Application of MTEX in steel research

Dr. Marco Witte

Salzgitter, 20th February 2016
MTEX in steel research

Overview

Introduction

Macro Texture Analysis (XRD & EBSD)
- Elasticity tensor
- Wave velocities and ultrasound
- Symmetry of pole figures

EBSD Analysis
- Homogeneity
- IQ Analysis
- Prior Austenite Grains

Summary
MTEX in steel research

Overview

Introduction

Macrow Texture Analysis (XRD & EBSD)
- Elasticity tensor
- Wave velocities and ultrasound
- Symmetry of pole figures

EBSD Analysis
- Homogeneity
- IQ Analysis
- Prior Austenite Grains

Summary
Introduction - Salzgitter Mannesmann Forschung

One of Europe’s leading research institutes in the steel sector
Central research company for steel activities in the Salzgitter Group

Salzgitter AG

Strip steel BU  Plate/section steel BU  Energy BU  Trading BU  Technology BU

SZMF Salzgitter  SZMF Duisburg

Two powerful locations with close thematic ties and cooperation
Direct connection to Salzgitter AG/CEO

SZMF is responsible for ensuring the innovation capability and innovation performance in the strip steel, plate/section steel and energy business units
MTEX in steel research

Introduction - SZMF: Concentrated expertise

300 employees develop the future of all aspects of steel materials – around 130 members of staff in Salzgitter and 170 in Duisburg

Scientific disciplines

- Mechanical Engineering and Process Engineering: 28%
- Material Science and Metallurgy: 19%
- Physics: 19%
- Chemistry: 13%
- Others: 12%
- Electrical Engineering and Computer Science: 6%
- Constructional Engineering: 3%

Total: 126 University Graduates
MTEX in steel research

My experience with MTEX

2009-2013

- PhD thesis: “Texture Optimization of Ni-5at.%W for Coated Conductor Applications” at Institut für Metallkunde und Metallphysik, RWTH Aachen
- Evaluation of very **sharp textures** measured with XRD on **non regular pole figure grids**.
- Project with F. Bachmann: “Development of an Adaptive pole figure measurement technique for sharp textures”
- MTEX made working with textures and orientations fun!
**MTEX in steel research**

**My experience with MTEX**

2013-now

- Evaluation of **XRD and EBSD** measurements to assist steel research and process optimization at Salzgitter Mannesmann Forschung.
- **Scripting** makes every day evaluation faster and easier.
- Approach: First evaluate “everything” and see later which information is useful.
- Automated creation of PDF reports
- MTEX still makes a lot of fun!
MTEX in steel research

Overview

Introduction

**Macro Texture Analysis (XRD & EBSD)**
- Elasticity tensor
- Wave velocities and ultrasound
- Symmetry of pole figures

**EBSD Analysis**
- Homogeneity
- IQ Analysis
- Prior Austenite Grains

**Summary**
MTEX in steel research

Macro Texture Analysis

- Pole figures, ODF plots, texture index, volume fractions of orientations of interest

- Texture fibres. For steel: $\alpha$-fibre ($\{110\} \parallel RD$) & $\gamma$-fibre ($\{111\} \parallel ND$)
Comparison with Young’s modulus from tensile tests shows **good correlation**.

Results from texture measurement too high as single crystal **tensor from literature** is for pure iron.
Can textures be measured with ultrasound (online process control)?

How does texture composition (γ-fibre & α-fibre) affect wave velocities in certain directions?

