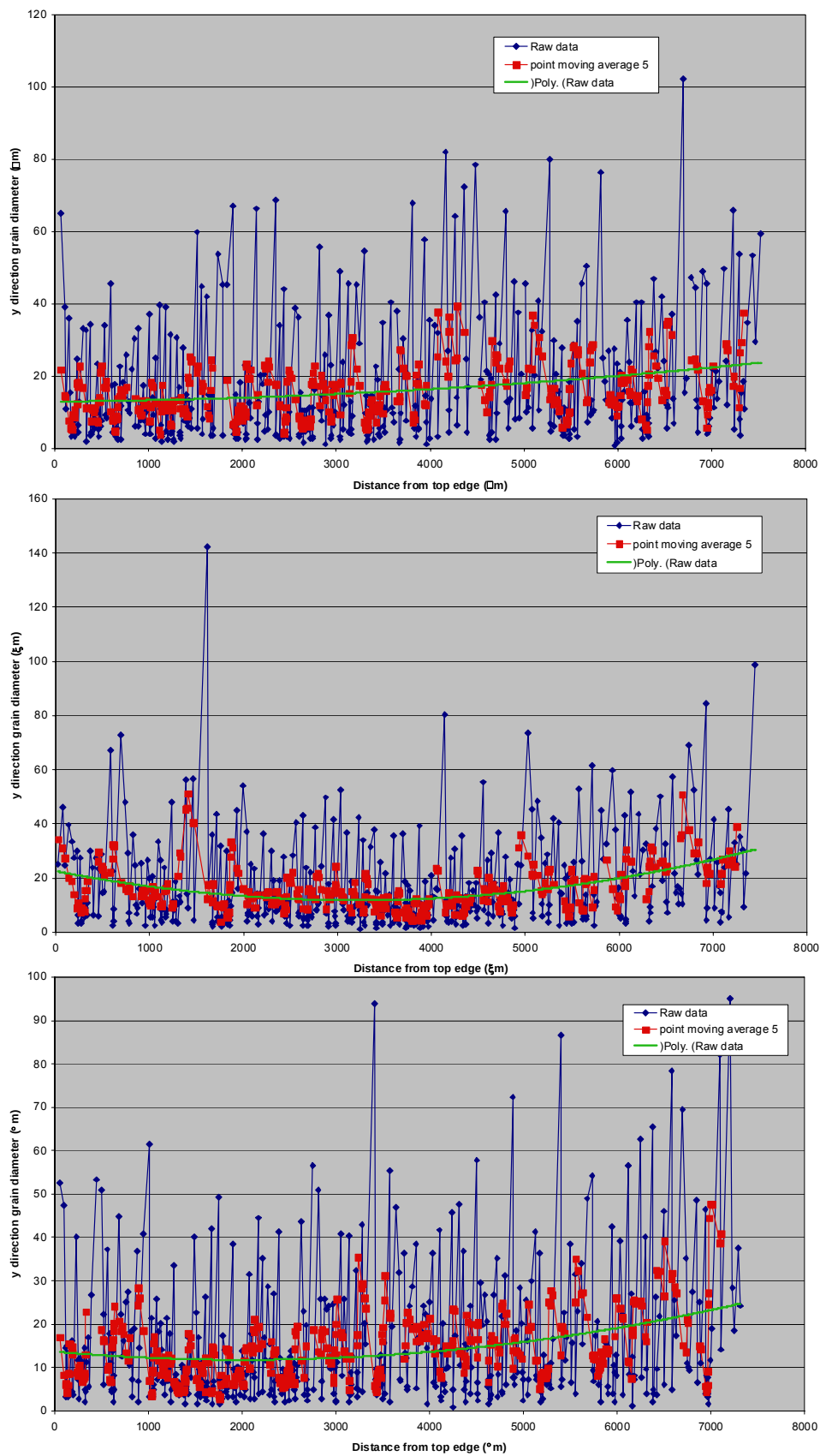


Figure C39 Centre line grain diameters for specimens W3: top to bottom (shoulder, L/2, L).

