Microscopy Focus

CORRELATING EBSD WITH ULTRASONIC MEASUREMENTS OF CRYSTALLOGRAPHIC TEXTURE IN METALS

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The quantitative measurement of crystallographic texture through determination of the Orientation Distribution Coefficients (ODCs) can provide critical information on a sample's suitability for being utilised in a particular manufacturing process or can be used to measure changes in the microstructure of components in service. Ultrasonic techniques have been developed by previous workers that measure these ODCs. Electron Backscatter Diffraction (EBSD), a microscopic technique that measures the crystallographic orientations of individual crystals, has been utilised to offer an alternative method to measuring these ODCs. As a technique, EBSD provides a much more detailed measurementof texture than ultrasonic measurements ever could. Ultrasonic methods are however non-destructive, can be used on components in service and are quicker and less expensiveto implement that EBSD measurements. EBSD is isa valuable method in validating ultrasonic measurements, and can help to guide us in determining the limitations of the ultrasonic measurements. Ultrasonic measurement of texture is, and will continue to be a useful approach to measuring texture, but it does have its limitations and advantages for application to "real world" samples.

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Poster in association with RMS and sponsored by ILM

WHAT IS TEXTURE?

Texture is a measure of anisotropy, describing the existence of preferential crystallographic orientations. Quantitatively expressed by the Orientation Distribution Function (ODF) w (ξ,φ,ϕ) [1], this is a probability distribution averaged over all possible crystallographic orientations (ξ,φ,ϕ) are Roe-Euler angles) (Eqn 1):

$$\int_0^{2\pi}\int_0^{2\pi}\int_{-1}^{1} w(\xi,\psi,\phi)\,d\xi\,d\psi\,d\phi = 1 \quad (1)$$

Alternatively, can obtain $w(\xi,\psi,\Phi)$ using (Eqn 2):

$$w(\xi,\psi,\phi) = \sum_{l=0}^{\infty}\sum_{m=-l}^{l}\sum_{n=-l}^{l} W_{lmn} Z_{lmn}(\xi) \exp^{-im\psi} \exp^{-in\phi} \quad (2)$$

where W_{lmn} are the Orientation Distribution Coefficients (ODCs).

ULTRASONIC DETERMINATION OF TEXTURE

For cubic materials three ODCs, W_{400}, W_{420} and W_{440}, are required to define the elastically determined texture of a sample. For thin sheets, it has been shown that these can be calculated by measuring the ultrasonic S_0 Lamb wave velocity (v) as a function of angle (θ) to the rolling direction (RD) with the ODCs determined as a best fit to the velocity plot (Eqn) [2]:

$$\rho v^2 = A + BW_{400} + C\cos(2\theta)W_{420} + D\cos(4\theta)W_{440} \quad (3)$$

ULTRASOUND SETUP TO DETERMINE ODCs

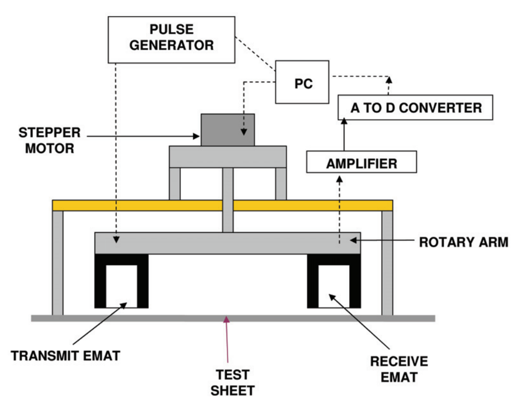

Figure 1. Schematic of EMAT-EMAT system setup

- Transmit and receive EMAT-EMAT system [3]
- Fixed separation of 15cm
- Transducers rotate in 3.6°increments
- 100 velocities taken per position for averaging

AIM: To show that ultrasonically determined ODCs are consistent with ODCs obtained using EBSD. By treating the sample to have orthorhombic symmetry, theory suggests we can generate a predicted S_0 Lamb wave velocity plot by calculating the elastic moduli from EBSD. This will allow validation of the ultrasound technique.

ELECTRON BACKSCATTER DIFFRACTION

This microscopic technique provides a map of a given sample's microstructure by assigning a colour (*Figure 2*) for any given orientation of a point on the surface.