Approach: Calculate the wave velocities for different compositions of fibre textures.
% crystal symmetry
CS = crystalSymmetry('m-3m');
% specimen symmetry
SS = specimenSymmetry('-1');

% kernel for odfs
psi = deLaValee PoussinKernel('HALFWIDTH',7*degree);

% gamma fibre odf
odf_gamma = fibreODF(Miller(1,1,1,CS),zvector,psi);

% alpha fibre odf
odf_alpha = fibreODF(Miller(1,1,0,CS),xvector,psi);

% fibre fractions to be calculated
x = 0:0.1:1;

% to save velocities
vs1 = cell(1,length(x));
for i = 1:length(x)
    % sum of fibre odfs
    odf = x(i)*odf_gamma + (1-x(i))*odf_alpha;
    % elasticity tensor of ferrite from MPOD
    Ein_krist = [[231.4 134.7 134.7 0 0 0];...
                  [134.7 231.4 134.7 0 0 0];...
                  [134.7 134.7 231.4 0 0 0];...
                  [0 0 0 116.4 0 0];...
                  [0 0 0 0 116.4 0];...
                  [0 0 0 0 0 116.4]];
    T = tensor(Ein_krist, CS,'name','elastic stiffness','unit','GPa');

% weight tensor with texture
Tmean = calcTensor(odf,T);

% density in g/cm^3
rho = 7.8;

% ultrasound measurement points of s-waves
polar_angle = -35*degree;
azimuth_angle = 0:15:90;
v_shear = vector3d('polar',polar_angle,azimuth_angle*degree);

% get s-wave velocities
[vp{i},vs1{i}] = velocity(Tmean,v_shear,rho);

% plot s-wave tensor
figure
plot(Tmean,'PlotType','velocity','vs1','density',rho,'complete','upper')
ax = colorbar;
xlabel(ax,'km/s')
saveFigure(['Gamma_' num2str(x(i),2) '_Elast_Welle_vs1.png'])
close
end
Texture Analysis - Elastic Wave Velocities

0% γ -fibre & 100 % α -fibre

ODF

p-wave velocity

s-wave velocity
MTEX in steel research

Macro Texture Analysis - Elastic Wave Velocities

50% $\gamma$ -fibre & 50% $\alpha$ -fibre

ODF

p-wave velocity

s-wave velocity
Macro Texture Analysis - Elastic Wave Velocities

100% $\gamma$-fibre & 0 % $\alpha$-fibre

- ODF
- p-wave velocity
- s-wave velocity
Distribution of pressure wave velocities \( v_p \) calculated from elasticity tensor

Calculated distribution of \( v_p \) with increasing amount of \( \gamma \)-fibre (rest \( \alpha \)-fibre)

Measured distribution of \( v_p \):
- V2: strong \( \gamma \)-fibre
- V1: more \( \alpha \)-fibre
- FV: \( \alpha \)-fibre & large grains

Difference \( \Delta v_P = 100 \) m/s can be expected (measurement error \( \sim 20 \) m/s)

Trend in measurement results not conclusive (errors?)

More measurements are necessary
Distribution of shear wave velocities calculated from elasticity tensor

Calculated distribution of $v_{S1}$ with increasing amount of $\gamma$–fibre (rest $\alpha$-fibre)

Measured distribution of $v_{S1}$.

V2: strong $\gamma$–fibre
V1: more $\alpha$-fibre
FV: $\alpha$-fibre & large grains

Ultrasound measurement difficult due to multiple reflections and sample surfaces

Only small differences at measurement positions

Measurement directions not optimal

Nevertheless comparison with measured velocities possible
Symmetry differences of pole figures can tell you about:

- Inhomogeneous deformation, measurement statistic, sample preparation, ...

Orthorhombic sample symmetry should never be imposed! (except for ODF plots...)
MTEX in steel research

Texture Analysis – Symmetry of pole figures

Approach: Flip pole figures about mirror axes and calculate difference with unflipped pole figure.

```matlab
%Calculate pole figure from ODF
pf_calc = calcPoleFigure(odf,h,'resolution',1*degree,'complete');

%Flip upside-down
rotation_ud = rotation('axis',zvector,'angle',180*degree);

%Flip left-right
rotation_lr = rotation('axis',yvector,'angle',180*degree);

%Do Flip
odf_ud = rotate(odf,rotation_ud);
odf_lr = rotate(odf,rotation_lr);

%flipped pole figures
pf_ud = calcPoleFigure(odf_ud,h,'resolution',1*degree,'complete');
pf_lr = calcPoleFigure(odf_lr,h,'resolution',1*degree,'complete');

%Calculate error between original and flipped pole figures
error_ud = mean(calcError(pf_calc,pf_ud));
error_lr = mean(calcError(pf_calc,pf_lr));
```

