

A HIGH-MAGNIFICATION EBSD MAPPING ANALYSIS OF Fe–Si–B POWDER PARTICLES

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We produced amorphous Fe–Si–B soft-magnetic powder using water atomisation. During annealing the powder particles developed a nanocrystalline structure, and annealing at over 700°C led to the formation of ferrite and boride phases. Here we present a high-magnification electron backscatter diffraction (EBSD) mapping analysis of the powder particles, in combination with a field-emission-gun scanning electron microscopy (FEGSEM) analysis. Some of the problems associated with the preparation of the powder particles for the EBSD analysis as well as the drift problems occurring during the EBSD mapping are reported.

Key words: EBSD, soft-magnetic materials, Fe–Si–B-based powder, nanocrystalline structure, amorphous

Fe-Si-B čestice praha studirane EBSD analizom područja kod velikog uvećanja. Amorfni Fe-Si-B magnetni prahovi su bili proizvedeni vodenom atomizacijom. Za vrijeme procesa žarenja čestice praha dobiju nanokristaliničnu strukturu. Žarenje iznad 700 °C prouzroči nastanak feritne i boridne faze. U ovom članku je prikazana EBSD analiza područja kod velikog uvećanja prašnih čestica u kombinaciji sa FESEM. Istaknuti su problemi pripreme čestica praha za EBSD analizu kao i problem pomicanja uzorka tokom EBSD analize.

Ključne riječi: EBSD, meki magnetni materiali, Fe-Si-B prahovi, nanokristalinične strukture, amorfnost

INTRODUCTION

Of the group of common soft-magnetic materials that includes high-purity iron, silicon steels, iron-nickel and iron-cobalt alloys, and ferrites, one material is increasing in terms of commercial importance: the amorphous or nanostructured soft-magnetic material based on Fe–Si–B. Nanostructured, soft-magnetic materials derived from rapidly solidified Fe–Si–B alloys exhibit excellent soft-magnetic properties due to the crystallisation of nanoscale α -iron grains in an amorphous matrix [1, 2].

They are usually produced in the form of thin ribbons, using either melt-spinning or planar-flow casting techniques; however, by using gas- and water-atomisation procedures, fine powder particles can be obtained. When these particles are combined with a binding insulation material they are described as soft-magnetic composites or SMCs [3].

In order to achieve the nanocrystalline structure, careful control of the annealing treatment is required [4]. Various studies have been published [5,6] relating to the characterisation of Fe–Si–B-based SMCs, but none of them have made use of electron backscatter diffraction

(EBSD) as an analytical tool for the phase identification. For an introduction to the determination of micro-textures using EBSD the reader is referred to [7,8]. In this study we present a high-magnification EBSD mapping analysis of Fe–Si–B-based powder particles in combination with a field-emission-gun scanning electron microscopy (FEGSEM) analysis.

EXPERIMENTALPROCEDURE

The experimental Fe–Si–B powders were prepared by water-atomisation using a Davy McKee-type D5/2 pilot water-atomiser. A charge of 10 kg was prepared, melted in an induction-heating furnace and rapidly cooled in the water spray produced by a nozzle of dimensions $\Phi 1,2$ dia. x 1,05 mm and at a pressure of 20 MPa. After applying a sieve-separation technique, the finest fraction ($< 45 \mu\text{m}$) was obtained with the following chemical composition (in weight percent): Fe 88,1 %, Si 6,84 %, B 3,14 %, Ni 1,45 %, C 0.025 % and Al 0,12 %.

Samples of water-atomised powder were air annealed from 300 to 900°C in increments of 100°C. The annealed powders were then hot mounted in Bakelite, followed by a grinding-and-polishing procedure. The last step in the preparation of the samples for the EBSD analysis was colloidal silica emulsion polishing (OPS),

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which lasted for 5 minutes. It should be noted that it is very important to wipe the sample surface with cotton wool immediately after the OPS polishing and then to clean the surface in an ultrasound bath. The surfaces of the samples were sputtered with a 1-nm-thick carbon layer using a GATAN Precision Etching Coating System PECS 682. The samples for the high-magnification SEM observation were etched in 2 % nital.

