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A peak position comparison method for high-speed quantitative Laue microdiffraction data processing

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ABSTRACT

Indexing Laue patterns of a synchrotron microdiffraction scan can take as much as ten times longer than collecting the data, impeding efficient structural analysis using this technique. Here a novel strategy is developed. By comparing the peak positions of adjacent Laue patterns and checking the intensity sequence, grain and phase boundaries are identified, requiring only a limited number of indexing steps for each individual grain. Using this protocol, the Laue patterns can be indexed on the fly as they are taken. The validation of this method is demonstrated by analyzing the microstructure of a laser 3D printed multi-phase/multi-grain Ni-based superalloy.

Although achieved earlier [1], polychromatic X-ray Laue diffraction was quickly superseded by monochromatic X-ray diffraction for structure analysis, because of the difficulties in interpreting the intensities. With the advent of synchrotron sources and the development of X-ray focusing optics, new applications for Laue diffraction have become possible, utilizing micro- or even nano-sized intense polychromatic X-ray beams under the form of Laue microdiffraction (μXRD) [2]. By scanning the crystalline sample surface with the focused X-ray probe and taking a Laue pattern (LP) at each position using a two-dimensional (2D) detector, this technique has been successfully applied to a range of scientific problems, including phase identification of tiny crystals [3], phase transformation at multi-scales [4,5], crystal orientation mapping [6], elastic strain/stress measurement [7,8], and defect quantification [9–11]. Since the spatial resolution is determined by the X-ray probe size and scanning step, each experiment usually collects massive number of LPs. Due to the high flux of synchrotron X-ray beam, only single exposure of 1 s or less is necessary to record a LP on a bulk crystal. However, analyzing a LP is not trivial. First, the Laue diffraction peak positions are determined accurately after removing background signal from the LPs. Second, for indexing the Laue peak positions are fitted with calculated ones based on a given crystal structure to derive crystal orientation and Miller indices of each diffraction peak. The indexing process may need to be repeated several times, depending on how many possible phases exist in the X-ray scanned area. Finally, refinement is performed to measure the lattice strain by taking into account the tiny deviations between the experimental Laue peak positions and the theoretical ones. This three-step analysis protocol is repeated sequentially and independently for all the LPs of a scan to map the phase, orientation, and strain distribution, which, for most polycrystalline samples, takes time on a regular desktop computer, much longer than that needed for data collection. This makes it incompatible with the study of complex materials with multiple phases, non-uniform grain size, or inhomogeneous defect distribution. For these materials researchers sometimes want to first scan a large area with coarse step size, and then after analyzing the data, map the microstructure of one or more selected areas of interests with finer steps. In these cases, faster data analysis is required. A close look at the conventional data analysis protocol reveals that over 70% of the time is consumed on the indexing process for single phase analysis by using the custom developed X-ray Microdiffraction Analysis Software (XMAS) [12], and up to 95% for multiple phases, indicating that indexing is the bottleneck of the analysis. Therefore, it is essential to shorten the time required to index the Laue scans, each of which contains thousands of LPs or even more.

In this study, we developed a new strategy to reduce the number of LPs that need to be indexed. Using the so-called peak position comparison (PPC) method, high-angle grain boundaries (HAGBs) and phase boundaries (PBs) are mapped rapidly. Only one LP needs to be indexed for each grain to assign the Miller indices to each reflection on the pattern, while the accurate orientation and lattice

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strain distribution can be obtained by carrying out a pattern-by-pattern refinement, which takes much shorter time than indexing. This strategy is employed to study the microstructure of both the epitaxial and stray grain region in a laser 3D printed Ni-based superalloy. A cross-check between the results obtained using the PPC method and the output from XMAS proves the analysis accuracy and reliability. More importantly, by applying the PPC strategy, simultaneous data analysis becomes realistic even on a regular personal computer as the μXRD scan is performed, raising the possibility of real-time analysis to facilities that do not have easy access to supercomputers.

The appearance of PBs and HAGBs is evidenced by a significant change of peak positions between two adjacent LPs. Here 'significant' is emphasized because a slight peak shift are more likely induced by local lattice strain or defects, in which case the indexing result from the previous LP is still valid. Consequently, it is necessary to quantify the peak position change on the 2D detector as a function of disorientations. For convenient description, a detector coordinate system **O-xy** is established, in which the original position **O** is fixed at the bottom left corner of the detector, **x**- and **y**-axes are parallel with the horizontal and vertical edges of the detector, respectively, and the unit length equals to the detector pixel size d_p. Any point P (x, y) on the detector can be expressed by vector **k** that points from the X-ray focal point to P:

$$\mathbf{k} = \mathrm{A} \cdot \begin{bmatrix} (x - x_c) \cdot d_p \\ (y - y_c) \cdot d_p \\ 0 \end{bmatrix} + \begin{bmatrix} 0 \\ 0 \\ d \end{bmatrix} \tag{1}$$

where (x_c, y_c) is the coordinate of the projection position of the X-ray focal point on the detector, and d is the focal point to detector distance. ***A*** is a transformation matrix defined by the tilt of the detector:

