Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 22(2012) 1801−1809
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Application of EBSD technique to ultrafine grained and nanostructured materials processed by severe plastic deformation: Sample preparation, parameters optimization and analysis

CHEN Yong-jun, Jarle HJELEN, Hans J. ROVEN

Department of Materials Science and Engineering, Norwegian University of Science and Technology, NO-7491, Trondheim, Norway

Received 10 November 2011; accepted 25 June 2012

Abstract: With the help of FESEM, high resolution electron backscatter diffraction can investigate the grains/subgrains as small as a few tens of nanometers with a good angular resolution (~0.5°). Fast development of EBSD speed (up to 1100 patterns per second) contributes that the number of published articles related to EBSD has been increasing sharply year by year. This paper reviews the sample preparation, parameters optimization and analysis of EBSD technique, emphasizing on the investigation of ultrafine grained and nanostructured materials processed by severe plastic deformation (SPD). Detailed and practical parameters of the electropolishing, silica polishing and ion milling have been summarized. It is shown that ion milling is a real universal and promising polishing method for EBSD preparation of almost all materials. There exists a maximum value of indexed points as a function of step size. The optimum step size depends on the magnification and the board resolution/electronic step size. Grains/subgrains and texture, and grain boundary structure are readily obtained by EBSD. Strain and stored energy may be analyzed by EBSD.

Key words: electron backscatter diffraction (EBSD); sample preparation; parameters optimization; step size; severe plastic deformation (SPD)

1 Introduction

Electron backscatter diffraction (EBSD) based on scanning electron microscopy (SEM) is a powerful technique to automatically and quantitatively measure the grain/subgrain size, local texture, point-to-point orientations, strain and phase identification [1−4]. With the help of field emission SEM (FESEM), high resolution EBSD can investigate the grains/subgrains as small as a few tens of nanometers [4] with a good angular resolution (~0.5°) [5]. It has been established that EBSD has a lot of advantages over transmission electron microscopy (TEM), such as simple sample preparation, automatic scanning and indexing, ultra-fast speed, large area investigation and a lot of post-processing results derived from one EBSD scan. After two important steps of development [4], the EBSD speed has increased gradually with the fast development of camera technique. It has increased sharply up to 1100 patterns per second (PPS) by adopting the offline EBSD techniques (Fig. 1). Therefore, the number of published articles related to EBSD has been increasing sharply year by year (Fig. 2, data are searched from www.scopus.com).

In recent years, severe plastic deformation (SPD) [6], e.g. equal channel angular pressing (ECAP), high pressure torsion (HPT), cyclic extrusion compression (CEC) and accumulative roll bonding (ARB), has been increasingly used in processing ultrafine grained (UFG, grain size in the range 100−1000 nm) or nanostructured (≤100 nm) materials directly from bulk samples. SPD techniques can easily reach an equivalent strains ≥10, which in turn leads to an equiaxed microstructure with a high density of grain boundaries (high angle grain boundaries (HAGBs) ≥60%). In order to understand the grain refining mechanism induced by SPD and to control the microstructure evolution, EBSD is very important to be employed, especially increasing demand for in situ heating and tension, and 3D EBSD investigation.

EBSD patterns are generated by backscatter diffraction of a stationary beam of high-energy electrons from a volume of crystal material within 50 nm depth in

Foundation item: Project (192450/I30) supported by the Norwegian Research Council under the Strategic University Program
Corresponding author: CHEN Yong-jun; Tel: +47-73594921; E-mail: happywinner01@gmail.com
DOI: 10.1016/S1003-6326(11)61390-3

