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Energy-selective neutron imaging with high spatial resolution and its impact on the study of crystalline-structured materials

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ABSTRACT

Crystalline-structured materials with preferentially large grains were investigated by means of energy-selective neutron imaging methods (transmission radiography and tomography) under the conditions of the best possible spatial resolution at the ICON facility, SINQ, and PSI. Because of the cold spectrum at that beam line, access to the Bragg diffraction features was possible even when the energy resolution of the used selector device was only 15%. Grains with a size below the detector resolution (approximately 25 μm) are not visible, and a quasi-homogeneous contrast variation is found when the neutron energy is varied.In the cases of welded stainless steel samples and rolled Al plates, we obtained structural information from a very short exposure of approximately 60 s. Tomographic examinations of these samples at suitable neutron energies qualitatively verified the radiographic findings by showing the same features in the bulk. Comparison to common electron backscatter diffraction (EBSD) investigations in selected regions of the samples provided a complete verification of the neutron-image data with respect to the grain size and the different grain orientations. The method of energy-selective neutron imaging provides an easy and straightforward approach for non-invasive material research that can be performed without any sample preparation if the most suitable neutron energy is chosen. Further studies will be necessary to extend the experimental data base to other materials with different crystal structures and grain sizes. A comparison to diffraction data will enhance the quantitative value of the investigations.

1. Introduction

Neutron-imaging investigations are commonly performed in transmission mode with a "white"=polychromatic thermal or cold beam at a suitable beam line. In the present day, the resulting image data are stored in a digital format that is provided by the detection system, which is, in most cases, camera based. Details on state-of-the-art neutron-imaging techniques can be found elsewhere [1,2]. Such investigations are very useful for, e.g., the non-destructive examination of objects on the macro-scale, similar to X-ray inspections. There is already a high potential for industrial application, which can be increased when neutron imaging becomes more common and suitable facilities become accessible to external users, including industrial partners.

The challenge of narrowing the neutron spectrum in the beam is mainly motivated by the properties of engineering materials, in particular, crystalline-structured metals (Fe, Al, Cu, Zr, Ni, etc.). They all diffract neutrons at the lattice planes in the grains of the material, which have different orientations and sizes in different materials. This scattering behavior follows Bragg's law (wavelength λ, scattering angle θ, and lattice plane distance d):

$$2d_{hkl}\ \sin\ \theta = \lambda \tag{1}$$

The determination of these lattice parameters has been the subject of various specific diffraction experiments that have used either neutrons or X-rays [3,4].

If the material under investigation is polycrystalline and has very small grain sizes (less than the normal imaging-detector resolution, e.g., 0.2 mm)—the measurement regime called the powder approximation—the transmission image of the sample shows uniform but varying contrast over the samples for all energies.

However, the resulting attenuation corresponds to the total cross section, which is dominated by the elastic coherent cross-sections σ_{coh}^{el}. It can be described theoretically as follows:

$$\sigma_{coh}^{el}(\lambda) = \frac{\lambda^2}{2V_0} \sum_{d_{\vec{h}}=0}^{2d_{\vec{h}}<\lambda} \left|F_{\vec{h}}\right|^2 d_{\vec{h}} \tag{2}$$

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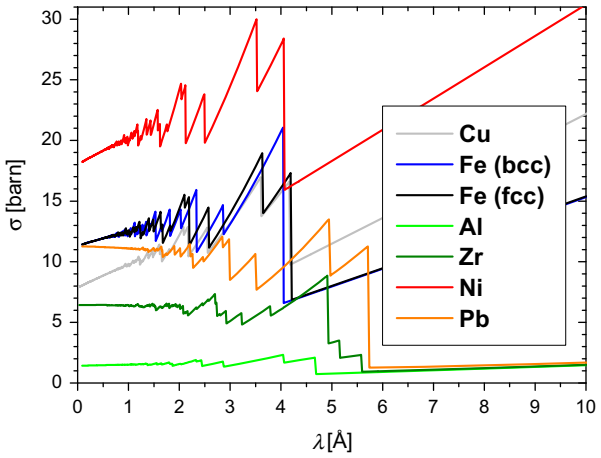

Fig. 1. Elastic neutron-scattering cross-sections of relevant crystalline-structured materials in the cold-neutron range based on the basic principles of scattering at lattice planes (powder approximation).

