Ultramicroscopy 186 (2018) 49–61

Contents lists available at ScienceDirect

Ultramicroscopy

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

Transmission scanning electron microscopy: Defect observations and image simulations

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ARTICLE INFO

Article history:
Received 9 July 2017
Revised 2 November 2017
Accepted 13 November 2017
Available online 6 December 2017

Keywords:
STEM

ABSTRACT

The new capabilities of a FEG scanning electron microscope (SEM) equipped with a scanning transmission electron microscopy (STEM) detector for defect characterization have been studied in parallel with transmission electron microscopy (TEM) imaging. Stacking faults and dislocations have been characterized in strontium titanate, a polycrystalline nickel-base superalloy and a single crystal cobalt-base material. Imaging modes that are similar to conventional TEM (CTEM) bright field (BF) and dark field (DF) and STEM are explored, and some of the differences due to the different accelerating voltages highlighted. Defect images have been simulated for the transmission scanning electron microscopy (TSEM) configuration using a scattering matrix formulation, and diffraction contrast in the SEM is discussed in comparison to TEM. Interference effects associated with conventional TEM, such as thickness fringes and bending contours are significantly reduced in TSEM by using a convergent probe, similar to a STEM imaging modality, enabling individual defects to be imaged clearly even in high dislocation density regions. Beyond this, TSEM provides significant advantages for high throughput and dynamic in-situ characterization.

1. Introduction

Analysis of the interaction of energetic electrons with crystalline matter in the transmission electron microscope (TEM) has led to unprecedented insights on the character of crystalline defects and their influence on the properties of a broad spectrum of materials. The TEM as an imaging, diffraction and microanalytical characterization instrument has undergone tremendous evolution over the decades, now capable of atomic-scale chemical and structural characterization. From the earliest designs [1], the characterization of dislocations, 1-D line defects that strongly influence structural, electrical and optical properties of materials [2,3], has been a priority. However, full defect characterization over multiple samples by conventional TEM (CTEM) typically requires substantial effort and beam time, particularly if knowledge of the dynamic aspects of dislocation motion and their interaction with other defects is required. In recent years, imaging of dislocations in the scanning transmission mode in the TEM (STEM) has also been considered [4], with the benefits of analysis of thicker specimens and the suppression of dynamical effects that can interfere with defect analysis.

While it has become routine to image line defects in the TEM, scanning electron microscopes (SEMs) have been used less frequently for this purpose. SEMs are typically used to analyze bulk samples, thus limiting defect observations to the near surface region; hence, the SEM has had only limited applicability for defect analysis. It was recognized in the 1970s that defects could be imaged in an SEM with the electron channeling contrast imaging (ECCI) technique [5], but the use of this technique has been relatively rare. ECCI has seen a recent resurgence enabled by high quality electron probes in FEG source SEMs [6]. ECCI has been used in research on metals [7,8], as well as semiconductors [9], where defects on the order of 1 μm below the sample surface can be detected [10]. The cathodoluminescence effect [11] has also been used to study defects at or near the surface. Nonetheless, despite the resurgence in the use of SEM-based techniques for defect observation, SEM-based defect studies have been rather limited.

Recently, solid state scanning transmission electron (STEM) detectors have become available for use in SEM instruments. These

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https://doi.org/10.1016/j.ultramic.2017.11.004
0304-3991/

detectors, combined with high quality field emission sources, now enable transmission imaging in the SEM, opening new pathways for more rapid defect analysis and observations of dynamic phenomena. The development of efficient sample mounting configurations enables users to load six or more samples into the SEM chamber simultaneously, making high throughput observation of many samples possible in a single vacuum cycle. Furthermore, the large vacuum chambers available on most SEMs provide versatility for in-situ experiments where the sample may experience thermal loads, mechanical deformation, electrical probing, and large applied electromagnetic fields. Many of these types of in-situ experiments are simply not possible or much more difficult in a TEM; unique hardware may be required due to the confined specimen port size, which is restricted by the proximity of the specimen between the objective lens.

A STEM detector can be used in concert with other detectors present in the SEM chamber, such as an electron backscatter detector (EBSD), either in the standard backscatter configuration or as a transmission Kikuchi diffraction (TKD) detector, conventional backscatter electron (BSE) detectors, secondary electron (SE) detectors, cathodoluminescence detectors, electron beam induced current (EBIC), and analytical detectors such as energy-dispersive x-ray spectroscopy (EDS) wavelength dispersive spectroscopy (WDS) detectors. It is relatively straightforward to study large thin foils in the SEM due to the large fields of view available with many SEM electron columns, and the large range of motion available with many SEM sample stages. Another advantage is that electron beam and oxygen sensitive materials can be studied. Typical accelerating voltages used in SEM are low enough not to damage the sample; however, above about 100 kV, lower Z materials start to exhibit damage [12]. Thus, materials that may be damaged at TEM accelerating voltages, such as magnesium, aluminum, or carbon nanotubes, can be studied in transmission mode in an SEM without defects being introduced by the electron probe during the observation process. With oxygen sensitive materials, if a dual beam FIB-SEM is equipped with a STEM detector, oxide growth can be avoided by excising a foil with the FIB column and then imaging with the electron column and STEM detector, all without exposure to air. Furthermore, significantly less training is required for SEM users than for TEM users, increasing access to defect observations to a wider range of researchers and enabling more diverse experiments.

