Microscopy & Microtechniques Focus

A POTENTIAL STANDARD FOR EBSD

Mike Matthews, Austin Day & Peter Quested

The RMS Electron Backscatter Diffraction Meeting (26 - 28 March) was held at the New Lanark Mill, Scotland

New Lanark Mill is a World Heritage Site and the meeting attracted support from the European Microscopy Society. The meeting provided a forum for EBSD users old and new to discuss the most recent ideas and innovations in EBSD and related techniques. This year was the largest meeting yet with registrations up 20% and delegates from all over Europe.

The developments in Electron Back Scattered Diffraction since the 1980s has led to commercial equipment for obtaining large amounts of data automatically. This has enabled Industrial users to become interested in adopting the technique for a wide range of applications such as grain sizing; simultaneous EDS and EBSD analysis for characterisation of particles and attempts at measuring the consequences of strain after deformation. As the technique moves from an academic environment there is a need for standards which enable an industrial laboratory to quickly perform the technique reliably and achieve accreditation in the area."

"It is hoped that the new standard will provide the information for laboratories to quickly and reliably perform EBSD and form the basis of accreditation for laboratories."

Peter Quested has worked at the National Physical Laboratory for many years. Amongst his interests are the characterisation of nickel base superalloys for high temperature applications and during his career he has applied a number of techniques. He first became interested in EBSD in the mid 1980s as a method for characterising single crystal superalloys and attempted to characterise the effects of creep strain on EBSD Patterns. Recently he was asked to help with the UK contribution to the standard through BS CII/9 and TC202. The poster was intended to build bridges between the academic community and the industrial users.

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WHAT IS A STANDARD?

A standard is an agreed, repeatable way of doing something. It is a published document that contains a technical specification, test method or other precise criteria designed to be used consistently as a rule, guideline, or definition. They are intended to be aspirational - a summary of good and best practice rather than general practice. Standards are created by bringing together the experience and expertise of all interested parties such as the producers, sellers, buyers, users and regulators of a particular material, product, process or service.

WHAT ARE THE BENEFITS OF STANDARDS?

- Attract and assure customers (an integral part of third party accreditation.)
- Demonstrate market leadership
- Create competitive advantage
- Develop and maintain best practice.

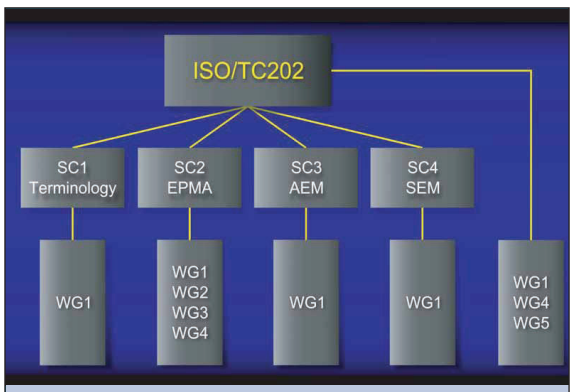
Source: http://www.bsi-global.com/en/Standards/-and-Publications/About-standards/

WHY A STANDARD FOR EBSD?

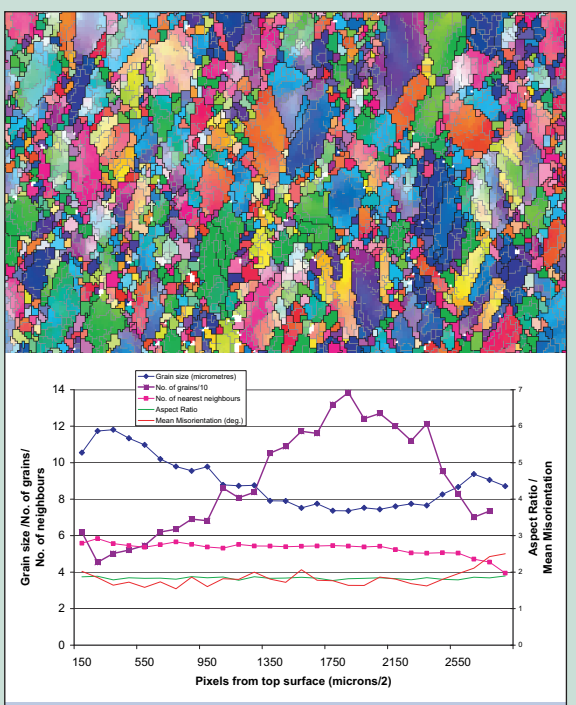
A number of presentations at this meeting are about characterising engineering materials for industrial customers. If Industry adopts EBSD for characterising their materials, it needs assurance that testing performed by different suppliers gives comparable results. This leads to third party peer review, such as, ISO 17025 for the accreditation of laboratories. Having a standard for EBSD means that ISO 17025 is more easily obtained in this area.