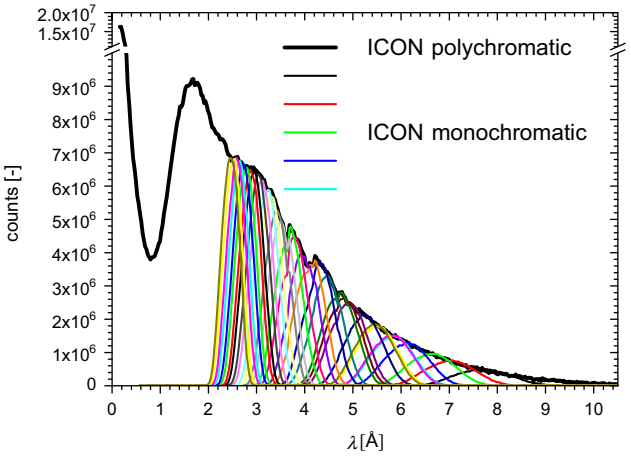

Fig. 2. Neutron spectrum obtained at the ICON imaging beam line with the white beam and the different partial spectra obtained with the turbine selector by changing the rotation frequency.

This formula represents the superposition of all diffractive contributions of the grains with specific lattice parameters up to the Bragg edge for the lattice-plane vector **h**. The structure factor F is specific to each material and each lattice plane. Some data for crystalline-structured materials are plotted in Fig. 1. The superposition of the scattering at lattice planes of different orientations and by the different involved families of planes described by the Miller (hkl) indices in (1) end when no more diffraction occurs according to (1)—the Bragg cut off.

It is well known that many practically used materials deviate in their structural properties from these ideal micro-crystalline features by the presence of assemblies of larger grains and in their preferred orientation. These properties are caused by the manufacturing and treatment procedures and need not be seen as drawbacks for the utilization and behavior of these materials. A characterization of these materials in bulk structures in a non-destructive way might, however, become essentially for the evaluation and understanding of their strength and long-term performance.

As is also visible in Fig. 1, the majority of the pronounced "Bragg edges" are in the cold-neutron energy range (from 2 to 6 Å). Therefore, the enhancement of structural features by narrowing the energy band of the arriving neutrons is important in that range and near the Bragg edges of the materials under investigation to allow measurements to focus on selected lattice planes only. In this manner, specific grain structures can be investigated when their size becomes greater than the spatial resolution of the neutron-imaging detection system.

First trials for energy-selective imaging were conducted at ENGIN-X and ROTAX at ISIS in the UK [5]. Although a time-of-flight (TOF) technique was applied in these studies, the image quality (resolution and signal/noise ratio) was not high enough to see structural details sufficiently clearly to be comparable to standard material-science techniques such as electron backscatter diffraction (EBSD). The image blurring has been attributed mainly to the poor beam collimation and performance of the preliminary detection system that was used.

The surprising findings under these conditions were local intensity changes in the transmission images accompanied by global changes over the whole of the quasi-homogeneous samples when different energies were resolved. However, the spatial resolution of only 0.3 mm caused by the beam divergence and the detector blurring did not allow a more quantitative evaluation.

In the meantime, it was found that when the energy resolution is even poorer than in the TOF studies, but a better image quality is provided by using an optimal detection system and improved beam properties, similar image features are obtained [6].

This paper will describe the investigation of steel and aluminum samples with a clear macro-crystalline structure larger than the pixel size of the detector; measurements were obtained in energy-selective mode using a turbine-type selector [9] with 15% resolution in $\Delta\lambda/\lambda$. The spatial resolution was the best that presently can be achieved [7]. As a result, the resulting imaging data could now be compared to EBSD measurements, which were performed in PSI labs for representative regions of the samples.

Because transmission-image data integrate over the sample volume in the beam direction, and the EBSD data describe only the surface properties of the investigated sample, there was no real bulk information obtained regarding the samples. Therefore, attempts were made to study the samples in 3D by extracting volume information from many single angular projections and applying standard filtered back-projection tomography reconstruction tools. Even if a general mistake may be contained in the reconstruction algorithm–the preferred orientation results in specific angular-dependent neutron reflections in conflict with the simple common attenuation law–it was possible to draw some convincing conclusions about the bulk properties.

2. Experimental setup

For energy-selective imaging, we used the imaging facility ICON [8] at the Swiss spallation neutron source SINQ, which provides high amounts of cold neutrons. The selection of narrowed energy bands was performed by using a turbine-type device [9] that enables a 15% resolution in $\Delta\lambda/\lambda$. The spectra obtained at ICON with the white beam and those behind the selector, which were measured by means of a chopper spectrometer, are compared in Fig. 2.

The transmitted beam intensity was registered with the high-resolution micro-tomography detection device [7] installed at the middle position of ICON.

This device provides 2048 × 2048 pixels, each with a 13.5 μm pixel size. In this respect, the image resolution is better than in previous studies at ISIS by a factor of more than 10. The integration time was between 30 and 120 s, depending on the installed scintillator screen and the chosen wavelength.

An overview image of ICON is given in Fig. 3, in which the selector and detector positions are indicated. The specifications of the setup are summarized in Table 1.