A new automated crystal orientation and phase mapping tool is described. This transmission electron microscope accessory combines a diffraction pattern acquisition system involving a CCD camera with pattern identification software that makes use of fast template-matching algorithms. The quality and reliability of pattern identification is substantially improved by combining the system to an electron beam precession device. Examples of phase and orientations maps constructed in only a few minutes are detailed.

**KEYWORDS**
transmission electron microscopy, electron diffraction, electron precession, diffraction patterns, orientation mapping, phase identification

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**INTRODUCTION**
Electron backscatter diffraction (EBSD) techniques on scanning electron microscopes (SEM) have proved to be very successful in the characterization of a wide variety of materials. Structures such as grain boundaries and precipitates, and the local texture of metals, alloys or semiconductors have been characterized by means of orientation and phase maps having a resolution from 0.1 µm to a few tens of nm, depending on the SEM resolution. Although SEM-based EBSD techniques are widespread today, comparable procedures on transmission electron microscopes (TEM) are not common [1-3]. As the interest in reliable and efficient characterization of nanomaterials is growing, it appears important to develop EBSD-equivalent tools for TEM, given that the spatial resolution of the latter is much higher than SEM (typically 1 nm for field-emission gun TEMs). This article describes the development and results of a novel tool that is able to automatically collect orientation as well as phase maps from nanostructured materials.

**MATERIALS AND METHODS**
EBSD-SEM indexing procedures are based on Kikuchi lines acquired for every point of the sample while the beam is scanning the area of interest. Although such procedures lead to precise orientation measurements, especially for well-recrystallized metals and alloys, Kikuchi lines are frequently weak or non-existent, particularly for deformed materials. For TEM studies, using spot diffraction patterns appears to be an interesting alternative and present computer indexing routines have been developed to extract the relevant information from them [3,4].

The proposed tool works as follows: electron diffraction (ED) spot patterns are collected with a CCD camera while the sample area of interest is scanned by the electron beam (Figure 1). Local crystallographic orientation and/or phase are identified through an original algorithm that compares the recorded ED pattern with pre-calculated (simulated) templates. This article describes the development and results of a novel tool that is able to automatically collect orientation as well as phase maps from nanostructured materials.
adequate connections to the deflector coils control boards (i.e. no STEM unit is required). While the thin foil is scanned, thousands of ED spot patterns are recorded and stored in the computer’s memory. The orientation/phase identification is currently performed off-line [3,4]. It involves a limited number of templates; for instance, fewer than 2,000 templates are sufficient to obtain a 1° angular resolution for copper (Figure 2). This enables the identification speed to be performed at a rate of up to 100 ED sec\(^{-1}\) for cubic materials. The degree of matching between experimental patterns and simulated templates is given by a correlation index. The highest value corresponds to the adequate orientation/phase [3,4]. For a given phase, the correlation indices calculated for every orientation are plotted on a map that represents a portion of the stereographic projection (reduced to a double standard triangle). The resulting map reveals the most probable orientation for every experimental spot ED pattern (Figure 2e).

**RESULTS**

For a typical map of 300×300 pixels, the beam scanning over the sample may last for a few minutes and subsequent data analysis (pattern matching for orientation/phase analysis) can be performed in 15 minutes for simple cubic cells. It takes 3 to 4 times longer for tetragonal or hexagonal cells that require more templates for the same angular resolution.

Orientation maps with sizes of 100×100 to 400×400 pixels have been obtained with an external CCD camera mounted in front of the screen of a JEOL 3010 (300 kV) LaB\(_6\) TEM with an acquisition frequency ranging from 10 to 175 frames per second (e.g. Figure 3a). The step size is typically 10-20 nm for a LaB\(_6\) TEM.

In addition to the orientation maps (OM), virtual bright field maps (VBF), index maps and reliability maps are also generated. VBF maps (Figure 3b) measure the fluctuation of the central beam intensity, revealing in this way a STEM-like brightfield contrast with a one-to-one connection to the OM maps [4]. Correlation indexes maps (Figure 3c) are mainly used to emphasize structural details, such as crystals having different orientations, but they are also sensitive to the foil thickness evolution. A reliability index has been defined (similar to an EBSD ‘confidence’ index [3,4]) in such a way that it has a low value when more than one solution is proposed, and a high value when the assigned solution index is much higher than any other proposed orientation or phase. Such reliability maps (Figure 3d) clearly reveal grain boundaries with a resolution that turns out to be smaller (i.e. better) than the beam size.

**Improved Orientation and Phase Identification with the Electron Precession Technique**

It is known from recent work [6,7] that electron precession may increase dramatically...
(almost double) the number of ED spots in SAED patterns and decrease dynamical effects (such as Kikuchi line contrast; see Figure 4 a,c). Consequently, identification through template matching will obviously be easier and this may be used to improve the quality of orientation/phase maps. According to our measurements, even small precession angles (e.g., 0.3°) substantially increase the correlation index factor for numerous acquired ED patterns (Figure 4b).

An example of such improvements is shown in Figure 5 where an area containing several randomly oriented mayenite crystals was scanned twice. The first attempt was performed in standard mode and the resulting orientation map (Figure 5a) exhibits colour fluctuations that are representative of frequent mis-indexed patterns. The second map (Figure 5c) was obtained using the ‘spinning star’ unit to promote precession with a 0.35° beam-rocking angle superimposed on the beam scanning. All other parameters (spot size, step size, camera setting, acquisition frequency, etc.) were similar. It may be noticed that the orientation map quality increases dramatically with precession: true uniform orientations are depicted within each grain.

Quality improvement for phase mapping with precession has been observed as well: in Figure 6 a replica sample containing different types of precipitates extracted from a 430 stainless steel sample can be seen. Carbide M23C6 with an fcc structure (a = 1.062 nm) and hexagonal nitride CrN precipitates (a = 0.483 nm, c = 0.451 nm) are present and neither their shape nor their size help to distinguish between them. Acquired phase maps without precession show ambiguous identification for two of the precipitates (half red, half blue in Figure 6c). By contrast, phase maps with precession reveal clear identification for all of them (Figure 6d).

Another interesting example that shows the effectiveness of template matching technique combined with precession for reliable TEM phase and orientation maps is the case of austenitic stainless TRIP steels (Figure 7) where three different crystallographic phases co-exist: austenite matrix (γ fcc phase with a = 0.358 nm), stacking fault bands leading to the ε hexagonal structure (a = 0.257 nm, c = 0.408 nm) and α’ martensite (quasibcc with a = 0.287 nm) produced locally when bands cross themselves. There is a spectacular improvement of orientation and phase mapping when preces-
combining spot ED patterns acquisition with CCD cameras, pattern matching techniques and electron beam precession. This novel technique goes far beyond equivalent EBSD-SEM techniques as TEM resolution is better than SEM, and the quality of spot diffraction pattern acquisition is less sensitive than the Kikuchi patterns used in SEM-EBSD to the crystalline deformation state, the crystallite sizes as well as the foil thickness.

REFERENCES

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