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The role of localized recoil in the formation of Kikuchi patterns

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ABSTRACT

In electron scattering from crystals, diffraction spots are replaced by Kikuchi patterns at high momentum transfer. Kikuchi pattern formation is based on the concept of effective incoherent electron sources (or detectors) inside a crystal. The resulting incoherence is a consequence of energy transfer connected with the momentum transfer in large-angle scattering events. We identify atomic recoil as a key incoherent process giving rise to electron Kikuchi patterns in the scope of the "channeling-in and channeling-out" model of electron backscatter diffraction (EBSD) and electron channeling patterns (ECP) in the scanning electron microscope (SEM). Using model calculations, we explore the characteristic role of the localization of the incoherent scattering event at specific places within the unit cell. In this way, we explain why sometimes inelastic losses do cause Kikuchi-type contrast, and sometimes inelastic losses result in the disappearance of this contrast in the SEM.

1. Introduction

Diffraction methods in the scanning electron microscope (SEM) are valuable tools for the analysis of the microstructure of materials. Depending on the way in which the diffraction information is obtained, we can distinguish two main techniques: electron channeling patterns (ECP) and electron backscatter diffraction (EBSD). EBSD has become a widely applied technique for the analysis of texture, strain and phase analysis [1,2], while the electron channeling technique is especially important in connection with direct imaging of dislocations in the SEM [3–6]. Comprehensive reviews of these methods can be found in [1–4].

Because both techniques are closely linked by the reciprocity principle, the formation process of electron channeling patterns and electron backscatter diffraction patterns can be discussed in a unified way in the context of the "channeling-in and channeling-out" model [7–10]. In this context, "channeling" means the result of coherent scattering producing diffraction effects that focus electron waves to different positions within the unit cell. The term "channeling" should not be misunderstood as the classical effect of electron trajectories being somehow confined to the open channels between the atomic planes (as is the case for ion channeling) [11].

The "channeling-in and channeling-out" model puts the atomic nuclei in a key position as they cause the large-angle scattering events required for the backscattered electron signal to be detected. In first approximation the probability of a large-angle deflection is given by the Rutherford cross section which is proportional Z^2, with Z the atomic number. The scattering probability can be modulated on the one hand by coherent scattering processes of the incident electron wave prior to the backscattering event. The coherent scattering and interference of the incident plane wave beam is setting up a wave field inside the crystal which can be described as a superposition of Bloch waves. This wave field, depending on the incidence angle with respect to a lattice plane, has a different overlap with the atomic nuclei which translates directly into the modulation of the total backscattering signal from the respective nucleus. By reciprocity, the same types of wave fields are describing the angular modulation of the exit probability of these backscattered electrons as a function of the final direction, i.e. the detection directions ending up on the phosphor screen. Thus coherent scattering of the backscattered electron wave after the backscattering event will also result in a redistribution of the angular intensity. If the backscattered signal is averaged over the exit direction in this unified "channeling-in and channeling-out" model, and we change the incoming direction then we obtain the method of ECP. If we keep the incident beam direction fixed, and study the variations in the outgoing intensity then we are looking at the method of EBSD [8].

It has been shown that the Kikuchi patterns seen in ECP and EBSD can be simulated [12–14,6] using the Bloch wave approach of dynamical electron diffraction well-known from transmission electron microscopy [15,16]. These simulations assume that the observed signal is proportional to the intensity of the wave field that is set up by a plane wave incident beam measured at the atomic positions. By reciprocity, the incident plane wave in ECP

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corresponds to an exit wave towards a specific point on the phosphor screen in EBSD.

In order to separate the coherent scattering in the incident part from the coherent scattering in the outgoing part of the total process, the backscattering event needs to break the coherence between these two parts. This means that the phase relationship between the incident wave and the exit wave has to be randomized by the backscattering process. It is instructive to realize that without the incoherent event, we would be simply looking at electron diffraction from a crystal which is described by collective coherent scattering from the crystal atoms. This would result in the conventional spot patterns of transmission high energy electron diffraction (THEED), low energy electron diffraction (LEED), and reflection high energy diffraction (RHEED). These, essentially coherent, techniques are, of course, always additionally influenced by incoherent scattering, which becomes especially important for a quantitative description of transmission electron microscopy measurements in the presence of phonon scattering [17,18]. This type of scattering is closely linked to the Kikuchi patterns which are observed in ECP and EBSD.

The nature of the virtual incoherent source inside the crystal which produces Kikuchi patterns is often discussed in rather general terms as caused by "inelastic scattering" or "diffuse scattering". One aim of this paper is to discuss recently obtained clear-cut experimental evidence for the pivotal role of recoil in the formation of Kikuchi patterns. Large-angle scattering of high energy electrons involves significant momentum transfer $\boldsymbol{q}$ (and thus an energy transfer $q^2/2M_a$ with M_a the atomic mass) from the incident electron to the incoherently backscattering atom. Experimentally, this recoil energy can be used in a compound crystal to determine the scattering atom. Thus site-specific diffraction information is obtained from the quasi-elastic electrons [19] and this observation suggests new ways to use diffraction for crystallographic analysis in the SEM.

