Materials Characterization 124 (2017) 206–214

Contents lists available at ScienceDirect

Materials Characterization

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

Non-destructive identification of unknown minor phases in polycrystalline bulk alloys using three-dimensional X-ray diffraction

Yiming Yang [a,b], Liang Xu [a,b], Yudan Wang [a], Guohao Du [a], Sam Yang [c], Tiqiao Xiao [a,b,*]

[a] Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201800, China
[b] University of Chinese Academy of Sciences, Beijing 100049, China
[c] Commonwealth Scientific and Industrial Research Organization, Melbourne, VIC 3168, Australia

ARTICLE INFO

Article history:
Received 4 November 2016
Accepted 22 December 2016
Available online 6 January 2017

Keywords:
Unknown minor phase
Non-destructive identification
3DXRD

ABSTRACT

Minor phases make considerable contributions to the mechanical and physical properties of metals and alloys. Unfortunately, it is difficult to identify unknown minor phases in a bulk polycrystalline material using conventional metallographic methods. Here, a non-destructive method based on three-dimensional X-ray diffraction (3DXRD) is developed to solve this problem. Simulation results demonstrate that this method is simultaneously able to identify minor phase grains and reveal their positions, orientations and sizes within bulk alloys. According to systematic simulations, the 3DXRD method is practicable for an extensive sample set, including polycrystalline alloys with hexagonal, orthorhombic and cubic minor phases. Experiments were also conducted to confirm the simulation results. The results for a bulk sample of aluminum alloy AA6061 show that the crystal grains of an unexpected γ-Fe (austenite) phase can be identified, three-dimensionally and nondestructively. Therefore, we conclude that the 3DXRD method is a powerful tool for the identification of unknown minor phases in bulk alloys belonging to a variety of crystal systems. This method also has the potential to be used for *in situ* observations of the effects of minor phases on the crystallographic behaviors of alloys.

1. Introduction

Metal materials are generally polycrystalline, and alloys in particular often contain more than one microstructural phase [1]. The mechanical and physical properties of metals and alloys are, to some extent, governed by their minor phases. Hence, the characterization of minor phases and their microstructural parameters (*e.g.*, volume fraction, morphology, texture and strain) is of great importance for understanding the overall properties of metal materials. Unfortunately, these minor phases are usually unknown and difficult to investigate. Most conventional metallographic methods, such as light optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), electron diffraction and focused ion beam (FIB) microscopy, are two-dimensional in nature and require the sample to be physically sliced. It is usually difficult to determine the three-dimensional distribution of the minor phases in a bulk sample using these two-dimensional methods. Therefore, non-destructive three-dimensional methods are preferred for probing minor phases with a heterogeneous distribution, irregular connectivity, varying orientations or complex morphologies. Three-dimensional methods for the determination of crystal structure [2–4] can also be used to investigate minor phases. However, these methods impose strict requirements on the samples, which must be single crystals or homogeneous, preferably monodisperse, powders. Consequently, new methods must be developed for application to the vast majority of real samples.

Three-dimensional X-ray diffraction (3DXRD) microscopy [5,6] is a method that can meet this challenge, particularly for *in situ* experiments. Unlike conventional determination methods, the 3DXRD methodology can be used to identify individual grains embedded within heterogeneous, polycrystalline materials while simultaneously providing information on their positions, sizes, morphologies, crystallographic orientations and elastic strains. Furthermore, *in situ* investigations of individual grains can be performed during processes such as deformation [7–9] or annealing [10–12].

Typically, the 3DXRD methodology were applied almost exclusively to monophase materials with the assumption of known space group or unit cell of the materials. A few applications on multiphase research were reported by Jimenez-Melero et al. [13] and Reischig et al. [14], but only limited to known phases. Furthermore, Sørensen et al. [15] introduced 3DXRD for the identification of minor phases in natural ore, which is based on a trial-and-error approach and requires some prior knowledge of the possible minor phases. As for the identification of unknown phases, a fast-Fourier-transform-based approach and a pattern recognition based method were summarized by Sørensen, Schmidt et al. [16]. In addition, the state-of-the-art indexing algorithm was proposed by Wejdemann et al. [17] based on simulated data. However, the direct identification of

* Corresponding author.
E-mail addresses: yangyiming1988@outlook.com (Y. Yang), tqxiao@sinap.ac.cn (T. Xiao).

http://dx.doi.org/10.1016/j.matchar.2016.12.025
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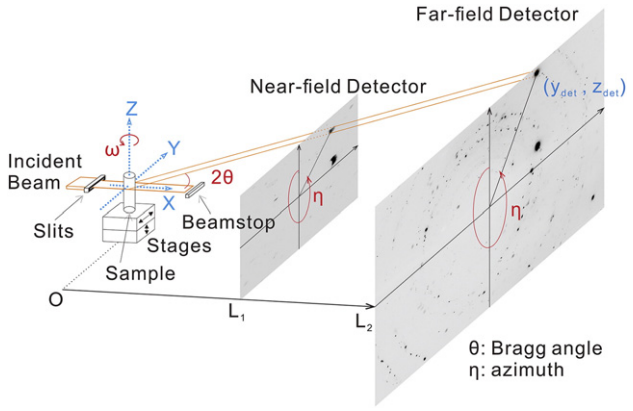

Fig. 1. Schematic diagram of 3DXRD measurements, with indications of the angles ω, 2θ and η and the directions X, Y and Z.

unknown minor phase remains unsolved because of the dispersive distribution and small size of the minor phase grains.