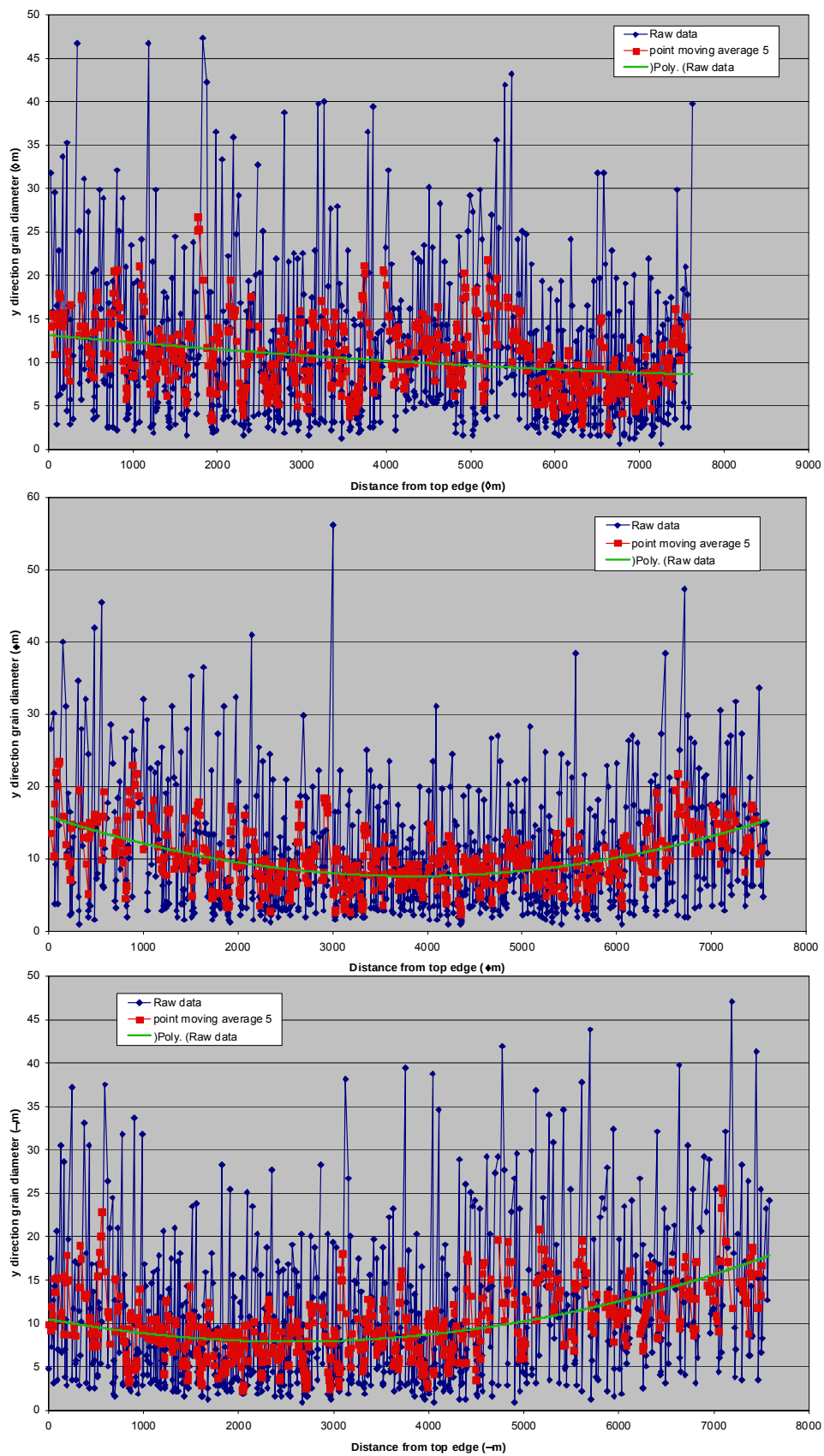


Figure C40 Centre line grain diameters for specimens W4: top to bottom (shoulder, L/2, L).

Appendix D

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IN THIS CHAPTER

- Scanning Microhardness System
- Microhardness Maps

APPENDIX D Scanning Microhardness Maps

Making detailed microstructural studies over large surface areas of metallographic samples is costly in time and effort. To provide some insight into likely variations in microstructure a new, rapid, method of scanning the surface was applied before undertaking extensive metallography. This method was based on the use of a scanning microhardness test system.

