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Unbiased evaluation of chord length distributions from orientation maps

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Abstract. Grain structures in crystalline material are commonly identified by orientation inhomogeneities in orientation maps. A statistical approach is introduced to extract quantitative information on the chord length distribution of grains from orientation data gathered by electron back-scattering diffraction.

Introduction

Areas with different reflectivity can be distinguished on metal surfaces by means of optical microscopy or sometimes even with the naked eye. These individual areas correspond to regions in the metal with different orientations of the crystalline lattice. Metallic specimens are usually built of many of such grains. Each individual grain in a polycrystal is characterized as a contiguous region of homogeneous orientation separated from grains of different orientations by grain boundaries. Grain boundaries are associated with a finite orientation difference, but the orientation difference can be rather small, especially for strongly textured materials. Distinction of individual grains by their unique orientation separating them clearly from their neighbors is only possible for as-deposited, as-grown or fully recrystallized material. During plastic deformation, for instance, grains of initially homogeneous orientations become subdivided by emerging deformation-induced boundaries into smaller regions (subgrains, dislocation cells or cell blocks) of slightly deviating, but similar orientations.

Electron back-scattering diffraction (EBSD, e.g. [1]) can resolve the spatial arrangement of orientations. By means of a scanning electron microscope local orientations are determined from electron backscatter patterns at individual points on a specimen surface. Orientation data are usually gathered for many points on regular grids on the specimen surface and displayed by representing different orientations by different colors as orientation maps (orientation imaging microscopy, OIM [2]). Any particular orientation of the crystalline lattice is described as rotation from a particular reference orientation [3]. The orientation difference between two orientations is characterized by a misorientation angle and a misorientation axis. Taking into account the symmetry of the crystalline lattice (e.g. 24 symmetry operations for cubic symmetry), the lowest of all possible misorientation angles is selected and termed disorientation angle θ.

Identification of original grain boundaries in an orientation map by a simple threshold for the disorientation angle might be misleading, as the disorientations within a single grain increase with proceeding deformation and some of the initial orientation differences between grains in the polycrystal are rather small. In order to gain information on the grain scale, statistical methods must be employed. The most relevant parameter for characterization of grain structures is their grain size distribution, but its evaluation from two-dimensional sections requires specific assumptions about the

grain shape. Therefore, the chord length, i.e. the intersection length along a test line, and its distribution will be considered here.

A strict mathematical scheme is presented for determining the chord length distribution from the dependence of the average disorientation angle on the step size between EBSD measuring points [4]. Such an indirect approach for determination of the chord length from loosing any orientation correlation can be applied even in heavily deformed material where grain boundaries and deformation-induced boundaries cannot be distinguished by the disorientation angle any longer.

Statistical information on grain structure

Outline of the problem. Grain structures are characterized by their chord length distribution. The chord length l of a grain is the length of the intersection of the grain with a test line. In traditional stereology, the number of intersections N_{is} of a (random) test line of length L with grain boundaries is counted. The number density $N_L = N_{\mathrm{is}}/L$ of intersections is the inverse of the mean chord length $\bar{l} = 1/N_L$. Alternatively, individual chord lengths l_{i} of the intersected grains can be recorded. From all collected distances l_{i} between two subsequent intersections the chord length distribution $f_{\mathrm{cl}}(l)$ as well as the average chord length $\bar{l}$ can be derived.

Both procedures can easily be adapted to orientation data: If orientation data are gathered on grid lines with a constant step size Δx between subsequent points, disorientation angles between all N point pairs consisting of neighboring points along the grid lines can be calculated. If a particular disorientation angle θ_{i} is above a certain threshold angle θ_{cr}, the existence of a grain boundary between the two points of the pair is assumed. From the number N_{cr} of such occurrences and the total line length $L = N\Delta x$ the intersection density $N_L = N_{\mathrm{cr}}/N\Delta x$ and the average chord length $\bar{l} = N\Delta x/N_{\mathrm{cr}}$ can be inferred.