For these reasons, based on both practical and fundamental imaging considerations, the prevalence of methods using transmitted electrons for imaging in an SEM environment (transmission-SEM or TSEM) has surged in recent years. Despite the introduction of transmitted electron detectors in commercial SEMs dating back to 1968 [13], many of the early efforts suffered from limitations in the detector technologies and control of the scattering geometries collected for imaging [13–26]. With the advent of commercially available solid state STEM detectors offering high sensitivity and flexibility of the scattering acceptance angle geometries (e.g. annularly segmented or stage-positioned diodes), the benefits of TSEM have been rekindled [25,26], with some of the early applications favoring biological and soft materials that suffer from low contrast and beam damage from the high-voltage TEMs [17]. In these cases, imaging based on high-angle scattering to reveal mass-thickness contrast was desired.

In parallel, recent work has shown the advantages of STEM imaging in TEM environments for defect analysis, generating images using relatively low scattering angles where diffraction contrast dominates. Defect contrast during STEM defect contrast imaging approaches benefit from reduced confounding dynamical effects, such as bending contours and thickness fringes [27–29], the reduction in zig-zag contrast from inclined dislocations [28], and the ability to retain sharp contrast even in relatively thick specimens [30,31]. The latter is particularly important in the adaptation of lower voltage SEMs for TSEM. In addition, STEM has improved signal to noise ratio compared to CTEM imaging due to the convergence of the beam [4].

In the present work, the efficacy of TSEM using commercially-available instrumentation is demonstrated for defect analysis, including stacking faults and dislocations. Simulations of TSEM images of defects, using methods developed elsewhere [4,32,33], are performed and compared to images of the same defect types collected using TSEM. Imaging modes that are similar to conventional TEM (CTEM) bright field (BF) and dark field (DF) are explored, and some of the differences due to the different accelerating voltages are highlighted. A methodology for the use of STEM diffraction in conventional SEM is developed and defect contrast observations are reported at SEM accelerating voltages.

2. Methods

2.1. Experimental

This study focuses on characterization of extended line and planar defects using TSEM along with CTEM, STEM, and simulations. We have studied stacking faults in a crept cobalt-base single crystal, dislocations in a femtosecond laser machined strontium titanate sample, and dislocations in a cyclically loaded nickel-base superalloy.

A precipitation strengthened single crystal cobalt-base superalloy with composition 79Co-6.7Al-8.1W-6.2Ti (atomic percent) was grown by the Bridgman technique. Samples of the single crystal were crept at 900 °C at a stress of 310 MPa, which produced a high density of deformation-induced superlattice intrinsic stacking faults in the ordered L12 precipitates. For more details about the processing and mechanical behavior of these alloy, see Titus et al. [34,35]. Thin foils were prepared from the alloy by twin jet electropolishing with a solution of 92.5% methanol-7.5% perchloric acid by volume at −40 °C, 16 − 20 V and 24 − 30 mA.

The stacking fault contrast in this cobalt-base superalloy was studied at 30 kV via TSEM in an SEM equipped with a STEM detector, at 200 kV via CTEM, and at 300 kV using a TEM equipped with an annular dark field detector. The SEM used in this work was an FEI TENEO equipped with a FEG source. An FEI Tecnai T20 equipped with a LaB_6 filament operated at 200 kV was used for the CTEM, and an FEI Titan equipped with a FEG source was used to collect STEM images at 300 kV. The real and reciprocal planar distances, Bragg angles, and extinction distances for cobalt at 30 and 200 kV are given in Table 1. The absorptive form factors of Weickenmeier and Kohl were used for the scattering calculations [36].

Dislocations were observed in a thin lamella of polycrystalline strontium titanate (STO) that was extracted from a femtosecond laser ablated surface using the FIB lift-out technique. A Ti:sapphire gain medium femtosecond laser was used to ablate the sample surface. It generates 150 fs pulses with a wavelength of 780 nm at a frequency of 1 kHz. The dislocation character and the depth of dislocation injection resulting from the femtosecond laser ablation process have been described in detail elsewhere [37]. The femtosecond laser ablation was performed *in situ* using the TriBeam microscope, described in [38]. The region studied contains two grains with different orientations relative to the laser machining direction (see Section 3.2). The Schmid factor of each grain was calculated according to the loading direction of the elastic wave generated during the ablation process, indicating that one grain is oriented in a soft configuration and the other one is oriented in a hard configuration with respect to the applied load [37].

Dislocations were also observed in the polycrystalline nickel-base superalloy René 88DT. The sample was cyclically loaded at