The purpose of this paper is to show (a) the implications of the necessary momentum transfer from the incident electron to the nucleus in the backscattering process, (b) the role of the specific localization of incoherent scattering at different positions inside the crystal unit cell and (c) that an increasing randomization of the incoherent source position over the whole volume of the unit cell leads to the suppression of diffraction information because, effectively, many different possible Kikuchi patterns are averaged in the resulting pattern which is measured. Thus we obtain an understanding when energy losses result in Kikuchi pattern formation, and when it washes them out. This understanding is important as a description of the specific suppression of diffraction information by inelastic scattering in an otherwise perfect crystal is necessary for an improved quantitative simulation of experimental Kikuchi patterns.

The suppression of diffraction information by inelastic scattering was experimentally established in the measurement of the contrast in Kikuchi patterns as a function of the energy loss [20,21]. A clear reduction in diffraction contrast was found when the outgoing trajectory was longer than the inelastic mean free path [21]. Thus a Kikuchi pattern obtained without energy discrimination (e.g. when a phosphor screen is used) contains a large background of inelastically scattered electrons without any diffraction features.

2. Results

2.1. Quasi-elastic backscattering

In order to demonstrate the decisive role of recoil in Kikuchi pattern formation of the quasi-elastically scattered electrons, we show in Fig. 1 results of angle-dependent electron spectroscopic measurements near zero energy loss from a sapphire sample with a small number of Au atoms deposited on the surface (Au coverage well below a monolayer). These experimental results have been obtained using high resolution electron spectroscopy and analyzed with dynamical Bloch wave simulations as described in detail in [19,21,22].

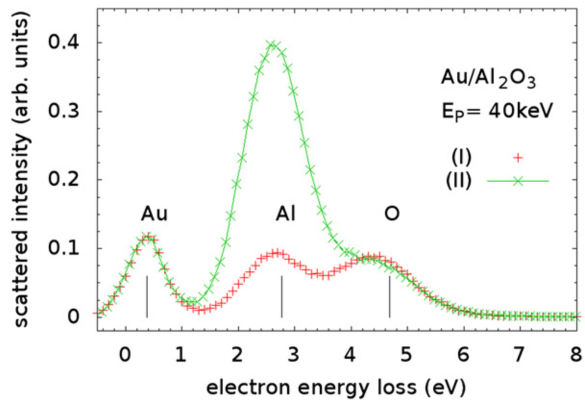

Fig. 1. Energy spectrum of quasi-elastically backscattered electrons from a sapphire (Al_2O_3) sample with gold atoms deposited on the surface. The vertical lines indicate the recoil loss expected for elastic electron–atom scattering. Curves (I) and (II) are measurements in two different directions. The difference between (I) and (II) is due to diffraction effects.

As can be seen in the data in Fig. 1, the backscattered electron energy distribution shows three peaks which can be assigned to scattering by Au, Al and O, respectively. The vertical lines show the expected electron energy loss of $q^2/2M_a$, i.e. what is expected for a classical, billiard-ball type, collision. The agreement of the classical expectations and the observed peak positions is very good, the remaining differences can be assigned to charging of the sapphire sample which results in a reduced scattering energy of about 35 keV [19].

In a quantum-mechanical picture, the recoiling atomic nucleus can be described by a superposition of many simultaneously excited phonons which take up the necessary recoil energy [23]. As a recoil energy of $\approx$ 1 eV (typical for backscattering from light elements at SEM energies) can excite many, nearly energy-degenerate, combinations of phonons, the exact phase relationship between the phonons and the electron is not tractable in each scattering event and the electron wave is thus incoherently scattered by each individual recoiling atom. The time evolution of these phonons results in oscillations spreading from the initially displaced atom over the whole crystal, corresponding to thermal dissipation of the recoil energy. For comparison, the completely coherent reflection of the incident electron by the crystal lattice would result in a single peak at the primary beam energy, since the recoiling mass is macroscopic and the recoil energy loss is infinitesimal. In this case, we would have no information on the mass of any individual atom involved in scattering. The probability of this elastic coherent scattering is described by the Debye–Waller factor, which exponentially decreases with the square of the momentum transfer. Thus, due to the large momentum transfer in the backscattering geometry, elastic coherent backscattering is largely suppressed in the typical EBSD geometry.

In combination with the recoil loss, we simultaneously investigated the influence of diffraction effects. These are seen in the two experimental curves (I,II) presented in Fig. 1 which are recorded for two different exit directions and a fixed incident beam. While Al shows an increase in intensity from (I) to (II), the O intensity is showing the opposite trend (taking into account that the O peak is on the wing of the Al peak). The Au atoms show the same intensity in both cases, which is consistent with the