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iCHORD-SI combination as an alternative to EDS-EBSD coupling for the characterization of γ-γ′ nickel-based superalloy microstructures

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ABSTRACT

Because they exhibit very similar diffraction patterns, the γ and γ′ phases of nickel-based superalloys cannot be distinguished by the conventional EBSD technique alone. This paper presents an original method which discriminates those phases on an orientation map by exploiting their chemical differences. Performed with a Focused Ion Beam (FIB) microscope, the method is the combination of the ion CHanneling ORientation Determination technique (iCHORD) – recently developed by Langlois et al. [1] – with the chemical information obtained from Secondary Ion (SI) images. The performances of the method are compared to the EDS-EBSD coupling which also discriminates phases based on their chemical compositions. Applied filters, angular resolution, spatial resolution of the phase discrimination and acquisition time are discussed. It results that the iCHORD–SI combination offers an orientation map with an angular resolution slightly decreased compared to that of the EBSD technique, but with a good phase resolution (down to 150 nm) and within a reasonable acquisition time. Thus, the iCHORD–SI combination appears to be an interesting method for the crystallographic systems where phases are difficult to discriminate by the EBSD technique alone but present a significant chemical contrast.

1. Introduction

Due to their superior resistance at high temperature, γ-γ′ nickel-based superalloys are used for the manufacturing of highly-constrained aero-engine parts [2]. Their mechanical properties at high temperature result from several features including the γ′ phase distribution. Indeed, the γ′ phase forms precipitates of different sizes which are spread inside the γ matrix, the finer precipitates strengthening the alloy [3]. Many interactions between the γ grains and the γ′ precipitates occur during hot forging operations: deformation triggers recrystallization mechanisms which closely depend on the precipitation and its evolution. For instance, precipitates may dissolve to potentially reprecipitate behind the front [4, 5], pin grain boundaries [6] or act as nucleation sites [7, 8]. Understanding these γ-γ′ interactions enables the optimization of the forging process so as to obtain the γ′ phase distribution leading to the best mechanical properties. Thus, for the γ-γ′ microstructures the characterization of the respective phases is a real need.

Electron Back-Scattered Diffraction (EBSD) is commonly used to characterize polycrystalline microstructures. However, in the case of the γ-γ′ microstructures, this technique is not able to discriminate the γ and γ′ phases. Indeed, their respective crystallographic structures, FCC and $L1_2$, lead to very similar diffraction patterns [9]. Superstructure reflections, which are theoretically present in the γ′ patterns and absent in those of the γ phase, have too low intensities to be detected with the conventional EBSD mapping settings (e.g. diffraction patterns of 160 × 120 pixels).

γ′ phase is easily recognizable on BackScattered Electron (BSE) images because its higher content in light chemical elements makes it appear darker than the γ phase. The overlay of a BSE image acquired at a 0° tilt angle with an EBSD map acquired at a 70° tilt is difficult since distortions are very likely to exist between the two images even after tilt correction. In the EBSD sample configuration, BSE can be collected either by a BSE detector beneath the SEM column or by a solid-state BSE detector mounted at the topside of the EBSD camera. But in both cases

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the collected intensity is low, which can result in noisy images [10]. In addition, with the solid-state detector optimal contrast settings can be difficult to find [10]. In order to collect more intensity, some research have been done to use the EBSD detector itself as a BSE detector [10]; but the technique is not widespread.

Performing Energy Dispersive X-ray Spectrometry (EDS) at the same time as the EBSD scan allows the discrimination of the phases thanks to their difference in chemical composition [11, 12]. One limit of this method is the spatial resolution of the EDS technique which depends on the chemical composition of the material as well as on the electron beam energy [13, 14]. Yet, γ′ precipitates as small as 100 nm must be considered in the analysis of the microstructures because they have a significant effect on the mechanisms and kinetics of recrystallization in addition to their impact on the mechanical properties.

The present paper aims at presenting another method to discriminate the γ and γ′ phases on an orientation map, also based on the chemical differences of the phases but performed with a Focused Ion Beam (FIB) microscope. This methods combines the ion CHanneling ORientation Determination (iCHORD) technique, recently developed by Langlois et al. [1] and which uses the intensity variations in Secondary Electron (SE) images to determine the crystal orientations, with the Secondary Ion (SI) signal whose intensity strongly depends on the atomic number [15]. Acquired with the same scan electronics and without changing the sample position, SE and SI signals are can be easily superimposed, providing an orientation map with phase identification. The performances of the iCHORD–SI combination on a γ-γ′ microstructure are analyzed in regards to that of the EDS-EBSD coupling. Applied filters (Section 3.1), angular resolution (Section 3.2), spatial resolution of the phase discrimination (Section 3.3) and acquisition time (Section 3.4) are discussed.