The scanning microhardness measurements were acquired using a new system developed at NPL [D1,2]. The system uses the principle of depth sensing hardness, allowing the measurement process to be fully automated. A diamond indenter is applied to the surface of the testpiece and hardness is calculated from the load and the indentation depth. A raster of indents can be placed by computer control to map the variation in hardness. Local mechanical property information can be rapidly obtained from a measurement of microindentation hardness. The NPL instrument, the SIMM, has been constructed with a relatively high speed facility to map large areas quickly. Typically an array of 100×100 indents takes about 10 h, i.e. about 4 s/indent. Force and displacement are monitored continuously during the complete indentation cycle, which can typically be completed in 15 s. The machine currently operates in the micro to mesoscale range, 0.02-20 N, (approximately 2 gf-2 kgf), with a load resolution of 2 mN (0.2 gf) and a depth-sensing resolution of 20 nm, using a specially designed capacitance displacement sensor. The loading mechanism uses a piezoelectric actuator. A piezoelectric system has the potential to be quick, with a high resolution. However, the main drawback is a limited range of movement of around 60 μm , so the complete assembly is mounted on a motorised Z stage. The motor is driven to within a few micrometres of the surface, and the piezoelectric mechanism used to perform the indentation.

Conventional microhardness values are calculated using the standard Vickers formula

$$HV = 2P \sin \theta / d^2; HV = 1.8544P/d \quad (D1)$$

where P is the indent load in kgf and d is the corner to corner diameter of the indentation in mm and θ is the angle between the indenter face and the vertical ($136^\circ/2 = 68^\circ$). The depth of the indentation, h, is equal to $d/7$ ($d/2\sqrt{2} \tan \theta$). Therefore, expressing HV hardness as a function of indentation depth.

$$HV = P/26.42 h^2 \quad (D2)$$

Vickers microhardness is defined as the ratio of applied load to contact area between indenter and sample under load. The SIMM microhardness was determined using the principles outlined in the draft ISO standard for Instrumented Hardness Tests, ISO DIS 14577. The depth sensing hardness HV (SIMM) is defined by P_{max}/A (i.e. $2P/d^2$), where A is the projected area of contact, and this is calculated from

$$HV (\text{SIMM}) = F/(24.5 * h_c^2) = 0.93 HV \quad (D3)$$

where h_c is the indentation contact depth under load; for relatively high loads, generally above about 2 N for metallic materials, where small discrepancies in the diamond indenter geometry are not a problem. For loads less than about 1 N, small differences in the indenter tip geometry can give rise to significant discrepancies with regard to h_c , compared with what

would be expected theoretically, and these discrepancies generally have to be calibrated by experiment. In these circumstances the nominal SIMM HVs can be significantly larger than the conventional microhardness HV value.

Scanning Microhardness Measurements

Typical SIMM maps from sectioned axisymmetric compression testpieces of 316 stainless steel and Waspaloy are shown in Figs D1 and D2.