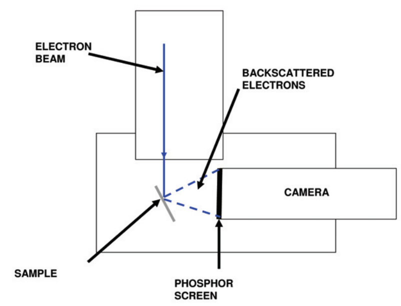

Figure 2. Schematic of EBSD setup

HOW IT WORKS

- Sample tilted at 70° in SEM
- At high vacuum
- Elastically backscattered electrons collected by phosphor screen camera
- Backscatter patterns are indexed to determine crystallographic orientation

MEASURING TEXTURE FROM EBSD

The orientation matrix g (equation 4) maps a cubic crystal's orthogonal axes system to the orthogonal axes system of the polycrystallite. The angles *(φ1,Φ,φ2)* are the Bunge-Euler angles which define crystallographic orientations which EBSD measures. An associated colour is assigned to that orientation from the coloured Inverse Pole Figure (IPF) as seen in *Figure 3*.

$$g = \begin{pmatrix} \cos\varphi_1\cos\varphi_2 - \sin\varphi_1\sin\varphi_2\cos\Phi & \sin\varphi_1\cos\varphi_2 + \cos\varphi_1\sin\varphi_2\cos\Phi & \sin\varphi_2\sin\Phi \\ -\cos\varphi_1\sin\varphi_2 - \sin\varphi_1\cos\varphi_2\cos\Phi & -\sin\varphi_1\sin\varphi_2 + \cos\varphi_1\cos\varphi_2\cos\Phi & \cos\varphi_2\sin\Phi \\ \sin\varphi_1\sin\Phi & -\cos\varphi_1\sin\Phi & \cos\Phi \end{pmatrix} \quad (4)$$

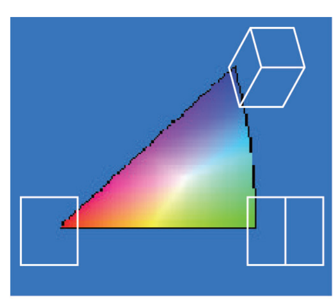

Figure 3. EBSD Inverse Pole Figure (IPF): indicates the colour distribution for all crystallographic orientations

Assuming orthorhombic symmetry, the nine independent elastic moduli of a rolled sample can be calculated [4] from crystallographic orientation data.

This implies EBSD can predict the elastic moduli in rolled sheets which can be used to ascertain the velocity profile [4] and ODCs (Eqn 5-8) α and β are constants dependent on the elastic moduli, λ and μ the lamé constants and c anisotropy parameter.

$$v_{s_0} = \left(\frac{\hat{C}_L}{\rho}\right)^{1/2} \left[1 + \frac{\alpha}{4}\cos 2\theta - \left(\frac{\hat{\beta}\hat{C}_T}{4\hat{C}_L}\right)(1 - \cos 4\theta) + \dots\right] \quad (5)$$

$$W_{400} = \frac{c_{33} - \lambda - 2\mu}{\left(\frac{32\sqrt{2}c\pi^2}{35}\right)} \quad (6) \qquad W_{420} = \frac{c_{22} - c_{11}}{\left(\frac{32c\pi^2}{7\sqrt{5}}\right)} \quad (7)$$

$$W_{440} = \frac{\frac{c_{12} - \lambda}{\left(\frac{4\sqrt{2}c\pi^2}{35}\right)} - W_{400}}{-\sqrt{70}} \quad (8)$$

RESULTS FOR 0.5mm THICK ROLLED ALUMINIUM SHEET (AL104232)

EMAT-EMAT S_0 LAMB WAVE ULTRASOUND RESULTS

Sample was AL104232 300mm x 300m x 0.5mm Aluminium Sheet. *Figure 5* shows the velocity profile as a function of angle, with fastest velocities perpendicular to RD. The ODCs calculated are also shown.

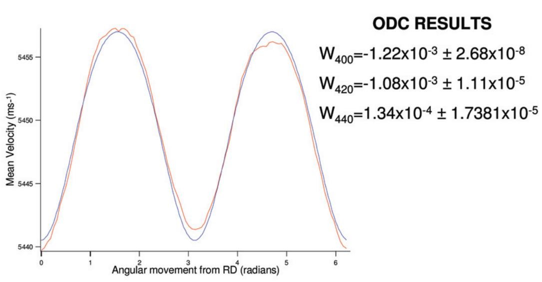

Figure 4. Ultrasonic Velocity vs. angle subtended from the rolling direction over a 360 degree rotation. The red trace is the ultrasound data, the blue trace is the solution to Eqn 3.

EBSD RESULTS

Through-thickness EBSD scans were taken. They display a definite layering within the sample, implying surface scans are not representative of the bulk. Therefore it was assumed that these through thickness scans would be a more appropriate measure of the bulk texture, as would be measured by the S_0 Lamb wave. Below are results obtained from the EBSD scan of a 0.5 mm thick sheet.