Mean asymmetry = 0.074

Mean asymmetry = 0.214
Introduction

Macro Texture Analysis (XRD & EBSD)
- Elasticity tensor
- Wave velocities and ultrasound
- Symmetry of pole figures

EBSD Analysis
- Homogeneity
- IQ Analysis
- Prior Austenite Grains

Summary
MTEX in steel research

EBSD Analysis

Orientation map

Phase map

Image Quality map

BCC  FCC
MTEX in steel research

EBSD Analysis

Misorientation angle distribution

- Typical distribution of bainitic microstructure
- Useful for **phase discrimination** ferrite-bainite-martensite
- Uncorrelated distribution (green) and random distribution (red)

Grain size

- For every phase
- For 4° and 15° segmentation angle (LAGB – HAGB)
MTEX in steel research

EBSD Analysis – Homogeniety

Kernel average misorientation maps of multi phase steels produced with different cooling conditions

Lorenz curves of KAM distribution
[Rossi et. al., 2014 Pract. Met., 51(3), 180-199]

Homogeneous distribution of dislocations (KAM) is important for **crack resistance**.

Can be characterized by **one value H**, the area under the Lorenz curve.

Statistical error \( \Delta H \approx 0.002 \)
MTEX in steel research

EBSD Analysis – Homogeniety

Calculation

%KAM values
KAM_1 = KAM(ebsd,'threshold',4*degree,'order',1)./degree;

%calculate cumulative KAM values
cum_sum_kam = cumsum(sort(snip(KAM_1,nan))).../
sum(snip(KAM_1,nan));

%calculate number of cumulative KAM values
cum_x = (1:numel(cum_sum_kam))./numel(cum_sum_kam);

%calculate homogeniety
homo_kam = 2*trapz(cum_x, cum_sum_kam);

Quantitative description of Homogeneity

Can easily be applied to all kind of distributions, e.g. grain size, particles,...

Combined homogeneities can be used, e.g. H = H_{size} * H_{shape}
Image Quality (IQ) describes the contrast of the Kikuchi-Patterns.

It is reduced by lattice distortions like dislocations, grain boundaries, micro strains, ...

IQ histogram of bcc iron may be the sum of two distributions. One with high IQ values (low distortion) and one with low IQ values (high distortion).

Phase quantification Ferrit – Bainite – (Martensite)
MTEX in steel research

**EBSD Analysis – IQ distribution**

Remove points at grain boundaries

```matlab
% IDs of ebsd measurements at grain boundaries
ids = grains.boundary.ebsdId;
ids = unique(ids(ids>0));

% vector with boundary points
isGrainBoundaryEBSD = sparse(ids,1,true,ebsd.size(1),ebsd.size(2));

% exclude points at grain boundaries
ebsd_iq = ebsd(~isGrainBoundaryEBSD);

% normalize IQ
iq_cor = ebsd.iq;
iq_cor_n = 100 .*(iq_cor - min(iq_cor))/(max(iq_cor)-min(iq_cor));
```

Grain boundary points removed
MTEX in steel research

**EBSD Analysis – IQ distribution**

Fit with two Gaussian functions (Curve Fitting Toolbox)

```matlab
% define binning
bins = 0:1:100;

% histogram counts
n = histc(iq_cor_n,bins);
n = n/sum(n);

load enso;
% two Gauss peaks, start conditions
options = fitoptions('gauss2','StartPoint',[0.1 65 10 0.1 40 10]);

% fit
f = fit(bins', n,'gauss2',options);

% integrate results
fun_1 = @(x) f.a1.*exp(-(x-f.b1)./f.c1).^2);
fun_2 = @(x) f.a2.*exp(-(x-f.b2)./f.c2).^2);
fit_area_1 = integral(fun_1,0,100);
fit_area_2 = integral(fun_2,0,100);

% plot result
figure
plot(f,0:1:100,n), hold on
fplot(fun_1,[0 100],'color','green')
fplot(fun_2,[0 100],'color','black')
xlabel('Image Quality Ferrit (Normalisiert & Korrigiert)','FontSize', 14)
ylabel('Anteil','FontSize', 14)
legend('Data','Gesamter Fit',sprintf('Fit 1 (%.2f)',fit_area_1),sprintf('Fit 2 (%.2f)',fit_area_2),'Location','NorthEastOutside')
grid on
set(gca,'FontSize', 14)
saveFigure('IQ_fit_histogramm.png')
hold off
```
MTEX in steel research