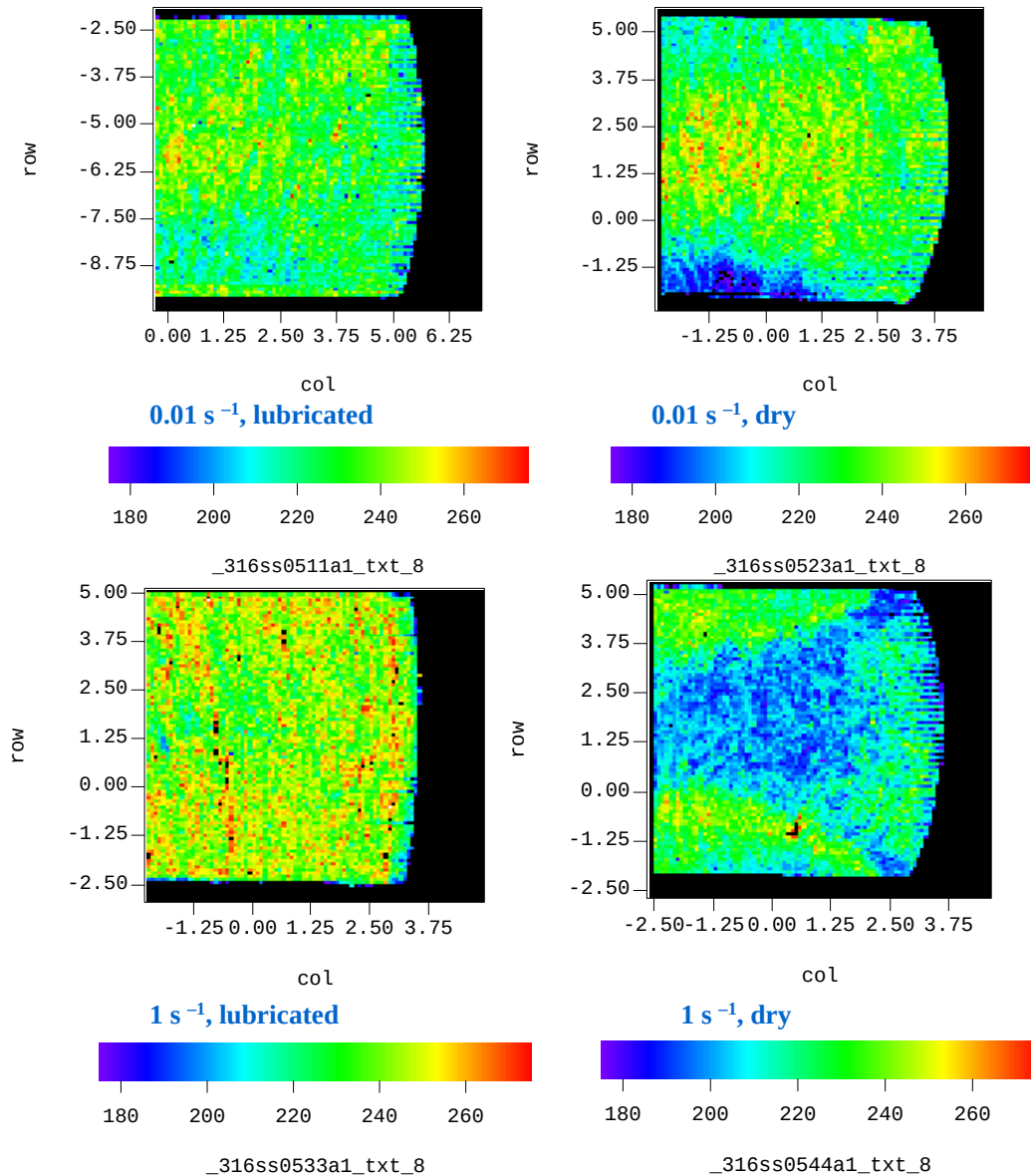


Figure D1 SIMM maps for tests on 316 stainless steel at 950 °C; 90 × 100 array, 2N, 0.03 mm diameter indents, 0.08 mm spacing