The microstructures of the powders were analysed using a JEOL JSM 6500F field-emission-gun electron microscope equipped with an EBSD INCA CRYSTAL 300. The accelerating voltage of the primary electron beam for the backscatter images was 15 kV, the aperture diameter was 30 μm , the probe current was 0,08 nA and the working distance (WD) was 6 mm. The EBSD spot analysis and mapping were performed using a probe current of 7 nA, a sample tilt angle of 70,5 $^\circ$, a WD of 15 nm and an aperture of 50 μm .

RESULTS

XRD (X-ray diffraction) and HRTEM (high-resolution transmission electron microscopy) analyses showed that the water-atomised Fe–Si–B powder had an amorphous structure. However, during air-annealing the amorphous structure changed to a crystalline one. At an annealing temperature of 500 $^\circ\text{C}$ the first isolated crystalline phases began to appear. These crystalline phases have a typical star-pattern shape and grow with two, three or four branches of dendrites (Figure 1).

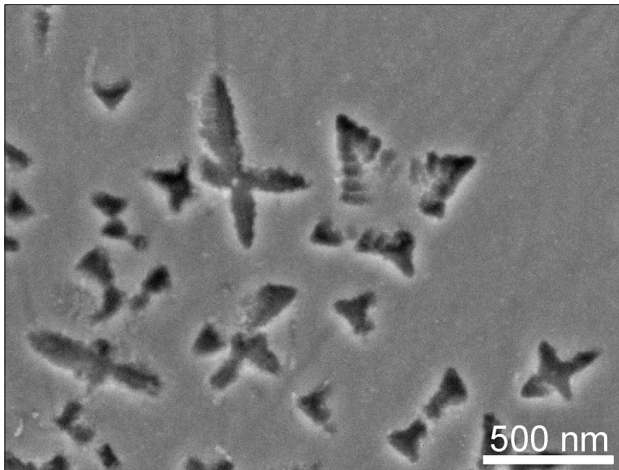


Figure 1. Secondary electron image shows Fe-Si-B powder particle cross-section. Powder particles were air-annealed at 500 $^\circ\text{C}$ for 15 minutes. The sample surface was polished and etched by 2 % nital.

The EBSD spot analysis showed that the first crystalline phase was α -Fe. Higher annealing temperatures led to a nanocrystalline structure, which was fully developed at 700 $^\circ\text{C}$. Figure 2 (a) shows a backscatter electron image of a cross-section of a powder particle annealed at 800 $^\circ\text{C}$. The microstructure consists of light- and

dark-grey grains. The average grain size of both grain types is approximately 300 nm. Using EBSD mapping, shown in Figure 2 (b), acquired at the same site of interest as the backscatter image, two phases, α -Fe and Fe_2B , were detected. The brighter grains in the backscatter image correspond to the boride phase and the darker grains

