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ESTIMATION OF THE RECRYSTALLIZED VOLUME FRACTION FROM LOCAL MISORIENTATION CALCULATIONS

SZACOWANIE UDZIAŁU OBJĘTOŚCI ZREKRYSTALIZOWANEJ NA PODSTAWIE ANALIZY RÓŻNIC ORIENTACJI

It's well known that density of defects and dislocations in the material have strong influence on material properties. The most important technological process reducing the density of dislocations is recrystallization. Material properties are depending on ratio between recrystallized and not-recrystallized parts. This paper describes a fast and precise method for the determination of the recrystallized volume fraction. The new method use Electron Back Scattered Diffraction measurements. The EBSD technique gives a lot of information about topology of grains, crystallographic orientation of measured points in the material and even estimated density of dislocation may be deduced from the image quality factor. The calculation in the new methods uses information about the local misorientation between measured points in the sample. Calculated misorientation is used as an estimator of the recrystallized volume fraction. The method is applied to polycrystalline copper and α-brass. The results are compared with Vickers microhardness measurements and with the analysis of the quality index distribution. The presented method is very simple to use and computer implementation is easy too. The method may be applied to old EBSD data previously measured with no intention to determine recrystallized volume fraction. Some limitations of the method were also discussed in th paper.

Keywords: recrystallization, copper, α-brass, EBSD, misorientation

Wiele własności różnych materiałów, w szczególności metali, zależy od znajdujących się w nich defektów i gęstości dyslokacji. Jednym z najważniejszych procesów technologicznych prowadzących do zmniejszenia gęstości dyslokacji jest rekrystalizacji. Własności materiału bezpośrednio zależą od stosunku objętości zrekrystalizowanej w materiale do tej, która jeszcze rekrystalizacji nie uległa. W niniejszej publikacji zaprezentowano nową, szybką i precyzyjną metodę wyznaczania zrekrystalizowanej frakcji (objętości) w prób-

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ce. Metodę oparto na wykorzystaniu danych pomiarowych uzyskanych techniką dyfrakcji wstecznie rozpraszanych elektronów (EBSD). Technika EBSD pozwala uzyskać wiele informacji na temat topologii ziaren, orientacji krystalograficznej zmierzonych punktów a nawet pozwala oszacować gęstość dyslokacji na podstawie wskaźnika jakości obrazu mierzonych punktów. Prezentowana metoda bazuje na wyznaczaniu różnic w orientacjach sąsiednich punktów pomiarowych. Na podstawie obliczonego rozkładu różnic orientacji wyznacza się oszacowanie wielkości frakcji zrekrystalizowanej. W publikacji zaprezentowano przykłady użycia metody do próbek miedzianych i mosiężnych. Wyniki zostały porównane z wielkością frakcji zrekrystalizowanej uzyskana innymi metodami, w tym metodą mikrotwardości Vickersa. Zaprezentowana metoda jest bardzo prosta w użyciu i łatwa w implementacji komputerowej. Metoda może być również zastosowana do starych danychpomiarowych, które nie były wykonywane z myślą o ocenie stopnia zrekrystalizowania materiału. Na zakończenie pracy przedyskutowano warunki stosowalności i ograniczenia metody.

1. Introduction

In industrial metallurgy, the recrystallization process is still concerned to a large domain of applications. Indeed, this is the only process through which a new microstructure nearly defect free can be produced during or after plastic deformation (called respectively *dynamic* and *static* recrystallization) [1, 2]. It is then evident that recrystallization is the crucial step in any thermomechanical treatment. Some works have directly shown that there exists a relation between crystallographic texture, microstructure and mechanical properties. But it is necessary to determine precisely the recrystallized volume fraction, as it is known that a low fraction of remained deformed material can strongly change the material properties [3].

