INTERNATIONAL STANDARDS AND INTERCOMPARISONS FOR EBSD ANALYSIS OF MICROSTRUCTURE

K.P.Mingard, P.N.Quested, M.S.Peck, GD Sims and J G Cabañas Moreno[§]

National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, UK, [§] Centro de Investigación y de Estudios Avanzados, CINVESTAV, Mexico

Why have standards for EBSD?

Many product standards and specifications are based on microstructural characteristics such as grain size or texture.

EBSD is increasingly replacing other methods for measurement of these characteristics because it produces:

- improved spatial resolution,
- crystallographic data
- automated procedures
- analysis of much greater areas than manual methods

What Standards Exist already?

Grain size standards have been developed for simple single phase materials only and do not yet include uncertainty data:

- ISO 24173:2009 - Guidelines for orientation measurement using EBSD
- ISO 13067:2011 - Measurement of Average Grain Size
- ASTM E2627-13 - Determining Average Grain Size in Fully Recrystallized Polycrystalline Materials

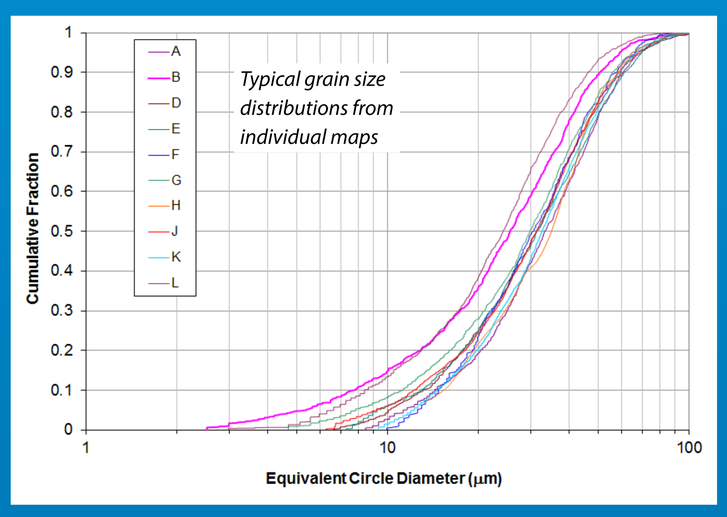
Grain Size Measurement Intercomparison

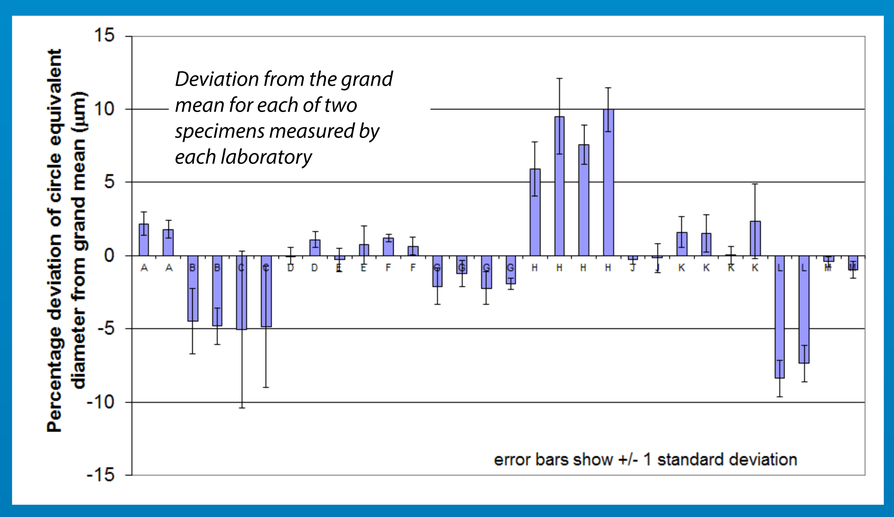
EBSD Grain size Measurement Intercomparison

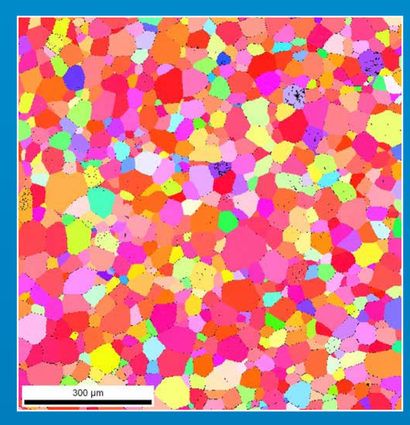
- The VAMAS (Versailles Project on Advanced Materials and Standards, technical working area **TWA 37, Quantitative Microstructural Analysis**, is tasked with assessment of procedures which include EBSD
- TWA37 organised an initial grain size intercomparison using equiaxed CP Titanium - a commercial product which can also be measured optically as well.
- 2 samples measured by 12 laboratories in 8 countries, with 4 different EBSD systems.
- Laboratories included equipment manufacturers, industrial users and National Metrology Laboratories (NMIs)
- Laboratories asked to follow ISO13067

Results

- 8 of the 12 labs are within +/-5% of the mean values (circle equivalent diameter **Dceq** shown here)
- Laboratories close to mean also have smaller spreads between maps for the same sample
- Step sizes chosen varied from 1 to 3 µm
- Grains/ map varied from ≈70 to >600
- Dceq ≈ 10% greater than Dlin
- Reasons for the smaller average grain sizes outliers include
  - Cut off value for smallest grains
  - Differences in Noise/ data cleaning