ISO/TC 202 WHAT'S IT ABOUT?

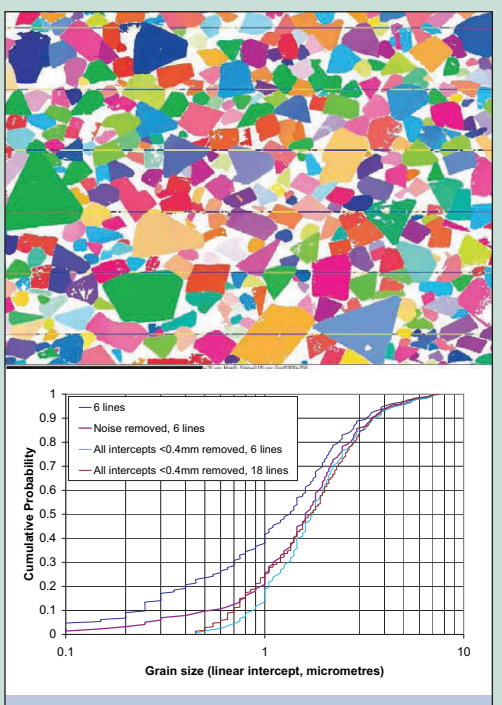
1) To define terms in microbeam analysis.
2) To detail requirements for:
   a. Measurement of instrumentation parameters and specifications.
   b. Operational methods
   c. Specimen preparation
   d. Data acquisition
   e. Data processing
   f. Qualitative and quantitative analysis
   g. Reporting results
3) To provide standardisation procedures, which may specify reference materials and to recommend data management procedures which allow precise, accurate and repeatable microbeam analysis to be accomplished.



Structure of TC202



Recrystallisation



Grain size in Hardmetals

Examples of industrial applications (ex Ken Mingard, NPL)

Who's involved in TC202?

Participating (P) Members
13 Countries
EMAS

Observer (O) Members
13 Countries

**UK uses BS Committee CII/9 for their input*

Table 1: Published ISO Standards Produced by TC202

BS ISO 15632:2002	Microbeam analysis. Instrumental specification for energy dispersive X-ray spectrometers with semiconductor detectors
BS ISO 14594:2003	Microbeam analysis. Electron probe microanalysis. Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy
BS ISO 14595:2003	Microbeam analysis. Electron probe microanalysis. Guidelines for the specification of certified reference materials (CRMs)
BS ISO 22029:2003	Microbeam Analysis: Standard file format for spectral data exchange
BS ISO 16700:2004	Microbeam analysis. Scanning electron microscopy. Guidelines for calibrating image magnification
BS ISO 17470:2004	Microbeam analysis. Electron probe microanalysis. Guidelines for qualitative point analysis by wavelength dispersive X-ray spectrometry
BS ISO 22309:2006	Microbeam analysis. Quantitative analysis using energy-dispersive spectrometry (EDS)
BS ISO 16592:2006	Microbeam analysis: Electron probe microanalysis. Guidelines for determining the carbon content of steels using a calibration curve method

Table 2: ISO Standards Under Preparation Working Drafts or Committee Drafts

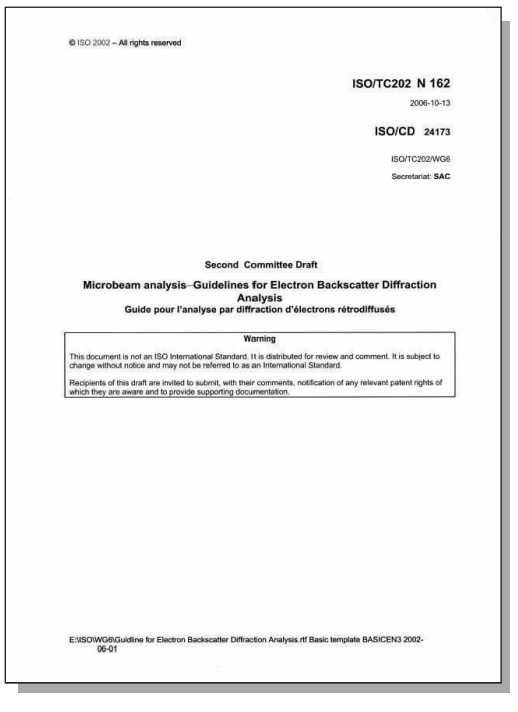
ISO 22489	Microbeam analysis: Electron probe microanalysis. Quantitative point analysis for bulk specimens using wavelength dispersive X-ray spectrometry
ISO 22493	Microbeam analysis: SEM Vocabulary
ISO 23833	Microbeam analysis: Electron probe microanalysis (EPMA). Vocabulary
ISO 24173	Microbeam analysis: Guideline for electron backscatter diffraction analysis
ISO 24597	Microbeam analysis: Scanning electron microscopy. Measurement methods of image resolution
ISO 25498	Method of selected area electron diffraction for transmission electron microscopy