$$\boldsymbol{A} = \begin{pmatrix} \cos\theta \cdot \cos\phi & \cos\phi \cdot \sin\theta & -\sin\phi \\ \cos\theta \cdot \sin\psi \cdot \sin\phi - \cos\psi \cdot \sin\theta & \cos\psi \cdot \cos\theta + \sin\psi \cdot \sin\theta \cdot \sin\phi & \cos\phi \cdot \sin\psi \\ \cos\psi \cdot \cos\theta \cdot \sin\phi + \sin\psi \cdot \sin\theta & \cos\psi \cdot \sin\theta \cdot \sin\phi - \cos\theta \cdot \sin\psi & \cos\psi \cdot \cos\phi \end{pmatrix}^{-1} \tag{2}$$

where θ, ψ, and ϕ denote the yaw, pitch, and roll of the detector (as shown in Fig. S1 in the Supplementary material), respectively, and usually ϕ is set to be 0. By combining Eqs. (1) and (2), the angle between any two positions on the detector can be calculated.

To employ the PPC methods, only the very first LP needs to be analysed following the conventional approach. An example of such indexed pattern is shown in Fig. 1a, with the Miller indices marked for each reflection. In a second step, a threshold deviation angle δ is defined by the user, depending on the deformation status. Usually higher threshold angle δ is assigned to highly deformed samples. In this example, δ is set to be 2°. The detector area within 2° around each peak is calculated and marked with dashed ellipses in Fig. 1. Then the Laue peaks in the adjacent patterns will be checked. If most of the peaks fall inside the ellipses, as shown in Fig. 1b, the Miller indices will be assigned to each peak in the new pattern, without calling the indexing algorithm. In the example, comparing the two patterns in Fig. 1a and b, three peaks are missing, resulting from the low signal to noise ratio of the intrinsically weak $0\bar{2}8$ and $\bar{3}\bar{1}9$ reflections, and/or the existence of blind areas on the detector such as for the $\bar{1}\bar{1}9$ reflection. In our computer program, user input parameters are therefore available to define a tolerance as a percentage of missing reflections. In this example, it is defined that if a reflection is found in 70% of the ellipses, these two patterns are regarded to be recorded from a same crystal grain. If most of the peaks, however, fail to fall inside the ellipses, as displayed in Fig. 1c, the indexing algorithm has to be called to obtain the Miller indices of all the peaks. Sometimes a couple of peaks may still fall into the ellipses even if the orientation changes, such as for the $\bar{2}06$ reflection in Fig. 1c, which is also taken into account in our program. In either case, once the Miller indices of each reflection is known, a rapid interior point algorithm [13] based orientation refinement is carried out to get the accurate crystal orientation, by minimizing the sum of the deviation angles between each experimental peak position and the calculated ones [12]. Afterwards a Nelder-Mead Simplex algorithm [14] based strain refinement is employed, similar to the one used in XMAS, to map the deviatoric strain distribution quantitatively. With this strategy, in theory, each crystal grain needs to be indexed only once, while for all other scanning points only refinements are necessary. Although in real case, more than one pattern needs to be indexed due to the significant Laue peak position change induced by high density of intragranular defects, the total analysis time can be still greatly cut down, no matter only single phase is considered, or multiple phases need to be indexed.

Because of the large penetration depth of the high energy synchrotron X-ray beam, diffraction may take place on more than one crystal grain and each grain will generate a set of Laue peaks (Fig. S2). Using the differential aperture method [15], the orientation distribution along the depth direction can be mapped, while aperture scan experiment and analysis take extremely long time. More conventionally, the strongest set of Laue peaks will be selected as the dominant signal at the scanning position, and its corresponding structure and orientation

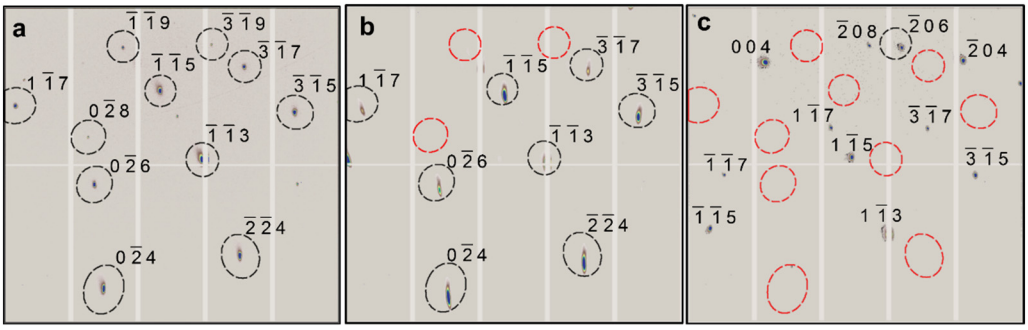

Fig. 1. A schematic explanation of the PPC method. (a) An indexed pattern with all Miller indices marked and the ellipses round each peak shows a tolerance region of 2°. (b) A Laue pattern taken in the same grain as (a), with all peaks falling in the ellipses except several missing ones. (c) A Laue pattern taken in a different grain.