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Nano-scale orientation mapping of graphite in cast irons

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ABSTRACT

A diametrical section of a graphite spheroid from a ductile iron sample was prepared using the focused ion beam-lift out technique. Characterization of this section was carried out through automated crystal orientation mapping in a transmission electron microscope. This new technique automatically collects electron diffraction patterns and matches them with precalculated templates. The results of this investigation are crystal orientation and phase maps of the specimen, which bring new light to the understanding of growth mechanisms of this peculiar graphite morphology. This article shows that mapping the orientation of carbon-based materials such as graphite, which is difficult to achieve with conventional techniques, can be performed automatically and at high spatial resolution using automated crystal orientation mapping in a transmission electron microscope.

1. Introduction

Mechanical properties of cast iron strongly depend on the structure of the graphite precipitates within the iron-rich matrix. In the 1950s, a spheroidization treatment led to a new range of mechanical properties for cast irons. Graphite, which would crystallize in a flake type without this treatment, grows as spherulitic crystals leading to improved ductility of cast irons. Although the production of spheroidal graphite cast irons is well-mastered, the spheroidization mechanism is yet unclear. Understanding of graphite growth mechanisms is of high importance to ultimately prevent the formation of degenerate graphite morphologies which lead to decreased mechanical properties of spheroidal graphite iron castings [1,2]. In order to do so, crystal orientations in graphite spheroids have been investigated by several authors by means of selected area electron diffraction and convergent beam electron diffraction [3,4] and the results have led to the development of various growth models for spherulitic growth of graphite. Nevertheless, generalising these local measurements to propose accurate growth models requires a global view of graphite's crystal orientations in a spheroid. The most common technique to map crystal orientations, that is to say electron backscatter diffraction in a scanning electron microscope, is not suitable for the study of carbon materials mainly due to carbon's low backscattering efficiency. For this reason, it is still very difficult to obtain orientation maps of carbon using this technique: only single point measurements have been reported [5] but these are not sufficient to fully determine crystal

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orientations of a whole sample. This limitation can be overcome by using different techniques, such as the recently developed electron diffraction based automated crystal orientation mapping (ACOM) in a transmission electron microscope.

The aim of this article is to present the crystal orientation of graphite within spheroidal precipitates in cast irons and to illustrate the potential of studying the crystallography of carbon-based materials using ACOM-TEM technique [6,7].

2. Materials and Methods

2.1. Sample Preparation Using a Focused Ion Beam

A commercial grade spheroidal graphite cast iron was used in this study. Its composition and microstructural features have been described elsewhere [8]. A section of this material was mechanically ground and then mirror polished with a 0.05 μm alumina suspension. A transmission electron microscopy specimen, containing a diametrical section of a spheroid, was extracted from the area shown in Fig. 1a using the focused ion beam-lift out technique in a JEOL JIB-4600F focused ion beam (FIB) scanning electron microscope.

This preparation technique was chosen because it enables precision cross-sectioning at the area of interest. Unlike other preparation methods, which lead to specimens with limited electron transparent areas, FIB sectioning facilitates homogeneous thinning thus producing a relatively large specimen which can be investigated by transmission electron microscopy. This is essential to the present work in which the growth features of a graphite spheroid are investigated.

2.2. Transmission Electron Microscopy Investigation

A JEOL JEM-2100F transmission electron microscope, operated at 200 kV, was used for imaging the sample. Energy dispersive X-ray spectroscopy was carried out using a Bruker Quantax silicon drift detector. A photomontage of bright field images (Fig. 1b) presents an overview of the sample's microstructure.

Automated crystal orientation mapping (ACOM) was carried out in a JEOL 3010 transmission electron microscope, operated at 300 kV, equipped with a DigiSTAR external source device. The electron beam scans the selected area of the sample and electron diffraction patterns are collected using an external CCD camera. The system compares the experimental diffraction patterns to pre-calculated templates and selects the best-matching patterns. The matching quality is measured by two parameters: the correlation index and the reliability. The first one evaluates the correspondence between the experimental pattern and those in the database. The calculated pattern giving the highest correlation index is selected and its corresponding orientation is assigned to the investigated spot. The reliability is determined by comparing the two orientations having the highest correlation indexes (best and second best matches). If the two calculated diffraction patterns have similar correlation indexes, more than one solution is possible and the reliability is low. It is important to keep these parameters in memory when orientation maps are presented.

3. Results and Discussion

Fig. 1b shows that there is a slightly darker area in the central part of the specimen, from which conical sectors seem to radiate. Variations in the sample's contrast suggest that graphite presents multiple orientations. Selected area diffraction patterns were repeatedly taken at different locations of the sample and showed that, within these sectors, the [0001] directions of graphite (*c* axes) are roughly parallel to the spheroid's radius. Following this procedure to study the orientation of graphite in large areas proved to be tedious and time consuming. Therefore, ACOM measurements were performed automatically over the whole sample with steps of 33 nm so as to obtain complete mapping of phases and crystal orientations, as shown in Fig. 2a and b. 44 diffraction patterns were taken every second resulting in a scanning time of roughly 30 min for an area of 66 μm^2.

As this system is based on electron diffraction patterns, the different phases of a sample can be distinguished as long as they have been declared in the template database. An example of this is shown by the dark areas at around 3 μm from the sample's centre seen in Fig. 1b. It is well-known that graphite spheroids can contain iron-rich inclusions and therefore, the

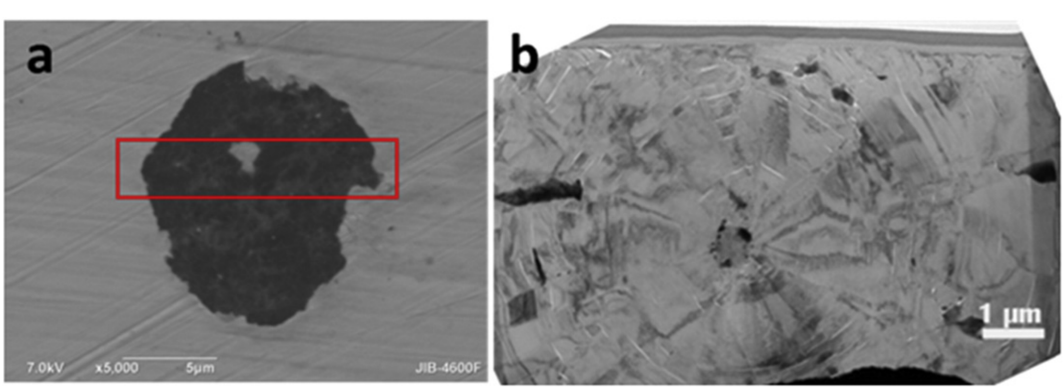

Fig. 1 – (a) Scanning electron micrograph showing the area where the sample was extracted; (b) photomontage of bright field images of the sample.