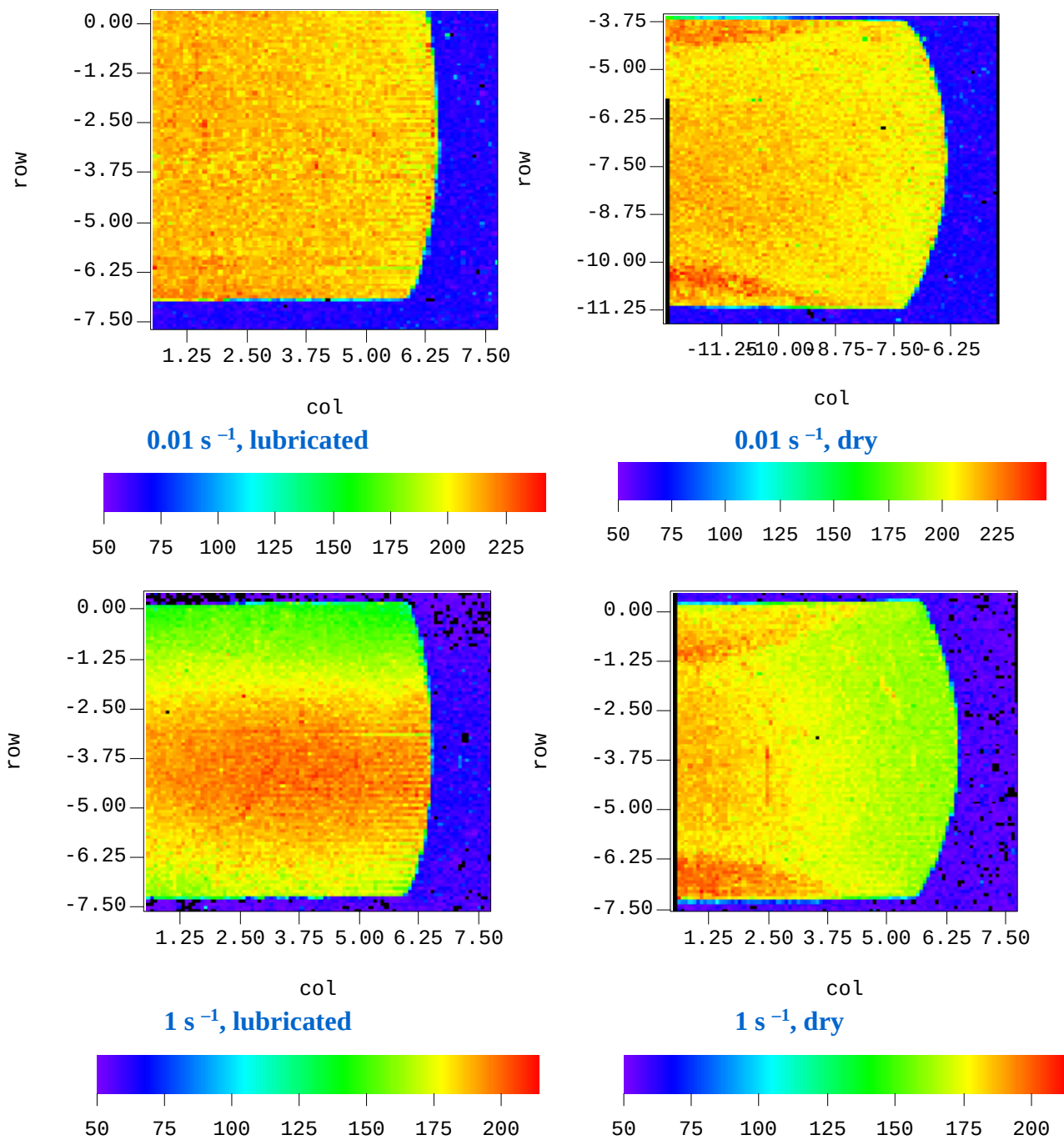


Figure D2 SIMM maps for tests on Waspaloy at 1040 °C; 90 × 100 array, 2N, 0.03 mm diameter indents, 0.08 mm spacing

The zones associated with constraint by the platens are clearly seen in the high rate tests with no lubrication and compare very well with FE model predictions of temperature and effective strain variation (Fig D3).

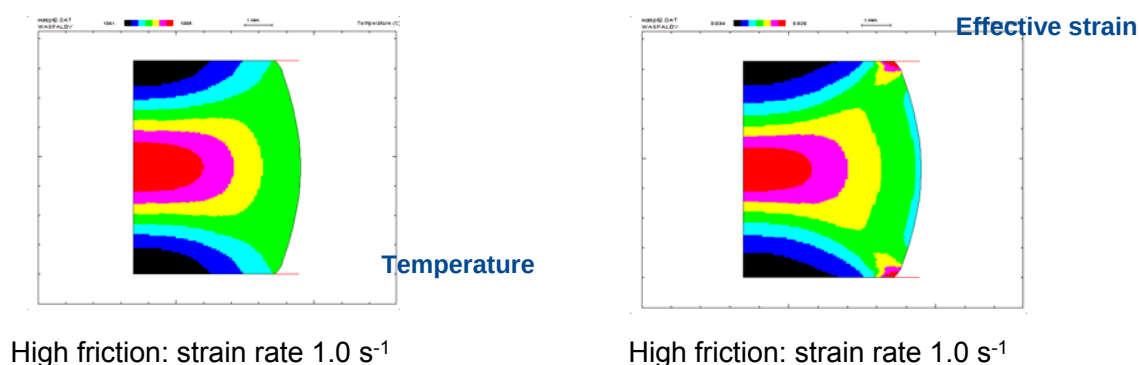


Figure D3 FE model predictions of temperature and effective strain variations in high friction, high strain rate tests on Waspaloy

The hardness array maps can thus be effectively used to give insight on where to concentrate for the microstructure measurements within the testpieces. Typical line plots of averaged values of hardness are shown in Fig D4 for the 316 stainless steel testpieces.