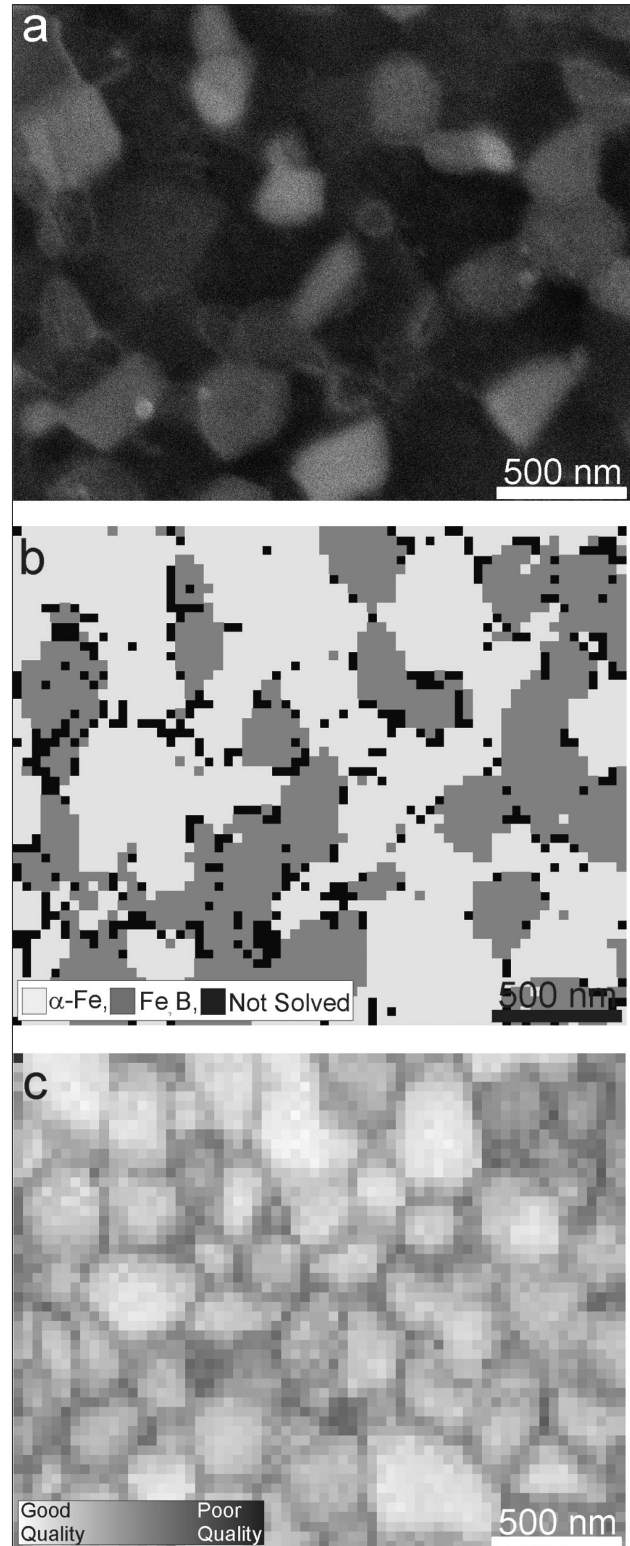


Figure 2. Cross-section images of Fe-Si-B powder particle annealed at 800 $^\circ\text{C}$ (a) Backscatter electron image, (b) EBSD map and (c) Pattern Quality map

to the ferrite phase. Both images coincide very well in spite of the slight drift that takes place during the long acquisition time. Additionally, a pattern-quality map was produced. Because the pattern-quality map depends on the orientation of the crystals, the one in Figure 2 (c) exhibits the grain structure. In Figure 2 (c) one can see that the boride phase has a slightly poorer pattern quality than the ferrite phase; this is due to the different crystal structure. In Figure 3, the Kikuchi patterns of both phases are shown. The orientations of the α -Fe and the Fe_2B crystallites in the polycrystalline aggregate are shown in

Figure 4. The textures of both phases are presented separately, as the normal direction orientation map in the colour-key mode. It is clear that there are no preferred orientations of either phase. The powder particles were rapidly solidified during the water atomisation and, as a result, no texture is to be expected.

DISCUSSION

The main problem encountered during high-resolution EBSD mapping is drift. There are, however, two kinds of drift. The first is sample-holder-stage drift, which can be overcome to some extent by waiting a longer time before the data acquisition, and the other sort of drift is electron-beam drift, which is more or less always present during longer data-acquisition times. Up until now, no commercial software has been available for correcting the drift during data acquisition. The reason is that the sample is highly tilted and therefore it is almost impossible to make a drift correction. There are, however, some solutions that are able to speed up the process of EBSD mapping. One is to use a higher accelerating voltage, which results in faster EBSD acquisition as well as sharpening the Kikuchi bands. Unfortunately, we had to decrease the beam's accelerating voltage due to charging of the sample, despite the 1-nm-thick carbon layer on the surface. A thicker conducting layer would have had an adverse effect on the Kikuchi pattern's quality, resulting in the need for a longer acquisition time. There might be some possibilities to use an ion-etching procedure, but the powders would

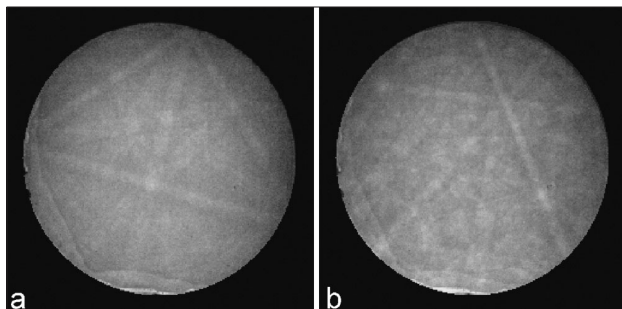


Figure 3. The Kikuchi pattern of (a) α -Fe phase and (b) Fe_2B phase

have to be embedded in some soft, conductive material with a similar sputtering rate.

