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Experimental evidence concerning the significant information depth of electron backscatter diffraction (EBSD)

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ABSTRACT

Experiments concerning the information depth of electron backscatter diffraction (EBSD) are performed on samples featuring an amorphous wedge on a crystalline substrate and a crystalline wedge on an amorphous substrate. The effects of the acceleration voltage and exemplary software settings on the ability to measure through an amorphous layer are presented. Changes in the EBSD-signal could be detected through a ≈142 nm thick layer of amorphous Si while orientation measurements could be performed through a ≈116 nm thick layer when using a voltage of 30 kV. The complexity of the information depth significant to a given EBSD-pattern and the multiple parameters influencing it are discussed. It is suggested that a "core information depth" is significant to high quality patterns while a larger "maximum information depth" becomes relevant when the pattern quality decreases or the sample is inhomogeneous within the information volume, i.e. in the form of partially crystalline materials or crystal layers in the nm scale.

1. Introduction

While Electron backscatter diffraction (EBSD) has become a standard method in scanning electron microscopy (SEM), the experimental evidence concerning the information depth of the method is doubtable as recently outlined [1]. A statement that the information depth of EBSD ranges from 10 to 40 nm [2] is frequently cited in the literature. However, it is based on the assumption that the entire energy loss of electrons contributing to an electron backscattering pattern (EBSP) is caused by diffraction [2]. By contrast, EBSD has been described as a two-step process of incoherent backscattering followed by diffraction on the outgoing path [3], which seems to be a more logical model.

Experimental results obtained by placing an electrostatic energy filter between sample and EBSD-camera show that electrons with only 80% residual energy may still contribute to an EBSP [4], i.e. a residual energy of 16 kV if the acceleration voltage is 20 kV. EBSPs have been obtained using incident electron beam energies as low as 3 keV using a digital complementary metal-oxide semiconductor (CMOS) hybrid pixel detector which allows direct electron detection and energy filtering without a phosphorous screen [5]. While it would appear logical that the backscatter signal of an electron beam, e.g. 20 kV, contains a significant quantity of electrons with residual energies larger than these 3 keV, e.g. 15% of the incident energy, cut off values of more than 95%, e.g. ~98.5% [3], are frequently used in simulations resulting in very small information depths. The detailed critique of the existing literature in Ref. [1] was accompanied by a detailed discussion of what the information depth actually is and principle experimental setups and requirements necessary for the analysis [1].

In short there are at least three relevant information depths for EBSD: the depth relevant for any signal change from the EBSD-camera (i.e. proving crystallinity), the depth relevant for orientation analysis and finally the depth relevant for strain measurements [1]. The recent utilization of the EBSD-detector as an information source for imaging [6] has increased the importance of the full information depth responsible for any signal change. The three principal experimental setups which can contribute information to the topic are discussed in Ref. [1] and outlined in Fig. 1: a) an amorphous layer covering a crystalline substrate, b) a crystalline wedge covering an amorphous substrate and c) an amorphous wedge covering a crystalline substrate. Please note that none of them directly measure the information depth of EBSD.

Setup a) was e.g. applied when sputtering amorphous Cr on a crystalline Si (*c*-Si) substrate [7], but a simulation of this setup shows that only a small fraction of the backscatter electrons (BSE) actually propagated into the crystalline substrate due to the high density of Cr,

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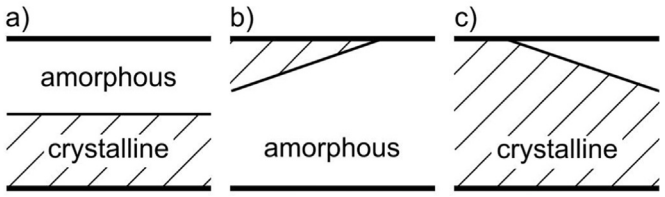

Fig. 1. Principal experimental setups for measurements concerning the significant information depth of EBSD.

making this material pair unsuitable for the analysis. Ideally the amorphous and crystalline phases should show identical properties, i.e. same density, electrical and thermal conductivity etc. especially with respect to electron interaction. While this is of course impossible, some pretty close approximations exist as will be outlined below. Setup a) additionally poses extreme challenges for sample preparation: reproducible, plane-parallel layers in the nm scale without any porosity that must be applied on the substrate without any damage to its crystal lattice. Any damage to the substrate lattice would be interpreted as a thicker amorphous layer and lead to incorrect results.

The setups b) and c) in contrast deliver relative results where the EBSD-pattern qualities for 0% and 100% crystal lattice within the information volume of EBSD may be acquired [1]. Here the challenge lies in defining discrete boundaries in a continuous system. Setup b) can only provide information on the minimal amount of crystal lattice in the information volume necessary to produce an EBSD-pattern while setup c) can provide the maximum thickness of the amorphous top layer through which EBSD-patterns may still be acquired. However, the diffracted electrons penetrating the amorphous layer to form an EBSD-pattern must have at least interacted with the minimal amount of lattice necessary to produce an EBSD-pattern. Hence the information depth of EBSD must at least be as large as the combined thicknesses obtained from the setups b) and c).