These definitions rely on a properly chosen sampling distance Δx and even more critical on the selection of a suitable threshold value θ_{cr}: If the disorientation angles θ_{i} between all point pairs of (valid) neighboring points are determined and sorted in increasing order, the probability $P_\theta(\theta_{\mathrm{cr}})$ for finding a disorientation angle θ less than an angle θ_{cr} is easily obtained from the serial number in the sorted list. The accumulated frequency distribution

$$P_\theta(\theta_{\mathrm{cr}}) = 1 - \frac{N_{\mathrm{cr}}}{N} \tag{1}$$

is intimately linked to the number N_{cr} of incidences of point pairs with angles above the critical angle. Consequently, the resulting mean chord length

$$\bar{l}(\theta_{\mathrm{cr}}) = \frac{N}{N_{\mathrm{cr}}}\Delta x = \frac{\Delta x}{1 - P_\theta(\theta_{\mathrm{cr}})} \tag{2}$$

depends strongly on the selection of a suitable threshold value θ_{cr} (and the step size Δx).

As discussed in the introduction, neighboring grains in an ideal grain structure may have very small disorientations (especially for strong textures) and a rather small threshold value is advisable. Unfortunately, there are two restrictions for lowering the threshold to too small values: (i) the first is the limited accuracy of the orientation measurement. With standard EBSD equipment and evaluation software the disorientation can be determined reliably within 1° (sometimes 0.5°). A more accurate determination of the disorientation would require a different type of pattern analysis, e.g. simultaneous evaluation of two overlapping patterns. (ii) Plastic deformation, on the other hand, gives rise to deformation-induced boundaries with significant disorientation angles. For instance, the average disorientation angle across geometrically necessary boundaries (GNBs) is 2° after 10 % cold-rolling

and increases with further deformation [5]. Both effects cause certain 'noise' in the orientation determination. This makes the definition of a proper threshold value difficult (if not even impossible) and different evaluation schemes must be employed as the statistical approach described in the next subsection.

A second effect should be mentioned briefly: Occasionally, a particular electron backscatter pattern cannot be indexed properly due to the overlap of two distinct diffraction patterns or the general quality of the pattern. Then the orientation at a particular position cannot be determined. These non-indexed points have to be handled with care: both suggested methods, ignoring the affected point pairs entirely or calculation of disorientation with the next neighbor instead, bias the result in a certain, but at least known and quantifiable manner. Other attempts presented in literature for establishing the missing information at a certain non-indexed point from weighted averaging over its neighbors or a certain filtering of the data are highly questionable, because their effect on statistics is difficult to access.

Statistical evaluation. Aiming towards an improved chord length evaluation, the distribution of the disorientation angles is considered in detail for an ideal grain structure (without any orientation differences within the grains). If individual orientations are measured along a grid line of length L with a step size Δx between subsequent points, the disorientation between all $N = L/\Delta x$ point pairs can be calculated. Only for N_{rel} point pairs, there will be a grain boundary between the two points, but no grain boundary will be found between the points of the other $N - N_{\mathrm{rel}}$ point pairs. The first mentioned point pairs form the relevant fraction and must possess non-vanishing disorientation angles θ, whereas the later point pairs should not have any disorientation at all and are considered to be irrelevant. (This distinction between relevant and irrelevant point pairs has been introduced earlier in a similar approach of the author for analyzing deformation structures [6].) Due to the influences mentioned in the previous subsection, there will be a certain disorientation originating from the 'noise', even if no boundary is present between the two points of a pair. Consequently, the total distribution of disorientation angles will be a superposition

$$f(\theta) = \xi f_{\mathrm{rel}}(\theta) + (1-\xi) f_{\mathrm{irr}}(\theta) \tag{3}$$

of a relevant distribution $f_{\mathrm{rel}}(\theta)$ from grain boundaries and an irrelevant distribution $f_{\mathrm{irr}}(\theta)$ from the accuracy of the measurement. The weighting factors are the relevant fraction $\xi = N_{\mathrm{rel}}/N$ and the irrelevant fraction $\bar{\xi} = 1 - \xi$ of point pairs, respectively. The relevant distribution is a direct consequence of all orientations present in the material, i.e. the texture, and can be calculated from the texture as part of the 'misorientation distribution function' MODF (at least, if no orientation correlations in the grain structure are present). The irrelevant distribution is an instrumental distribution and depends on the EBSD equipment, the pattern recognition software as well as the particular patterns or the particular orientations investigated.

