Journal of Physics and Chemistry of Solids 110 (2017) 36–42

Contents lists available at ScienceDirect

Journal of Physics and Chemistry of Solids

journal homepage: www.elsevier.com/locate/jpcs

# Determination of contrast factors for cubic slip-systems and their application in the microstructural characterization of binary Fm$\overline{3}$m materials

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ARTICLE INFO

*Keywords:*
Contrast factors
Strain anisotropy
Distortion tensor
Williamson-Hall method
X-ray line profile analysis

ABSTRACT

Theoretical attempts to rationalize the strain anisotropy of crystalline systems in terms of dislocations often include the calculation of contrast factors. However, the evaluation of such parameters can be cumbersome because elastic properties and symmetry restraints must be simultaneously taken into account, especially when calculating the distortion tensor and the elastic contributions in slip coordinate systems. In this study, a dislocation-dependent coordinate system is introduced to obtain straightforward expressions for the evaluation of individual contrast factors by a first principles approach. Therein, we report the contrast factors for KCl and NaCl regarding edge and screw dislocations; a further analysis of their microstructure was carried out through the modified Williamson-Hall method.

## 1. Introduction

Diffraction methods are commonly used to characterize the microstructure of materials. X-ray diffractometry, for example, is a powerful tool to study the shape, size and distribution of crystallites; lattice faults and twinnings; and the arrangement and density of strain dislocations [1–3]. All the above information is simultaneously embedded within the sample's diffractogram, and thus several approaches to estimate apparent size parameters and mean square strain values have been proposed along the last few decades. The Williamson-Hall (WH) and the Warren-Averbach (WA) are two classical methods [4–8] that can describe the microstructure for bulk materials. However, several assessments to obtain microstructural information have resulted in the lack of a monotonic behavior [9–11], an effect which has been previously explained by strain anisotropy [12]. To effectively decouple the sample's size and strain contributions, Ungár and Borbély [13] modified the WH and WA methods with a scale transformation that accounts for strain anisotropy by making use of the contrast factors ($C_{hkl}$). The concept of a weight factor to distinguish each dislocation was described by Krivoglaz [14] and Wilkens [15], and further developed by Klimanek [16]. More recently, the meaning of these parameters has been interpreted as the *visibility* for each dislocation [17]. The accuracy and agreement of the microstructural parameters estimated by diffraction techniques is highly improved as a result of using such contrast factors, and can even be compared to those measurements carried out by transmission electron microscopy [18].

A fundamental step to implement the modified WH and WA methods relies on the calculation of individual contrast factors for each dislocation. Even though a parametric evaluation of these parameters has been previously implemented on cubic symmetries [19], its application does not transition directly to materials with more limiting degrees of freedom; that is, a parametric implementation gives rise to averaged contrast factors only. While such approach is suitable for materials like those featuring simple crystal Fmm$\overline{3}$ structures (e.g. Cu, Ni, Ag), it turns unreliable for more complex materials as it allows for non-physical slip-systems to be carelessly quantified. Binary rock-salt (Fmm$\overline{3}$) structures, for example, show additional restrictions in order to preserve the crystal's neutral charge, which consequently prevents some dislocations from occurring. Therefore, the use of a first principles approach to calculate individual contrast factors is necessary for such materials.

In this study, the Lekhnitskii complex potential [16,20] and the Stroh dislocation eigenvalues [21] were used in conjunction with a proposed dislocation-dependent coordinate system to obtain straightforward expressions for the computation of individual contrast factors. In particular, the distortion tensor for cubic slip-systems is described using a *stretched* coordinate system, which leads to the evaluation of the elastic component of the contrast factors in a more simple way. Thus, the contrast factors for binary Fm$\overline{3}$m materials are calculated from first principles and

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http://dx.doi.org/10.1016/j.jpcs.2017.05.027
Received 6 September 2016; Received in revised form 21 March 2017; Accepted 26 May 2017
Available online 30 May 2017

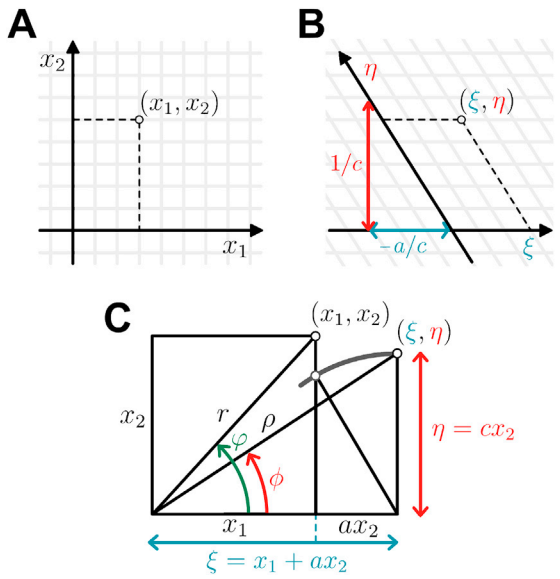


**Fig. 1.** Slip (A) and *stretched* (B) coordinate systems for a particular sextic polynomial root $p_\alpha = a + ic$. Geometrical relationships between both coordinate systems (C).

used to characterize the microstructure of KCl and NaCl salts through a modified WH analysis.