A hematite containing glass-ceramic has been used to show that the information depth of EBSD using a sample tilt of 70° and an accelerating voltage of 20 kV is comparable to that of an SEM-micrograph obtained from an untilted sample using 4 kV in the used setup [1]. This means that a layer of 100 nm may measurably contribute to an EBSD-pattern of Si obtained with a voltage of 20 kV [1], which is more than twice the thickness frequently cited [2]. While the material pair of hematite and its glass matrix is sufficient to provide this rough estimate, preparation problems such as a topographical edge of 30 nm at the glass-crystal interface and the necessary carbon coating [1] make it unsuitable for more detailed measurements.

By contrast, the deposition of amorphous Si (a-Si) layers [9–12] on Si single crystals provides an improved material pair. The amorphous state of Si thin films deposited by thermal evaporation can be controlled via the substrate temperature and can be proved separately by Raman-investigations as well as by X-ray diffraction analysis [13]. Amorphous Si films show a topography if applied to a rough substrate [12] and can absorb gases due to a microporosity [12]. Using polished substrates reduces both the topography and gas adsorption [12] but the density of the a-Si remains lower than that of c-Si and depends on the deposition method [12]. While there are some differences between the material properties of crystalline and amorphous Si as illustrated by the selected properties stated in Table 1, the good adhesion between these phases is a great advantage for the preparation of the respective wedges outlined in Fig. 1. The specimen heating caused by electron bombardment during SEM analysis is usually considered insignificant for inorganic samples [22] and only the mass density and the atomic number should be relevant for the energy loss of accelerated electrons in solid materials [23]. However, the EBSD-pattern degradation observed during the analysis of some glass-ceramics [24] as well as the substrate destruction [25] or swelling [26] observed in sensitive systems with relatively low thermal conductivities has drawn the general validity of these assumptions into question for EBSD applications.

Table 1
Selected properties of crystalline (c-Si) and amorphous silicon (a-Si) at room temperature.

	c-Si	a-Si
atomic number:	14	14
mass density:	2.33 g/cm^3	2.18 [12]–2.26 [14] g/cm^3
electrical resistivity:	10^{-3}-10^4 W/Ω cm	10^4–10^8 W/Ω cm[15]
thermal conductivity:	156 W/(m K) [16]	1.5 W/(m K) [17]
specific heat capacity:	703 J/(kg K) [18]	992 J/(kg K) [17]
Young's modulus:	130–188 GPa [19]	80[20]–140 [21] GPa

One argument sometimes used to support the limited information depth of EBSD is that only low loss electrons can contribute to an EBSP because otherwise the resulting spectrum of electron energies would lead to a spectrum of Bragg-angles which would prevent sharp Kikuchi band edges. For example, D. Dingley described a maximum energy spread of 250 eV in a high quality EBSP obtained from Si with 20 kV based on the detected line width [2]. However, even considering only low loss still electrons leads to a spectrum of Bragg-angles, because perfectly discrete Kikuchi bands can only be achieved with electrons of a single energy. As this would represent the perfect theoretical case, impossible to achieve in an actual experiment during general EBSD-application, the “sharpness” of a Kikuchi band is in fact only a question of perception or the ability to measure it. It has been proven that orientation measurement [27–29] or the proof of crystallinity [30] may be successfully performed on low quality patterns without edges as discrete as those e.g. in the pattern evaluated in Ref. [2]. EBSD has even been performed in wet-SEM conditions [31].

Determining the true information depth is relevant considering that EBSD is frequently applied to thin film systems. Another relevant field is the analysis of twinning systems on the nm-scale [32,33]. Additionally, the ability to perform standard EBSD-measurements through thin layers of glass [1,24] or acquiring EBSD-patterns from crystals *below* a 100 nm thick layer of Si_3N_4 using 30 kV [34] cannot be explained by the widely assumed information depth of EBSD. Most recently, indexable EBSD-patterns were obtained from crystals bellow a 30–40 nm thick layer of ZrO_2 using a voltage of 20 kV [29].

Although the concepts concerning the information depth presented in this article may also be relevant to the so called transmission-EBSD (t-EBSD) [35–37], the values are not transferable because t-EBSD utilizes the forward scattering signal while EBSD utilizes the back-scattering signal of the SEM.

2. Materials and methods

The primary samples were prepared by coating polished boron doped (001) Si wafers (resistivity 1–20 Ω cm) with a 4.7 µm thick layer of amorphous Si. The substrates were cleaned *ex situ* by the “RCA-method” and *in situ* by an electron beam dry clean etch [8] before coating. The deposition was performed in the Fraunhofer FEP batch tool “ELMASCAN” by crucible-free electron beam physical vapor deposition. Here an electron beam from an axial e-gun (*CTW*-type) is applied to a rod of high purity electronic grade Si (resistivity 15,000 Ω cm) at a process pressure of 1×10^{-3} Pa [9,10]. The rod was carefully arranged so as to minimize the contamination contact and thermal contact. A low electron beam power input was applied to the Si rod top face until the temperature increased to 500–1000 °C where Si undergoes a brittle-to-ductile transition associated with increasing the plastic flow. The transition temperature depends on the applied type of silicon and the strain rate [11]. After reaching the transition temperature of the crystal rod, the electron beam power was increased to achieve evaporation conditions to the tool maximum of $P_{EB.max}$=2.55 kW and substrates mounted on a portable carrier were