Relation between relevant fraction and chord length distribution. For a grain structure of known chord length distribution $f_{\mathrm{cl}}(l)$ the relevant fraction of point pairs can be derived theoretically in dependence on the step size Δx. A point pair cannot contain any grain boundary, if the first point of the pair is located further than Δx apart from the next grain boundary. The irrelevant fraction

$$\bar{\xi} = \frac{\int_{\Delta x}^{\infty} (l - \Delta x) f_{\mathrm{cl}}(l)\,\mathrm{d}l}{\int_{0}^{\infty} l f_{\mathrm{cl}}(l)\,\mathrm{d}l} \tag{4}$$

can be derived from the probability of finding a point located in a distance larger than the step size Δx from the next grain boundary along the grid line.

Consequently, the relevant fraction (derived by partial integration from Eq. 4)

$$\xi = 1 - \bar{\xi} = \frac{1}{\bar{l}} \int_0^{\Delta x} \left(1 - \int_0^{l} f_{\mathrm{cl}}(l')\mathrm{d}l' \right) \mathrm{d}l \tag{5}$$

depends strongly on the sampling distance Δx. Independent of the chord length distribution, two limiting cases can be discussed: For large step sizes, at least a single grain boundary is located between nearly all point pairs and all contribute to the relevant fraction ($\xi = 1$). For small Δx, the linear relationship $\xi = \Delta x / \bar{l}$ obtained in an earlier approach [6] on deformation structures is confirmed.

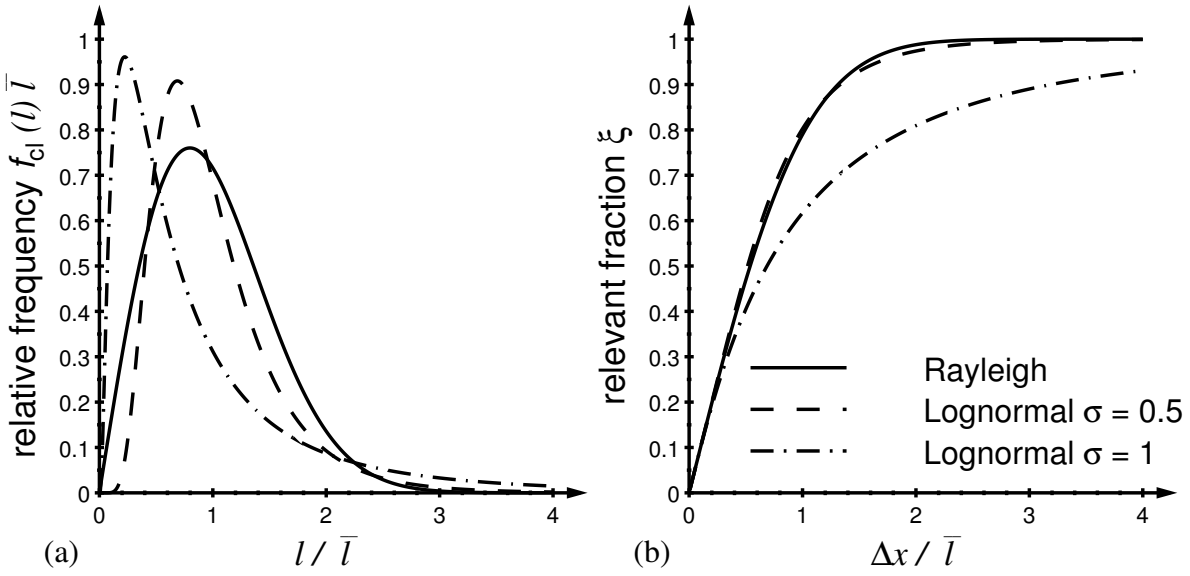


Fig. 1: Chord length distributions (a) and resulting theoretical relevant fractions (b) for the Rayleigh distribution and two logarithmic-normal distributions with different coefficients of variance.

