EBSD in steels characterization

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Presentation overview

- Introduction
- Theory
- EBSDs at the University of Oulu
- Sample preparation
- Examples of EBSD scans
- Conclusions
Introduction to EBSD: Electron Back Scatter Diffraction

- EBSD is automated collection and indexing of electron diffraction (Kikuchi) patterns from bulk samples in the SEM
- General microstructural characterization technique
- EBSD provides information about:
  - Crystal lattice
  - Discrimination between phases based on their crystallographic differences (e.g. austenite (FCC) and ferrite (BCC) in steels)
  - Surface, information from 10 – 50 nm thick layer
- EBSD can not:
  - Analyze non-crystalline materials (e.g. glass, wood, ...)
  - Provide information within the volume of a sample – it is purely a surface technique
  - Analyze samples with thick coats (e.g. > 10nm thick)
  - Discriminate phases with similar crystallography without EDX info
- Before going to EBSD one should know:
  - Phase / phases
  - Grain size
  - Purpose of the study
EBSD Setup

Typical measurements

- Misorientation data
- Grain size
- Boundary properties (twin boundaries, CSL…)
- Phase identification
- Phase distribution
- Image quality
- Texture
- Recrystallised / deformed fraction
- Intra-granular deformation
- …
EBSDs at the UO

- Accelerating voltage: 0.02V - 30 kV
- Resolution: 1.0 nm/15 kV, 1.7 nm/1 kV
- EDS, C-U
- EBSD (Oxford Instrument HKL, AZtecHKL)

- Accelerating voltage: 0.1 - 30 kV
- Resolution: 1.5 nm/15 kV, 2.8 nm/1 kV
- EDS, C-U (EDAX, Apollo X)
- EBSD (EDAX, Hikari XP, EDAX, TSL OIM Data Analysis)
(Stainless) steel sample preparation:

1) Polishing with diamond suspension down to 1 \( \mu m \)
2) Chemical polishing using a 0.05 \( \mu m \) colloidal silica suspension

Limitation:
- Metastability of the steels

EBSD phase map of a) mechanically polished sample and b) electropolished sample; Ferrite is shown in blue (bcc) and austenite in red (fcc).

Austenitic-ferritic stainless steel: Fe-0.02%C-19.2%Cr-0.5%Ni-3.1%Mn-0.5%Si-0.6%Cu-0.2%N (wt-%).

Crystal orientation

(a) Image quality and grain boundary and b) inverse pole figure maps of deformed Fe-18Mn-8Al-0.8C (wt-%) steel specimen at 1100 °C under strain rate of 0.1 s⁻¹ up to the strain of 0.6; white arrows in b) indicate the annealing twins, ferrite is marked as F (bcc) and austenite as A (fcc).

Grain size determination

Inverse pole figure and grain boundary maps after cold rolling and annealing at 1100 °C for reversion treated austenitic low-Ni high-Mn stainless steels alloyed with a) 0Nb, b) 0.11Nb, c) 0.28Nb and d) 0.45Nb; the black and green coloured boundaries have misorientation higher than 15° and 3°.

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Average grain size vs annealing duration of the reversion treated austenitic low-Ni high-Mn stainless steels alloyed with 0Nb - 0.45Nb, annealed at a) 900 °C, b) 1000 °C and c) 1100 °C.

Combined microstructure and grain size

(a) Image quality map and (b) grain size distribution (taken from the coloured areas) of reversion treated austenitic low-Ni high-Mn stainless steel after annealing at 800 °C for 1000 s.

Band contrast and phases maps for reversion treated austenitic low-Ni high-Mn stainless steel after annealing at 1000 °C for 200 s and tensile strained a) 2%, b) 10% and c) 20%; austenite is shown in grey, ε-martensite in yellow and α’-martensite in red.

Multi-peaks analysis for 75% cold rolled ferritic EN1.4016 (AISI 430) stainless steel.

Band contrast EBSD map for 75% cold rolled ferritic EN1.4016 (AISI 430) stainless steel.

Co-operation with A. DeArdo, Basic Metals Processing Research Institute (BAMPRI), University of Pittsburgh
Texture analysis

Inverse pole figure map for 79% cold rolled ferritic EN1.4016 (AISI 430) stainless steel.

Combined EBSD and EDS

EBSD-Inverse pole figure map and associated EDS (EDS = Energy-Dispersive X-ray Spectroscopy) analysis of element distributions after deformation at 900 °C/0.01s-1/e=0.25: (a) analyzed region, (b) Cu distribution in green, (c) Ni distribution in gold and (d) Fe distribution in brown.

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- Crystallography
- Surface