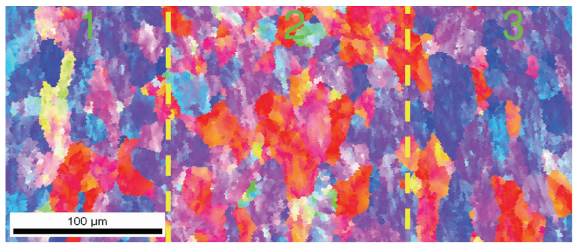

Figure 5. 1_11 EBSD scan taken from AL104232. The left and right hand side are below the RD-TD plane surfaces. [111] orientation dominant in layers 1 and 3.

Layering consistent throughout all scans taken.

Table 1. Shows calculated elastic moduli and their fluctuations for each layer after rotation into RD-TD plane.

EBSD Scan name → / Longitudinal Elastic Modulus (GPa) ↓	1_11 lhs layer	1_11 centre layer	1_11 rhs layer	1_11 overall
C11	111.917	111.635	111.967	111.834
C22	112.595	111.225	112.636	112.148
C33	112.826	111.850	112.826	112.432

The three zones have distinctly different elastic anisotropy

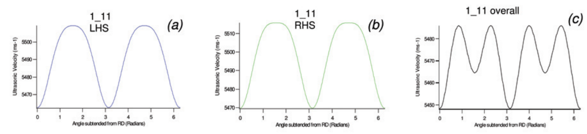

Figure 6:Graphs of predicted ultrasound results from 1_11 left hand side partition (a) and right hand side partition (b) showing good correlation to initial ultrasound data. Black trace (c) is for all 6 scans taken from section [1].

Sub-surface layers give accurate velocity profiles compared to ultrasound (*See Figure. 4*).

CONCLUSIONS

- Ultrasonic system delivers a measure of elastic modulus variation as a function of direction - linked to average through thickness texture where ODCs can be calculated.
- EBSD scans on through thickness of sheet show correlation in elastic modulus prediction, as demonstrated by velocity plots.
- Numerous scans sampling over a large number of grains are required – demonstrated local & surface-bulk texture variations.

FURTHER WORK

- Continue current work on different thickness Al sheet. Is elastic moduli fluctuation banding significant in other alloys / heat treatment / rolling processes?
- Finite Element modelling Analysis of Lamb wave propagation. How does the variation in Elastic modulus through the bulk effect velocity?
- Go on to study other cubic materials such as steel.

Microscopy Diary - September 2007

3-7 September
Cell Imaging Techniques Course, Oxford Brookes University
Contact: events@rms.org.UK

3-7 September
EMAG 2007, Electron Microscopy and Analysis Group Conference 2007, Glasgow Caledonian University & University of Glasgow, UK
Further details: www.iop.org/Conferences

9-12 September
6th Abercrombie Meeting – cell migration: from molecules to organisms, St Catherine's College, Oxford, UK. Contact: events@rms.org.uk

10-14 September
Flow Cytometry Course, University of York, UK.
Contact: events@rms.org.uk

24 September
Raman and Fluorescence in Bio Diagnostics, Medical Sciences Teaching Centre, Oxford UK

24 September
AGM of the Royal Microscopical Society's Light Microscopy Section, Medical Sciences Teaching Centre, Oxford, UK.
Contact: events@rms.org.uk

Circle no. 256

Enabling Innovation through Micro and Nano Technologies

The Centre of Excellence in Metrology for Micro and Nano Technologies (CEMMNT), the National Physical Laboratory (NPL), and The Innovation Advisory Service South East (IASSE) are hosting a one-day meeting which will benefit companies using micro and nano technologies to enable and accelerate new products and processes. This event, to be held at the National Physical Laboratory, Teddington UK, will identify the key drivers which accelerate innovation and provide the networking platform to solve challenges, build partnerships and access funding opportunities. Anyone involved in business, product and process development is encouraged to attend.

Presentations highlighting the latest technology developments and applications will be complemented by discussion forums which define the key barriers for innovation and determine how these can be overcome. Guidance will be provided on regional, national and European funding initiatives and how to successfully leverage funding for collaborative projects. Surgeries offer the opportunity for delegates to receive free problem solving advice from NPL and CEMMNT. Networking and establishing partnerships will form an integral part of the day.

This free event will take place on Wednesday the 26th of September at the National Physical Laboratories in Teddington.

Circle no. 257

EMAG 2007

The Electron Microscopy and Analysis Group (EMAG) biennial conference has established a strong reputation as a key event in the calendars of the national and international microscopy communities, since it inaugural meeting in 1946. EMAG 2007, September 3-7 will continue this tradition in Glasgow, a city that has a long history in science, engineering and innovation. The excellent transport links mean that EMAG 2007 is easily accessible for day visitors as well as for delegates attending the whole conference and the Advance School. The exhibition and conference will be held at the Glasgow Caledonian University, a central point for the conference. Registration, poster sessions, lunch and refreshments will all be taking place all within the exhibition hall. There is also an opportunity for exhibitors to present a commercial workshop to outside visitors in the theatre area situated in the exhibition hall throughout the event.

Circle no. 258