The dependence of the relevant fraction on $\Delta x / \bar{l}$ is illustrated in Fig. 1 for two chord length distributions commonly used for grain structures. If the chord length distribution can be described by a Rayleigh distribution

$$f_{\mathrm{cl,R}}(l) = \frac{\pi}{2} \frac{l}{\bar{l}^2} \exp\left(-\frac{\pi}{4} \frac{l^2}{\bar{l}^2} \right) \tag{6}$$

the relevant fraction becomes

$$\xi_{\mathrm{R}}(\Delta x) = \mathrm{erf}\left(\frac{\sqrt{\pi}}{2} \frac{\Delta x}{\bar{l}} \right) . \tag{7}$$

A logarithmic-normal distribution (containing an additional parameter σ describing the coefficient of variance $\sqrt{\exp(\sigma^2) - 1}$)

$$f_{\mathrm{cl,ln}}(l) = \frac{1}{\sqrt{2\pi} l \sigma} \exp\left(-\frac{(2\ln(l/\bar{l}) + \sigma^2)^2}{8\sigma^2} \right) \tag{8}$$

leads in an analogous manner to a relevant fraction

$$\xi_{\mathrm{ln}}(\Delta x) = \frac{1}{2}\left[1 + \mathrm{erf}\left(\frac{2\ln(\Delta x/\bar{l}) - \sigma^2}{\sqrt{8}\sigma} \right) \right] + \frac{1}{2}\frac{\Delta x}{\bar{l}}\left[1 - \mathrm{erf}\left(\frac{2\ln(\Delta x/\bar{l}) + \sigma^2}{\sqrt{8}\sigma} \right) \right] . \tag{9}$$

Determination of chord length distribution from experimental orientation data. The dependence of the relevant fraction $\xi(\Delta x)$ on the step size can be evaluated from the average disorientation angle $\langle\theta\rangle$, as the average disorientation angle depends on the relevant fraction analogous to the disorientation angle distributions in Eq. 3

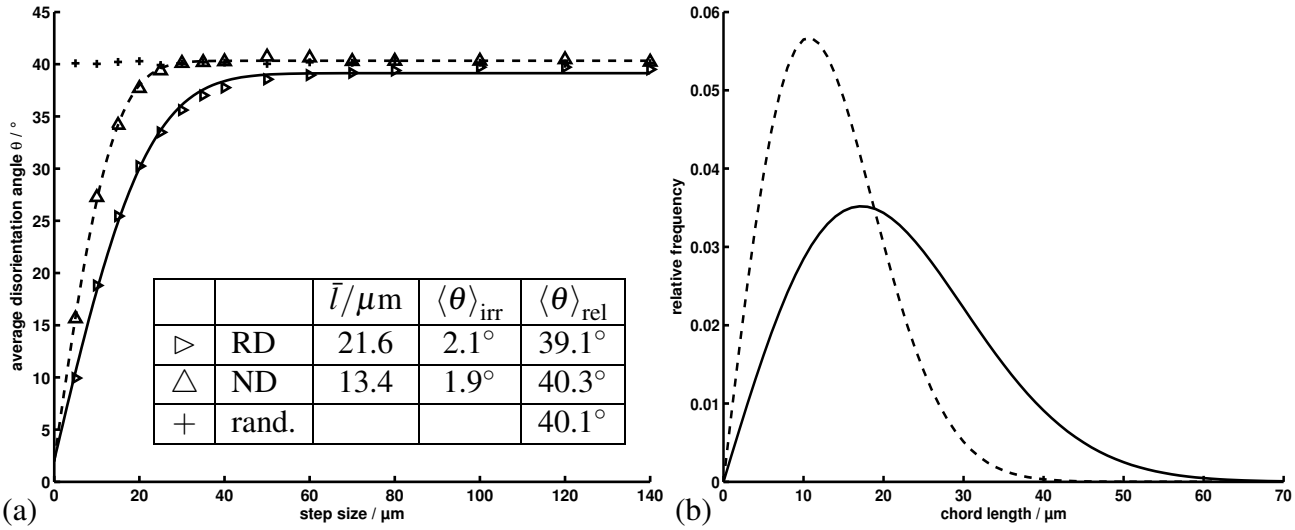
$$\langle\theta\rangle = \xi\langle\theta\rangle_{\mathrm{irr}} + (1-\xi)\langle\theta\rangle_{\mathrm{irr}} = \xi\left(\langle\theta\rangle_{\mathrm{rel}} - \langle\theta\rangle_{\mathrm{irr}}\right) + \langle\theta\rangle_{\mathrm{irr}}. \tag{10}$$

