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Crystal c-axis mapping of hcp metals by conventional reflected polarized light microscopy: Application to untextured and textured cp-Titanium

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ABSTRACT

Nowadays the crystallographic characterization of polycrystalline metals and metallic alloys is usually performed by means of the electron backscatter diffraction (EBSD) technique. However, when dealing with coarse structures presenting intrinsic optical anisotropy, polarized light microscopy (PLM) imaging might be an alternative or a complementary time-efficient technique. Accordingly, a new computer-aided methodology has been developed in the present study and successfully applied on the crystal c-axis characterization of a hexagonal close-packed (hcp) structure, alpha commercial pure-Titanium (cp-Ti). In this sense, the correlation between EBSD-based crystallographic parameters and PLM-based parameters under certain light microscope settings has been first established by direct comparison using a representative amount of random orientations. Finally, PLM-based c-axis mapping and subsequent c-axis texture plotting have been performed and validated with EBSD for untextured and textured cp-Ti.

1. Introduction

Properties of polycrystalline metals and metallic alloys, such as mechanical and surface-related properties, strongly depend on the local crystallographic orientations. Therefore, mapping these orientations is of major importance for a detailed microstructural examination. The electron backscatter diffraction (EBSD), a SEM-based technique, is currently the most commonly used tool for the analysis of crystallographic orientations of bulk samples. Although this technique is well established, the required sample surface condition efforts high demands in terms of metallographic sample preparation. So, performing an EBSD scan is rather time consuming and the equipment accessibility is often limited. In case of optically anisotropic materials, a less demanding alternative methodology based on the polarized light microscopy (PLM) technique might be applicable for partial crystallographic characterization when the microstructure is coarse enough (grain size > 500 nm) so that the resolution of light microscopy is not a limiting factor.

PLM is a proven fundamental analytical instrument, which has been continuously used during almost the last two centuries. Improvements in microstructural characterization in sciences like mineralogy and more recently in biology and polymer science have come by the hand with the development of new PLM functionalities and applications to examine thin sections of the mentioned materials [1–4]. The typical microscope in these cases is the so-called petrographic microscope, which works either only with transmitted light or with both transmitted and reflected light configurations [5,6]. This technique can be used when the material presents optical anisotropy or birefringence. Then, PLM with the cross polarizer position, which is a perpendicular orientation of the oscillation planes of polarizer and analyzer, pictures crystal orientation contrasts. Hexagonal and tetragonal structures are optically uniaxial anisotropic. In that case, PLM is sensitive to the crystal c-axis orientation with respect to the incident light direction and the plane of vibration. As explained by the generalized Fresnel equations [7], this phenomenon occurs by the split of an incident plane wave into two refracted (or reflected [7]) individual wave components that are each polarized in mutually orthogonal plane waves, the ordinary and the extraordinary wave. Each wave has a different diffraction index. Destructive interferences of these waves upon exiting the sample produce color changes, which are function of the thin section thickness and the difference of the diffraction indices (the birefringence value) according to the Michel-Levy birefringence interference color chart [8,9]. Rotating the crystals at the microscope stage varies therefore their color appearance. For materials with very low birefringence values (< 0.005) and film thicknesses < 0.05 mm, in absence of retarder plates, the crystal orientation does not have influence on the color but just on the light intensity [9–11]. Traditionally, the estimation

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of the c-axis orientation in uniaxial structures has been manually done by making use of a U-stage, which allows the thin section to be freely rotated at the polarized light microscope [4]. In order to automatize the process, a PLM-based computer-integrated methodology [12–14] was developed for the determination and mapping of the c-axis orientations in thin sections of quartz, and its analogous implementation for other uniaxial minerals was proposed. The procedure entails the subsequent automatic transformation of color into gray scale and this one into light intensity. As opposed to the U-stage-based method, basically the only required rotation is that of the planes of vibration of the incident light about the light direction, i.e., rotation of the polarizer and, correspondingly, also the analyzer (in order to maintain the cross condition).

Contrary to the above mentioned sciences, in metallurgy the use of reflected light microscopy for the observations of bulk samples is preferred [15], transmitted light being used only for very specific applications [16]. As for the above mentioned cases, Fresnel equations also apply for metals if the complex refractive indices are used; and reflection from metallic surfaces is qualitatively similar to that of dielectrics [17,18]. It is well known that the rotation of a uniaxial structure bulk sample around the incident light direction results in a change in the reflected intensity, and such intensity evolution correlates with the corresponding c-axis orientation. Decades ago, the information obtained by PLM was “translated” into crystal orientations by the visual microscopic examination of twin traces or by means of X-ray diffraction using the back reflection Laue method for large grains [19,20]. Some recent studies on birefringent metals have included the examination of same surface areas by both reflected-PLM and EBSD for a direct comparison of the microstructure. However, to the knowledge of the present authors, in such cases PLM has been qualitatively used to reveal the grain boundaries; the crystallographic characterization being exclusively competence of the EBSD technique (see for example [21,22]). Quantitative PLM is up to now approachable only by the use of laser scanning confocal microscopes [23].