Typical EBSD map from intercomparison. Inverse pole figure colouring, scale bar = 0.5 mm

Implications for Revision of Grain Size Standards

1. Emphasise grain size distributionmeasurement
2. Include Reproducability and Repeatablilty data
3. Reduce requirement for well indexed pixels from 95 to 80% BUT
4. Increase emphasis on choice of minimum size cut off in µm
5. Increase minimum number of grains analysed to >200

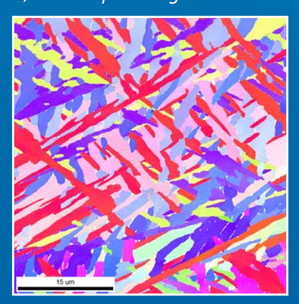
For full results and recommendations see:
National Physical Laboratory Report MAT 56, ISSN 1754-2979

Future Standards

The intercomparison above considered one single, simple material grade. EBSD is used to measure much more complex alloys, so uncertainties are likely to increase with the following examples.

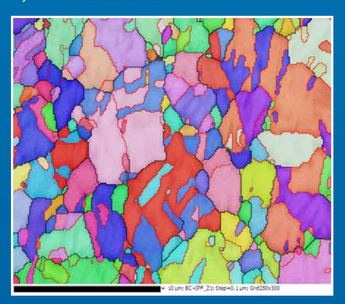
With input from end users and EBSD practitioners, TWA37 needs to prioritise intercomparison work by selection from materials such as the following:

a) Non –equiaxed grain size



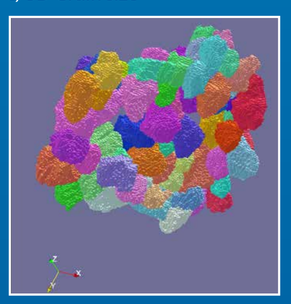
α-Titanium
How do you report size for needle-like grains

b) Finer Grain size



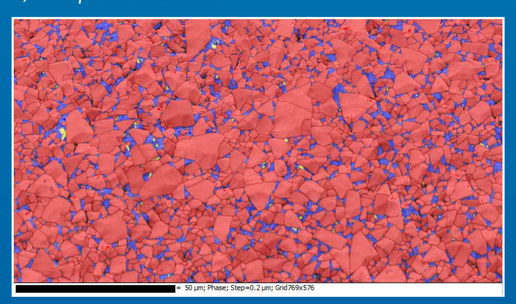
Electrodeposited Cu
Do errors increase with much smaller grain sizes ≈ 1 µm Does twinning increase errors.

c) 3D Grain size



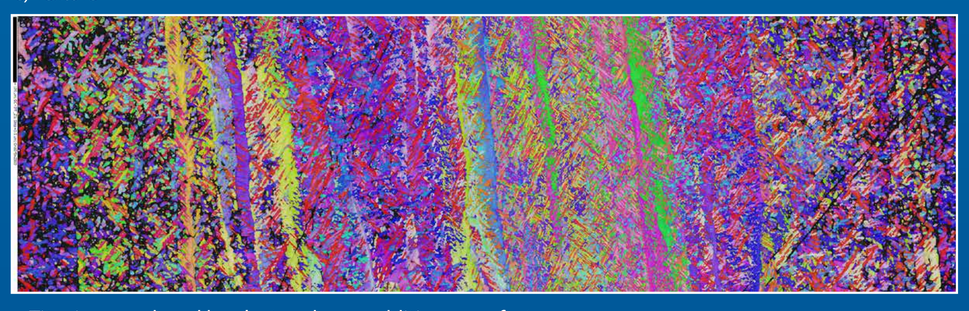
WC Grains
In the long term, how do 2D measurements compare with 3D grain sizes?

d) Multiphase Materials

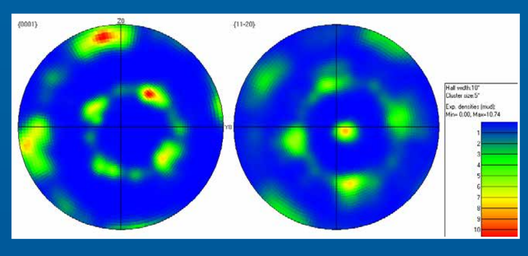


WC (red) in Co matrix (blue –fcc, yellow hcp)
How accurately are sizes and volume fractions reported for multiphase materials?

e) Texture



α-Titanium produced by electron beam additive manufacture.
How accurately is texture measured by EBSD?



For more details, suggestions, comments and offers to participate in future intercomparison work, please contact
Ken Mingard *at the* ***National Physical Laboratory****,*
Hampton Road, Teddington, Middlesex TW11 0LW, UK.
e: **ken.mingard@npl.co.uk**
t: **+44 20 8943 6558**

Acknowledgements*: Adam Creuziger, NIST, Clive Oliphant, NMISA, Daniel Goran, Bruker, David Wilcox, Tata Steel, Fabrice Gaslain, Centre des Materiaux Mines Paristech, Joacim Hagstrom, Swerea KIMAB AB, Mark Vaudin, NIST, Matt Nowell, TSL EDAX, Sascha Dieter, BAM, Singh Ubhi, Oxford Instruments, Stuart Wright, TSL EDAX, Taeko Fukuhara, INMETRO, Yang Ping, University of Science and Technology, Beijing*

FUNDED BY BEIS

www.npl.co.uk