EBSD Analysis – IQ distribution

IQ histogram

Drop of IQ at grain boundary

Result: 70% Bainite, 30% Ferrite

Reminder: IQ values are strongly influenced by sample preparation and measurement conditions

Effect of grain boundaries to reduce IQ extends ~300nm, thus grain boundary effect may not be completely eliminated.

Results are hence somewhat biased and give no spatial information
The size of the final \( \alpha \)-Fe microstructure is **determined by the prior austenite grain size**.

The prior austenite grain size can be directly influenced by rolling temperature, amount of deformation during hot rolling and microchemistry.

But it is very **difficult to measure** this austenite grain size in-situ.

After hot rolling the **phase transformation** austenite (\( \gamma \)-Fe) to \( \alpha \)-Fe occurs with certain **orientation relationships** between parent and daughter grains.

- Kurdimov-Sachs (K.-S.): 90° rotation about <1,1,2>
- Nishiyama-Wassermann (N.-W.): 95.3° rotation about <3,6,2>
- The observed orientation relationships are somewhere in between these two.
- Not all symmetrical equivalent relationships occur (variant selection)

**→** Determination of the fcc \( \gamma \)-Fe **from EBSD measurements** of the bcc \( \alpha \)-Fe microstructure
EBSD Analysis – Prior Austenite Grains

Approach: **Delete all grain boundaries** that deviate less than 5° from K.-S. and N.-W.

```plaintext
%Kurdimov-Sachs misorientation
ori_KS = orientation('axis', Miller(1,1,2,CS), 'angle', 90*degree, CS, CS);

%Nishiyama-Wassermann misorientation
ori_NW = orientation('axis', Miller(3,6,2,CS), 'angle', 95.3*degree, CS, CS);

%CSL3 misorientation
ori_twin = orientation('axis', Miller(1,1,1,CS), 'angle', 60*degree, CS, CS);

%grain boundaries of phase 1 (ferrite)
gb = grains.boundary('1','1');

%indices of grain boundaries to delete
ind_5deg = angle(gb.misorientation, ori_KS) < 5*degree | ...
          angle(gb.misorientation, ori_NW) < 5*degree | ...
          angle(gb.misorientation, ori_twin) < 5*degree;

%merge grains
[grains_merge_5deg, grains_merge_5deg_parent_id] =
  merge(grains, gb(ind_5deg));
```

- CSL3 boundaries occur at martensitic transformation.
- To recover the austenite structure from martensite CSL3 boundaries are also deleted.
MTEX in steel research

EBSD Analysis – Prior Austenite Grains

Test with K.-S. and arbitrary misorientation

Only with correct orientation relationship merging occurs.

Measurement field too small for austenite microstructure.

35.3° about <1,2,3>
5° tolerance

K.-S.
5° tolerance
Recrystallized grain structure. Prior austenite grain size ~200 µm
Elongated grain structure $\rightarrow$ No recrystallization before transformation

Measurement field still too small…

K.-S. & N.-W. + CSL3
5° tolerance
-32% grain boundaries
Summary

Using MTEX scripts can automate XRD and EBSD evaluation.

Implementation into Matlab gives access to large amount of analysis tools.

Examples shown

- Elasticity tensor
- Wave velocities and ultrasound
- Symmetry of pole figures
- Homogeneity
- IQ Analysis
- Prior Austenite Grains
No matter what you have planned ...

Thank you for your attention!