As opposed to EBSD, as said, PLM applied to uniaxial structures is just able to reveal partial crystallographic information, since this technique is only sensitive to the c-axis orientation. Specifically, for the case of hcp metals and metallic alloys such as Ti, for instance, the interest in knowing this parameter can be well justified. To mention some correlations between the c-axis orientation (basal poles) and material properties:

The orientation of the crystallites is very important for the mechanical properties. According to the inclination of individual crystallographic planes, which can act as glide planes for dislocation movement or as twinning planes, and the direction of the applied forces it is possible to derive critical resolved shear stresses (CRSS) acting in individual crystallographic planes. In pure Ti, the prismatic slip is easier than basal slip [24,25]. This has been recently confirmed by a study based on an alternative experimental technique [26], consisting in high energy X-ray diffraction microscopy (HEDM)-based 3D mapping of crystal grains, able to determine in a direct way the CRSS values of both plane systems. With some rare exceptions, explained by the local elastic strain state [26], the following general observation holds true: grains with their c-axis perpendicular to the tensile stress are “soft” (yield easily), because their orientation generally ensures a high Schmid factor for one or more of the three prismatic slip systems; and those with their c-axis parallel to the tensile stress direction are “hard”, because prismatic slip is difficult to be activated due to very low Schmid factors [27]. In the mentioned work [26], yielding resulting in c-axis tilting was attributed to basal slip, whereas yielding resulting in crystal rotation around the c-axis was attributed to prismatic slip. Additional analyses of the grain orientation distribution at different strains are given in [28].

It is known that basal planes in a hexagonal structure have the lowest surface energy due to the highest planar atomic density [29]. The c-axis orientation with respect to the materials surface can therefore have a strong influence on in situ properties of Ti such as corrosion, wettability [30], and cell-material interactions; extremely important attributes for bioimplant applications [29,31], among others. Even in cases of surface machining like grinding or milling the individual grain orientation is of important concern for the resulting machining forces, the chip formation, and the quality of the final surface (e.g. [32]). During cold spraying the individual grain orientation dominates the frequency of bonded particles [33].

To conclude, the revival of conventional reflected-PLM will be proposed in this work as a simple crystal-mapping technique for Ti and other metallic hcp structures where the crystal c-axis orientation (basal pole) may be relevant, taking advantage of the capabilities of present-day image processing tools and by making use of EBSD as a calibration technique.

2. Material and Experimental Procedure

2.1. Material and Sample Preparation

In the present study, two alpha-Ti samples with different microstructural characteristics were investigated, sample A and sample B. Sample A was extracted from a continuously casted round bar of Ti-Grade 2. Sample B was extracted from a unidirectionally rolled sheet of Ti-Grade 2 subjected to a subsequent annealing treatment. In both cases, extracted sections were perpendicular to the normal direction (ND). Both samples were embedded within an electrically conductive mounting epoxy resin, appropriate for the examination in the scanning electron microscope (SEM). The samples were wet grinded using a fine SiC grinding paper with 1200 grit size (during 60 s with a pressure load of 15 N) and 2000 grit size (for at least 90 s with a pressure load of 20 N) and then polished using an oxide polishing suspension (OP-S) with 0.05 μm particle size and 35% H_2O_2, in a ratio 80:20 (for at least 10 min with a pressure load of 10 N), until the microstructure/grains can be observed with sufficient quality under PLM [34,35].

2.2. Microscope Procedure and Data Analysis

EBSD scans were performed by means of an OIM EBSD detector attached to a JEOL JSM 6460LV SEM at the Institute for Surface and Thin Film Analysis, Kaiserslautern, with a step size of 3 μm, on representative areas of the surface normal to ND in both A and B samples.

PLM images were acquired with a Leica DM2500M light microscope equipped with standard polarizing light filters: an analyzer and a polarizer, working under reflection light conditions, originally designed to qualitatively reveal crystallographic contrast. The polarizer and analyzer oscillation plane angles were fixed in cross position. The capture of digital micrographs in gray scale was assisted by the microscope software platform Leica Application Suite, using the settings listed in Table 1.

The following PLM image capture and image processing methodology was carried out:

The samples were consecutively rotated on the light microscope stage counterclockwise about an axis normal to the plane of sight, from an angle $\gamma = 0^\circ$ to $\gamma = 180^\circ$, acquiring an image every 10°. It resulted in a set of 19 PLM images per sample, all of them being centered approximately at the same point. Rotating the polarizer and analyzer instead, as in ref. [13], is not appropriate in this case in which a

Table 1
Settings for the capture of digital light micrographs.

Optical magnification	×2.5
Brightness	10
Exposure time	102.1 ms
Gain	×1
Gamma	2.60
Saturation	Auto (1.00)