CONTENTS OF ISO 24173 MICROPROBE ANALYSIS: GUIDELINE FOR ELECTRON BACKSCATTER DIFFRACTION ANALYSIS

Second Committee draft (2nd CD)

- Foreword
- Introduction
  1. Scope
  2. Normative references
  3. Terms and defi nitions
  4. Principle
  5. Data representation
  6. Equipment for EBSD
  7. Calibration
  8. Sample preparation
  9. Operating conditions
  10. Analysis Procedure
  11.Measurement Uncertainty
  12. Issuing of analysis results
- Annexes (Informative)
  A.Sample preparation
  B.Estimates of measurement uncertainty
  C.Applications
  D.Simple crystallography
- Bibliography

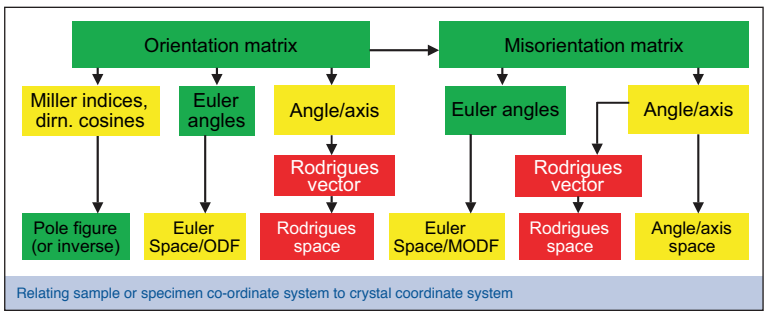
The convenor is an industrialist Dr Jiaguang Chen, Bao Steel, China



ISSUES ABOUT STANDARD EBSD STANDARD

The scope

Should the standard only cover how to obtain basic orientation data or be extended to how you use that data e.g. grain orientation maps and grain size?



Based upon figure 2.1, V Randle and O Engler "Introduction to Texture Analysis Macrotexture, Microtexture and Orientation Mapping" Gordon and Breach Science Publishers, 2000, ISBN 90-5699-224-4

Calibration

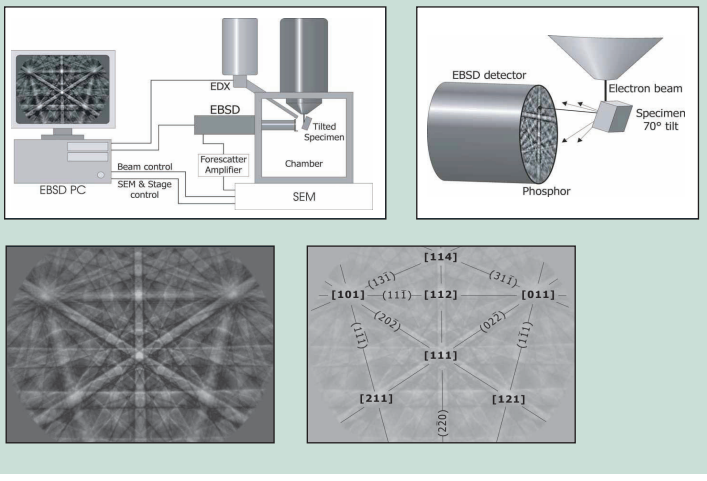
Is it acceptable to rely on automatic calibration of Pattern Centre and Detector distance?
Do the more traditional ways of checking these parameters need to be included?

Reference materials

Validation of calibration; grain size and texture?
How to handle distortions in the image?
Challenges associated with Sample drift

OTHER STANDARDS ASTM

WK3577 New Guide for Grain Size by Electron Backscatter Diffraction (EBSD) Developed by Subcommittee: ATSM E4.11 Technical
Contact: Tom Murphy and John Friel



NEXT STEPS

- Prepare the 3rd Committee Draft (CD) from comments.
- Ballot 3rd CD version and obtain votes and comments.
- Prepare Draft International Standard (DIS)
- Vote on DIS
- Circulate Final Draft International Standard (FDIS) for final approval
- International standard published and available

About the Authors

Mike has been involved in the microanalysis field, primarily EPMA and SEM, for over twenty years, working with metals, ceramics, glasses and composite materials. He has been active on the ISO/TC202 committees for over ten years, and since 1997 has been Convenor of WG1, the working group on general procedures. Mike is also actively involved in EMAS, the European Microbeam Analysis Society, holding the post of Editor for the society's newsletter since 2001.

Austin has worked in EBSD for twenty years - starting at Bristol University, then working at the National Physical Laboratory and in Industry. He has designed & built a wide range of EBSD detectors and software and is currently working as an independent EBSD consultant.