In this paper, a method based on 3DXRD is developed for the direct identification of unknown minor phases in bulk polycrystalline metals and alloys without prior knowledge of the possible phases. The method is introduced, and a systematic investigation of its effectiveness based on simulations is presented. More specifically, the present method enables the identification of an unknown minor phase using 3DXRD with the simultaneous acquisition of the spatial distribution, sizes, morphologies and crystallographic orientations of individual grains. Finally, experiments conducted on a sample of commercial 6061 aluminum alloy (AA6061) are reported to confirm the practicability of the proposed method.

2. Methods and Simulations

In an alloy, any minor phase has a different lattice structure from that of the main phase, which should manifest in the diffraction pattern as long as the spatial resolution and signal-to-noise ratio are sufficiently high during data acquisition. By merging the frames collected during a full 360° rotation, single-crystal-quality pseudo-powder diffraction pattern of an unknown minor phase can be extracted from 3DXRD data. Subsequently, the unit-cell and probable cell symmetry can be determined through relatively straightforward methods derived from the standard powder diffraction methodology. Once the unit-cell of an unknown minor phase has been determined, standard 3DXRD analysis can be performed to reconstruct the three-dimensional distribution of the individual grains, including their positions, sizes and crystallographic orientations.

To demonstrate the method, a set of experimental data is used as an example. A schematic illustration of the experimental setup is shown in Fig. 1. First, the recorded far-field diffraction spots of single crystals are segmented as connected components in (y_{det}, z_{det}, ω) space by setting an intensity threshold value, and they are sorted by their positions (y_{det}, z_{det}, ω, 2θ, η) and intensities (I). Second, as shown in Fig. 2, (2θ, η) plots of the diffraction peaks are obtained by merging the far-field

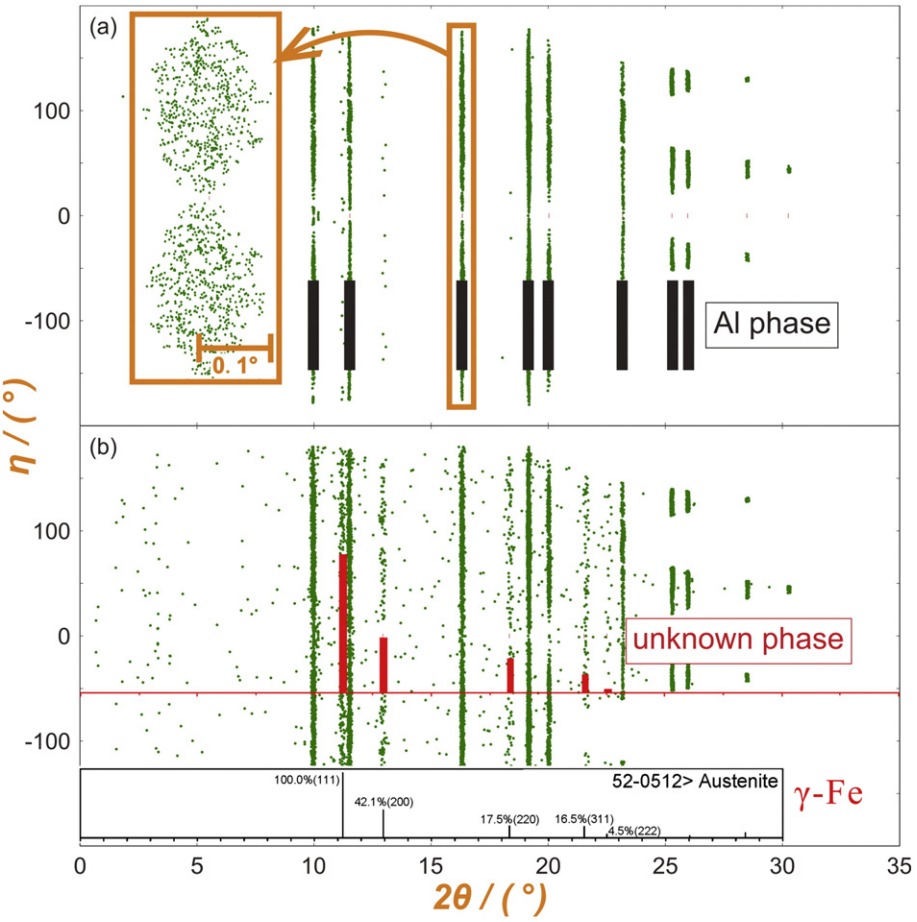

Fig. 2. (2θ, η) plot of the searched peaks obtained by merging the far-field ω frames at a high threshold level (a) and a low threshold level (b). The black bars correspond to the Al phase, whereas the red bars correspond to the unknown second phase. The height of each red bar represents the corresponding relative integrated intensity. The inset in (a) shows a magnified image of the region enclosed by the orange box. The inset in (b) shows the solution for the unknown minor phase, which are consistent with γ-Fe (austenite) phase.