2. Experimental

2.1. Material

The AD730™ alloy is a polycrystalline γ-γ′ nickel-based superalloy recently designed by the Aubert&Duval Company for aero-engine applications [16]. Its chemical composition is given in Table 1.

The γ phase forms the matrix of the alloy and is a Face-Centered Cubic (FCC) solid solution. The γ′ phase forms precipitates of various sizes and has a Simple Cubic $L1_2$ ordered structure. The chemical composition of each phase measured on the as-received billet by EDS at 15 kV is presented in Table 2.

Compression samples were machined out of the AD730™ industrial billet. To trigger the recrystallization of the alloy and observe γ-γ′ interactions, the samples were isothermally compressed below the γ′ solvus (which is about 1110 °C) and water quenched. Sample sections were ground and polished with first diamond suspensions down to 1 μm then 0.02 μm colloidal silica (OPS) on a vibratory polishing machine. Hot compression led to a partially recrystallized microstructure exhibiting many microstructural features, including recrystallized/unrecrystallized grains and several γ′ populations with characteristic sizes. Thus, this kind of microstructure was found relevant to discuss the performances of the iCHORD-SI combination.

2.2. The iCHORD-SI Combination

The ion CHanneling Orientation Determination (iCHORD) technique has been recently developed by Langlois et al. [1] to obtain orientation maps using the ion channeling effect. Ion/matter interactions produce secondary electrons whose intensity depends on how deep the ions are channeled through the crystal, and so on the orientation of the crystallographic planes relative to the ion beam [15, 17, 18]. Then, if the crystal position changes relative to the ion beam, the intensity of the collected secondary electrons varies following an intensity profile which is characteristic of the crystallographic orientation of the crystal.

The experimental setup of the iCHORD technique (Fig. 1a) has been optimized so that each intensity profile corresponds to a unique crystallographic orientation. The sample is first tilted at a 40° fixed angle around an axis ($\mathbf{e_y}$) which lies in its surface. Next, with a fixed azimuthal rotation step, the sample performs a complete rotation around its tilted normal ($\mathbf{e_z}$) to make secondary electron intensity vary (Fig. 1c). One Secondary Electron (SE) image is acquired at each azimuthal position. Then, using the Fiji image processing software [19], SE images undergo tilt correction, rotation correction, are aligned and cropped all together to get a stack of aligned images. The intensity of a given point of the scanned area varies as the stack is browsed, allowing the plot of a so-called intensity profile (i.e. intensity of the point in the SE image as a function of the azimuthal rotation angle, Fig. 1b). Finally, during the post-treatment stage, intensity profiles are compared to theoretical intensity profiles stored in a database, each theoretical intensity profile corresponding to a known crystallographic orientation. The building of the theoretical profiles and the database search algorithm are precisely described in [1]. The theoretical profile which matches the best with the experimental intensity profile gives the crystallographic orientation of the point.

The ion-matter interactions also create secondary ions whose intensity mainly depends on the atomic number of the excited atoms [15]. Thus, collected by a Secondary Ion (SI) detector, the Secondary Ion signal highlights the chemical contrast of the microstructure. In the present case, the γ′ phase, which contains lighter elements (Table 2), appears much brighter than the γ phase on SI images (Fig. 1d). To properly extract the γ′ phase from SI data, several SI images have been acquired at different rotation angles and summed all together so as the channeling contrast of the SI images does not interfere with the chemical information. Finally, to separate the γ′ precipitates from the γ matrix an intensity threshold is applied to the sum image.

Using the TESCAN Lyra3 FIB-SEM of the Orsay Physics Company, combined iCHORD and SI scans have been performed on a sample section. The FIB was equipped with a Gallium ion source and the beam setup for data acquisition was 30 kV/48 pA. The pixel size was 36 nm and the azimuthal rotation step was 4°, which implied the acquisition of 90 SE images to achieve the complete rotation of the sample. With the same 36 nm pixel size, four SI images were also acquired at different azimuthal rotation angles.

Table 2
Compositions of the γ and γ′ phases measured on the as-received billet by EDS at 15 kV (wt% - semi-quantitative values obtained without using a suitable standard - average values over an area of few μm^2).

Element	Ni	Fe	Co	Cr	Mo	W	Al	Ti	Nb	B	C	Zr
γ phase	54.6	5.1	9.9	19.8	3.1	2.1	2.3	2.5	0.6	NA	NA	NA
γ′ phase	70.7	1.5	5.1	3.6	0.3	0.9	6.0	10.6	1.4	NA	NA	NA

Table 1
Composition of the AD730™ nickel-based superalloy (wt%) [16].

Element	Ni	Fe	Co	Cr	Mo	W	Al	Ti	Nb	B	C	Zr
AD730™	Base	4.00	8.50	15.70	3.10	2.70	2.25	3.40	1.10	0.01	0.015	0.03