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Review

In-situ observation of crystal surfaces by optical microscopy

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Abstract

In this experimental course, attendees will learn how to obtain useful information about growth processes of crystals using ordinary optical microscopes, which are usually available in laboratories. We will demonstrate how thicknesses of crystals can be estimated from interference colors. We will also show in-situ observations of spiral steps and strain distributions by differential interference contrast microscopy and polarizing microscopy, respectively. © 2016 Elsevier Ltd. All rights reserved.

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1. Introduction

In many cases, crystals are bounded by flat facets (low index faces), and such crystals grow layer by layer, as illustrated in Fig. 1 [\[1,2\].](#page--1-0) We deal with such growth processes. First, we show how to measure thicknesses of plate-like $CdI₂$ crystals utilizing their interference colors. Next, we observe bunched spiral growth steps on spiral growth hillocks of $CdI₂$ crystals using a differential interference contrast microscope. Then, we also observe strain distributions around screw dislocations on SiC crystal surfaces using a polarizing microscope. Through these observations, we hope that attendees realize how ordinary optical microscopes are beneficial for obtaining valuable information about crystal growth. For general information about optical microscopy, see references [\[3,4\].](#page--1-1)

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Fig. 1. A schematic illustration of the layer growth of a crystal.

2. Measurement of thicknesses of plate-like crystals using interference colors

First please remind yourself of the principles behind Newton's rings [\(Fig. 2A\)](#page-1-0). An illuminating light beam is reflected from two glass-air interfaces. When the difference R in the light paths of two reflected light beams is equal to the integral multiple i of a wavelength λ , interference of the light occurs. In the case of a monochromatic light beam, with increasing *R*, the interfered light shows repeating dark and light contrasts, as shown in [Fig. 2B.](#page-1-0)

In contrast, in the case of a white light beam consisting of light beams of all visible wavelengths, each light

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Fig. 2. Newton's rings formed by monochromatic light (A), interference of monochromatic light (B) and interference colors of white light (C). (A) A cross-sectional illustration of a setup is also shown. *λ* is a wavelength, *t* a height difference in a hemisphere glass, *R* an optical path difference, and *i* an interger. (B) *d* is a thickness of a plate-like crystal and *n* the refractive index of a plate-like crystal (C) $|n_a - n_c|$ shows an absolute value of the difference in refractive indexes in the *a-* and *c-*directions. The color charts in B and C were taken from reference [\[5\]](#page--1-2) and modified. (For interpretation of the references to color in the text, the reader is referred to the web version of this article.).

beam with a certain wavelength shows repeating dark and light contrasts, with increasing *R*: a blue light beam presents dark, blue, dark, and blue, and a green light beam depicts dark, green, dark, and green (Fig. 2B). As a result, the interfered white light shows an "interference color" that is determined by the summation of the interfered light of all visible wavelengths [\[5,6\].](#page--1-2) For example, since the interfered light of zero order $(R \sim 0)$ shows a dark contrast irrespective of wavelength, interfered white light presents a black color (Fig. 2C). In contrast, when *R* = 200– 300 nm, all visible wavelengths are in the light contrast conditions. Hence, interfered white light shows a white color. With further increases in *R*, the interference color changes as shown in the color chart in Fig. 2C.

A thin plate-like crystal follows the same principle of the Newton rings. Two light beams reflected from top and bottom surfaces of a plate-like crystal show an interference color. By comparing the interference color of a sample crystal and that shown in the color chart (Fig. 2C), we can evaluate the optical pass difference *R*. Hence, if the refractive index *n* of a sample plate-like crystal is known, one can obtain the thickness *d* of the crystal. When $R \ge 1500$ nm, the interference color becomes white, showing that one cannot evaluate *R* from the interference color any more. In other words, if a transparent platelike crystal shows a certain interference color, *R* is smaller than 1500 nm.

3. Observation of spiral growth hillocks using a differential interference contrast microscope

Differential interference contrast (DIC) microscopy provides three-dimensional (3D) contrast using the interference of light [\[7\].](#page--1-3) [Fig. 3](#page--1-4) shows a schematic optical

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