In principle, the chord length distribution can be obtained from the second derivative of the relevant fraction with respect to the sampling distance (cf. Eq. 5)

$$f_{\mathrm{cl}}(l) = -\bar{l}\left[\frac{\mathrm{d}^2}{\mathrm{d}(\Delta x)^2}\xi\right]_{\Delta x = l}. \tag{11}$$

Due to numerical errors caused by differentiation, such a procedure is however not advisable.

Alternatively, a reversed procedure can be applied: a proper distribution function (with free parameters) is selected as chord length distribution, the resulting relevant fraction calculated from Eq. 5 and fitted to experimental data of e.g. the average disorientation angle in dependence on the step size according to Eq. 10. The later can be gained from experimental data gathered with an elementary step size Δx_0 by calculating the disorientation angles and their average not only between neighboring points in the elementary distance Δx_0, but also for all possible pairs of points in larger distances $\Delta x = n\Delta x_0$. In this manner, average disorientation angles $\langle\theta\rangle(n\Delta x_0)$ can be obtained for all possible integers n.



		$\bar{l}/\mu$m	$\langle\theta\rangle_{\mathrm{irr}}$	$\langle\theta\rangle_{\mathrm{rel}}$
▷	RD	21.6	2.1°	39.1°
△	ND	13.4	1.9°	40.3°
+	rand.			40.1°

Fig. 2: (a) Average disorientation angle $\langle\theta\rangle$ in dependence on step size Δx between point pairs (along the initial rolling direction, RD, and normal direction, ND) for a recrystallized aluminium sample initially cold-rolled to 90 %. The full and dashed curve are fits using relevant fractions corresponding to Rayleigh distributions for the chord length distributions as illustrated in (b). 'Randomized' average disorientation angles are obtained by applying the same analysis to an orientation map constructed by distributing exactly the same orientations randomly on the grid points.

Example. The suggested methodology is illustrated for an aluminium polycrystal cold-rolled to 90 % reduction and annealed to complete recrystallization. A large orientation map has been obtained with a step size of 5 μm covering an area of 400 μm $\times$ 1000 μm (along the initial normal and rolling direction, respectively). Only 4 % of the points could not be indexed automatically.

From the recorded orientations the average disorientation angles have been calculated for different effective sampling distances $\Delta x = n\Delta x_0$ separately along the previous rolling and normal direction. As seen in Fig. 2, fitting of relevant fractions ξ_R according to Eq. 7 (assuming a Rayleigh distribution for the chord length distribution with $\bar{l}$ as single fitting parameter) leads to a rather good correspondence between experimental observation and theoretical description. For the mean chord length along the previous rolling direction (21.6 μm) a larger value is obtained then along the previous normal direction (13.4 μm) indicating a still elongated grain structure.

For comparison purposes all orientations present in the orientation map have been randomly distributed on the map and evaluated again. As expected, the average disorientation angle (40.1°) does not depend on the effective sampling distance in this case. The resulting average disorientation angle from the randomized orientations is equivalent to the average disorientation angle calculated from the texture (or MODF, respectively) and corresponds obviously (cf. Fig. 2) to the average disorientation angle obtained from the original orientation map for large sampling distances, where no orientation correlations are preserved any longer. The small deviation of the average disorientation angle (39.1°) at large distances along the rolling direction from the analogous value (40.3°) along the normal direction as well as the value from the randomized arrangement may indicate an existing orientation correlation along the rolling direction as a result of e.g. orientation pinning.

Conclusion

A new evaluation scheme for orientation maps from EBSD is presented. Based on classification of relevant and irrelevant point pairs and a statistical approach, the chord length distribution of a grain structure can be evaluated unambiguously from the dependence of the average disorientation angle on the effective step size of the orientation measurements. The proposed method for ideal grain structures can be adapted for deformation structures as well. In the later case, only the physical origin of the noise in the orientation measurements is different and the irrelevant fraction is caused by the deformation-induced boundaries, but the mathematical treatment remains similar.

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