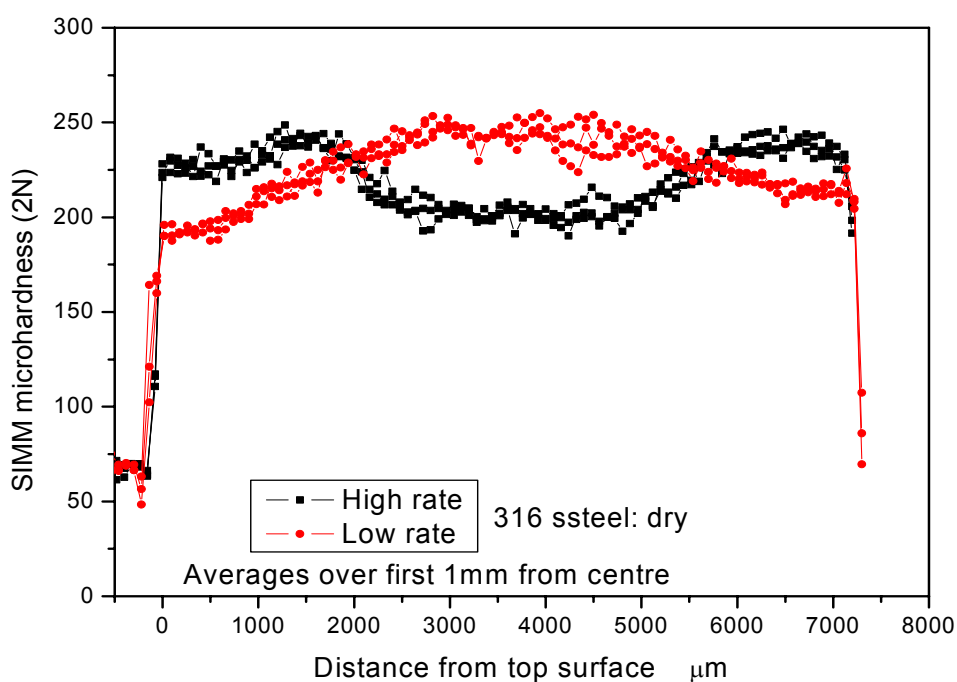


Figure D4 Line plots of microhardness for the 316 stainless steel axisymmetric compression testpiece

Similar measurements can be made on the PSC testpieces (Fig D5) and the maps show obvious shear bands corresponding with FE model predictions.

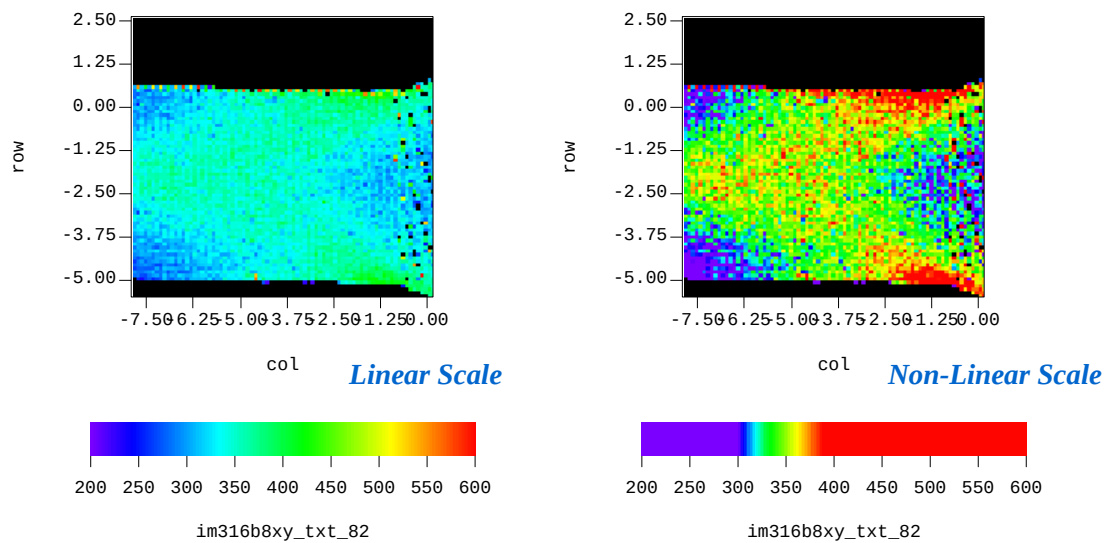


Figure D5 Typical hardness map from 316 stainless steel sample deformed dry at 950 °C and a strain rate of 0.01 s^{-1} ; 80×80 array, 2N, 0.03 mm diameter indents, 0.1 mm spacing.

References

- D1 B Roebuck, M Stewart, R Morrell, M G Gee and G Plint. Mapping Hardness Using Automated Depth Sensing Process. *Surface Engineering*, 2001, 17(6), 447-449.
- D2 B Roebuck, K P Mingard, M Brooks, E J Palmiere, B P Wynne and M J Thomas. Mapping Microstructure in AC and PSC Testpieces Deformed at High Temperatures Eds M Pietrzak, J Kusiak and D Szeliga, University of Krakow, 2007. *Kom Plas Tech* 2007, Proceedings of VIF Workshop, Zakopane, Poland, January 2007; *Computer Methods in Materials Science*, 7(4), 2007, 406-415.