## 2. Theoretical basis

### 2.1. Modified Williamson-Hall method

The classic WH method [8] resolves both size ($\beta_p$) and strain ($\beta_s$) broadening contributions on real crystals by taking advantage of their different order dependence with respect to Bragg's angle ($\theta$). The former contribution occurs due to the finite size effects of the diffracting system, while the later one arises from its lattice distortions. More specifically,

$$\tilde{\beta} = \frac{1}{\tau} + 2\,\zeta\,\tilde{d} \tag{1}$$

where $\tilde{\beta} = \beta\cos(\theta)/\lambda$ and $\tilde{d} = 2\sin(\theta)/\lambda$ are respectively the integral breadth and plane spacing, both terms described in a reciprocal space, and $\lambda$ is the source wavelength; $\tau$ is the *apparent* crystallite size as originally defined by Jones [4], while $\zeta$ is the *apparent* strain as indicated by Stokes and Wilson [5]. Both $1/\tau$ and $\zeta$ correspond to the integral breadths of peak profiles related to crystallite size and micro-strain, respectively. In particular, $\tau$ was interpreted by Hall [6] as a characteristic length scale for the lattice regions which diffract coherently within the system, and $\zeta$ depends directly on the distribution curve which governs the system's strain. If the distribution is uniform and isotropic then $\zeta = 2\varepsilon$, where $\varepsilon$ is the maximum relative displacement ($\Delta d/d \equiv \Delta\tilde{d}/\tilde{d}$) of a lattice point from its ideal position. The modified WH method [13] broadens the scope of the original approach into systems on which strain anisotropy is significant. This approach proposes a proper scaling factor $\delta = \tilde{d}\sqrt{C}$ instead of $\tilde{d}$ as in eq. (1), with $C$ being the average dislocation contrast factor. In particular, $\tilde{\beta}(\delta)$ is to take a quadratic form as in

$$\tilde{\beta}(\delta) = \tilde{\beta}_0 + \tilde{\beta}_1\delta + \tilde{\beta}_2\delta^2 \tag{2}$$

where $\tilde{\beta}_0 \equiv 1/\tau'$, $\tilde{\beta}_1 \equiv 2\zeta'$ and $\tilde{\beta}_2 \propto \sqrt{Q}$, where $\tau'$ and $\zeta'$ are the modified WH parameters; $Q$ is the correlation coefficient between adjacent lattice points, often interpreted as the fluctuation $Q = \langle\rho^2\rangle - \langle\rho\rangle^2$ of the dislocation density, $\rho$, [15,22]. It is observed that eq. (2) can be reduced to a linear case as in eq. (1) when $Q$ is zero or negligible.

### 2.2. Contrast factors

The computation of the average contrast factor $C$ from each individual $C^n_{hkl}$, as extensively described by Armstrong and Lynch [17], can be performed as:

$$C \equiv \langle C^n_{hkl}\rangle = \frac{1}{N}\sum_{n=1}^{N} C^n_{hkl} = \frac{1}{N}\sum_{n=1}^{N}\sum_{K,L=1} G^n_{KL}E^n_{KL} \tag{3}$$

where $K$, $L$ are the indexes for the reduced form of each 4-rank tensor, and $N$ is the total number of degenerate slip-systems (see Appendix 1). If not all slip-systems are equally populated, appropriate weight factors should be calculated for each system and included to the overall ensemble [17]. Specifically, the right-hand side of eq. (3) is split into a geometric component $\boldsymbol{G} \equiv G_{KL} = G_{ijkl}$ and an elastic one $\boldsymbol{E} \equiv E_{KL} = E_{ijkl}$, respectively described by:

$$G_{ijkl} = \gamma_i\,\gamma_j\,\gamma_k\,\gamma_l \tag{4}$$

and

$$E_{ijkl} = \frac{1}{\pi}\int_0^{2\pi} T_{ij}\,T_{kl}\,d\varphi \tag{5}$$

with $\gamma$ being the direction cosines between the scattering vector and the slip coordinate system for a particular geometry and dislocation type (see Ref. [17]). $T_{ij}$ and $T_{kl}$ are the distortion tensors associated with the displacement field of each dislocation, with $\{i,\ k = 1,2,3\}$ and $\{j,\ l = 1,2\}$.

The calculation of the geometric components for each contrast factor is relatively straightforward [21] and thus it is the evaluation of eq. (5) which turns out computationally demanding. Even though most calculations like those carried out by Borbély et al. [23] make use of numerical methods to perform this task, a closed-form for the distortion tensor can be obtained by introducing a *stretched* coordinate system which readily integrates information about a particular dislocation —this within the Stroh-Lekhnitskii formalism. Moreover, $T_{ij}$ can be written as a linear combination of the distortion displacements, which takes a closed, symmetric form when expressed in this *stretched* coordinates as will be

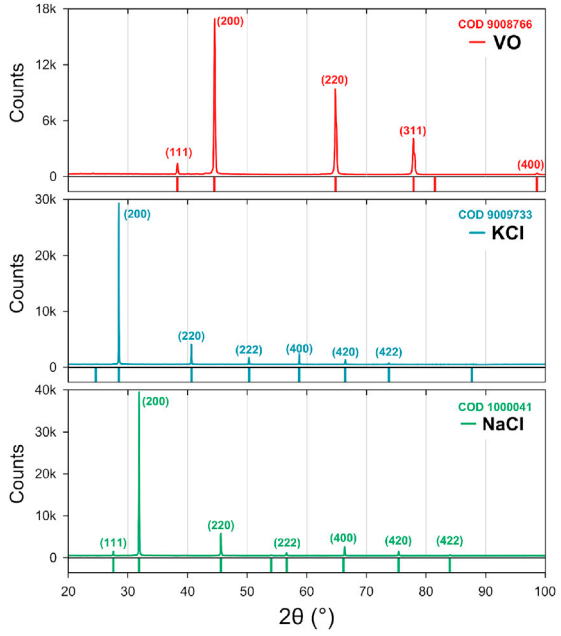


**Fig. 2.** Recorded diffractograms for a ready-made VO plaque and powder samples of KCl and NaCl. Their respective COD card is included at the bottom of each plot as a reference.