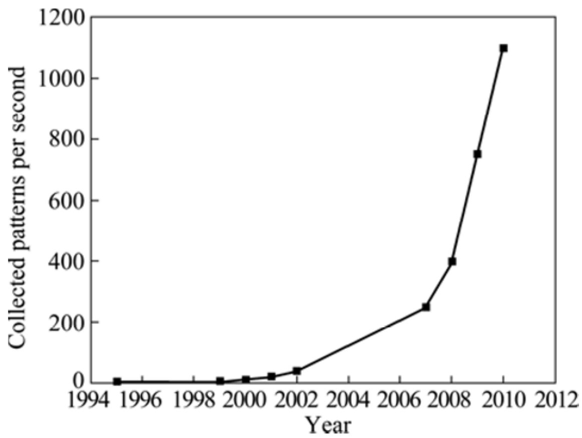

Fig. 1 Collected patterns per second as function of year

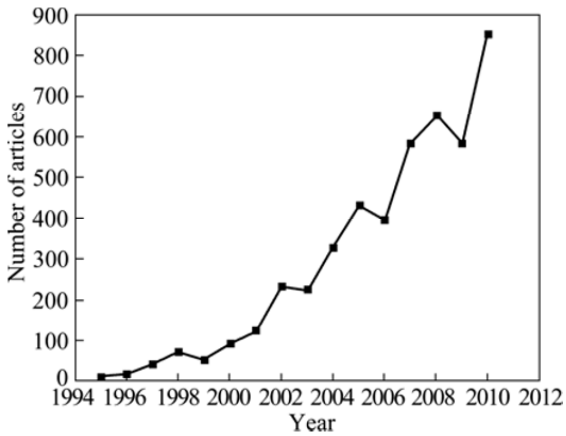

Fig. 2 Published articles related to EBSD as function of year

the specimen [3,7]. Therefore, the sample preparation is extremely crucial. This paper is concerned with the sample preparation, parameters optimization and results analysis.

2 Sample preparation

Despite a number of recommended preparation methods are available on internet, in publications or books, detailed and practical information about the preparation parameters is still missing. In general, almost all samples for EBSD observation need to be mechanically polished carefully. The suggested steps based on the experience in our group for the mechanical polishing sequence are listed in Table 1. When preparing soft materials (e.g. pure Al and its alloys), it is recommended to grind with a soap coating on SiC paper to avoid deep scratches or being embedded by hard particles. The grinding/polishing force depends on the hardness of materials, and less force is normally used with softer materials. Some materials such as magnesium alloys should never touch water after the last step listed in Table 1.

Table 1 Basic mechanical polishing sequence

Step	Method	Lubricant	Time
1	80 grit SiC paper	Water	Until plane
2	600 grit SiC paper	Water	20−90 s
3	1200 grit SiC paper	Water	20−90 s
4	2400 grit SiC paper	Water	20−90 s
5	6 μm diamond solution	Ethanol	1−5 min
6	3 μm diamond solution	Ethanol	1−5 min
7	1 μm diamond solution	Ethanol	1−5 min

Normally, the samples cannot be used for EBSD after 1 μm diamond polishing. There are three main methods (electropolishing, silica polishing and ion milling) for the final polishing of EBSD samples to achieve good quality of patterns.

2.1 Electropolishing

Electropolishing is a popular polishing method for the final step of EBSD preparation. It is one of the most effective methods for samples processed by SPD because it removes the microstrain induced by mechanical polishing. Electropolishing is a process by which surface material is removed from the sample by passage of electric current while the sample is made an anode (or positive terminal) in a designed solution.

In general, the fresh surface which is easy to oxidize is recommended to be electropolished below room temperature. Sample preparation for some materials like Mg alloys, which is known to be very difficult, should be performed carefully. EBSD observation should be done immediately followed by electropolishing due to the easy oxidation on free surface.

The electropolishing parameters of some commonly used materials are listed in Table 2. It can be seen that almost all materials require a subzero temperature to avoid the oxidizing problem. The electrolytes of AC2 and A3 are the product names of Struers Company. More information can be found from Ref. [8]. Normally, the electrolytes that are used to produce TEM thin foils can be used on electropolishing of bulk specimens [9]. For some materials like magnesium alloys, it is very useful to have an additional mechanical polishing with diluted OPS solution just before electropolishing [10].

The quality of the polished surface is controlled by some parameters, e.g. voltage, temperature, flow rate and polishing time. It is important to note that some solutions have a short shelf life [9]. The common problems, possible causes and the actions during electropolishing, are listed in Table 3 [15]. Although Table 3 is an instruction manual for the electropolishing machine of Lectropol-5 made by Struers Company, it works to shoot most problems during electropolishing.