In Figure 2 (a), which shows grains with two different contrasts, it would be expected that the lighter grains would correspond to the phase containing elements with a higher atomic number; this is because it is a backscatter image that is carrying information about the chemical composition. However, this is not so in this case. A Thermo-calc simulation [9] demonstrated that nearly all the silicon is soluble in bcc α -Fe phase, and therefore the atomic numbers are to a large degree equalised. The remaining very high contrast in the backscatter image it seems results from the light-grey grains being harder, and so during the metallographic preparation a surface topography is generated. The backscatter detector used for these analyses is extremely sensitive to surface topography, because the backscatter image is derived from secondary electrons as well.

On closer observation, in Figure 4 (b) we can see that some of the grains of the Fe_2B phase have a central re-

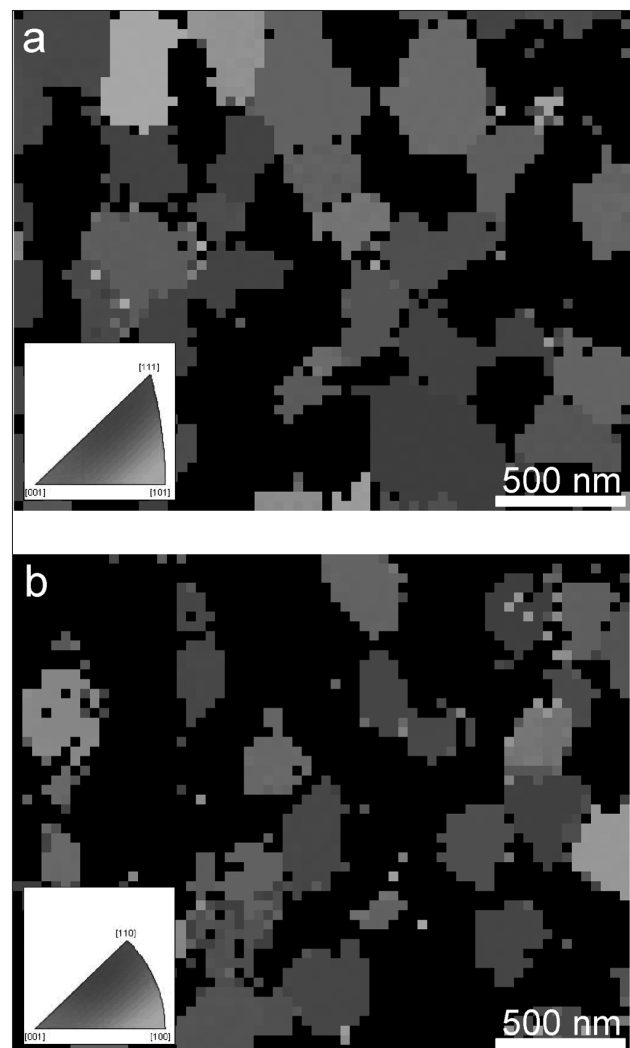


Figure 4. Normal direction map is shown with colour key inverse pole figure of Fe-Si-B powder particle cross-section annealed at 800 °C of (a) α -Fe and (b) Fe_2B phase

gion that is oriented very differently to the rest of the grain. This is due to a software error that occurred as a result of changing the most intense lines in the Kikuchi pattern. This most probably happened due to a poorer boride pattern. Longer OPS polishing would lead to a higher topography effect and a higher accelerating voltage, which would certainly improve the pattern quality, but would lead again to charging problems.

CONCLUSIONS

High-magnification EBSD mapping was performed on powder particles of the Fe–Si–B phase. We found that the annealed particles consisted of two phases, α -Fe and Fe₂B, both detected using the EBSD analytical tool in combination with a field-emission-gun scanning electron microscope.

This technique provides us with superior lateral resolution for the identification of different phases. To be able to obtain good results on powder materials the metallographic preparation prior to the EBSD is crucial. An additional problem is the need to deal with the drift of the EBSD sample image map.

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Note: The responsible language lecturer for English language dr. Paul McGuines, Ljubljana, Slovenia