Electron Back Scattered Diffraction (EBSD) has recently been used to get some important information concerning e.g. local orientations of defined zones and microstructural parameters (morphology, grain size, etc). Some authors have used EBSD measurements to obtain the recrystallized volume fraction values [4]. These methods present numerous advantadges and one of them is that it is non-destructive. The first developed method to estimate the recrystallized fraction, is based on image treatment, through the analysis of grain shape and size [5, 6]. Beside this approach, the present authors have used the quality index q affected to the Kikuchi diagram in order to evaluate more precisely the recrystallized volume fraction [7, 8]. They have shown that a better evaluation is obtained in some cases (connected to some experimental parameters, especially the grain size and the grain boundary area) through the analysis of the q distribution extracted from measurements of statistically representative points on different states (deformed, partially and fully recrystallized). An alternative of this last method needs to be developed when the limits of precision of EBSD device is reached. For example, when the Scanning Electron Microscope (SEM) is running with a standard tungsten filament, the finest EBSD spatial resolution obtained in this case is equal to 200-500 nm in the best cases (estimated in the case of aluminium [9]). When the dislocations microstructure size (dislocations cells) is lower than these values, the Kikuchi diagram is hard-

ly indexed or even non-indexed, and as a consequence, this tends to modify hardly the q values and distributions. In the present work, a faster and more precise determination of the recrystallized volume fraction is set taking into account the local misorientation calculated for each closed neighbors of the measured matrix, based on the fact that plastic deformation creates important local misorientation. The distinction between deformed and recrystallized areas becomes in this sense easier.

After presentation of the method used, the calculations done on copper and α-brass materials are explicited. The comparison with the recrystallized volume fraction values obtained with more classical methods (Vickers microhardness measurements and quality index q distribution) is done in order to validate the proposed procedure.

2. Experimental details

OFE (Oxygen Free Electronic) copper and α-brass (Cu-15%Zn and Cu-33%Zn in weight) have been industrially hot rolled, then cold rolled to a reduction level between Δ=70 and 98%. The deformed materials have been partially and fully annealed in oil (for temperature between 50° and 300°C) and salt (for temperature between 300° and 550°C) baths, in order to get a complete set of various states. The applied annealing time has been set to 15 minutes, for both materials and all temperatures. After mechanical surface preparation and electropolishing, EBSD measurements were performed on a Cambridge S360 SEM (with a tungsten filament) with the automatic software OIM^{TM} from the TSL company. Deformed, recrystallized and a few partially recrystallized states have been investigated in the rolling and transverse planes with a choosen step of 1 µm (an hexagonal grid has been selected). Four areas of 150 µm^2 have been selected for each state in order to get a good statistical representation. A first estimation of the recrystallized volume fraction has also been done through Vickers microhardness measurements, for the sake of comparison.

3. Methodological procedure

Local inhomogeneities are clearly visible at the EBSD scale, in both deformed and recrystallized states (see Fig. 1a and 1c for cold rolled copper): orientation changes are classically illustrated by color changes. A local misorientation parameter can also be calculated (see procedure below) and colored maps can be redrawn by affecting the same color to neighboring points presenting a misorientation below a given limit. This is illustrated in the case of copper in Fig. 1b and 1d with a tolerance misorientation value set to 1°. It is clear that the orientation is much more homogeneous in the recrystallized state than in the deformed one.

Only first order neighbors (i.e. six closest points) have been considered in the calculation of the local misorientation. Fig. 2 describes the calculation method of these misorientation values: each point of the matrix is compared with his closest neighbors

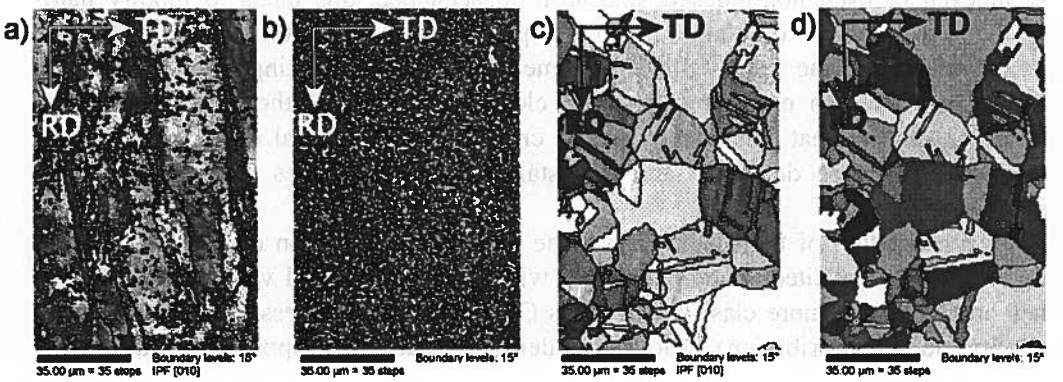

Fig. 1. Cold rolled copper to $\Delta = 70\%$. a), c) Inverse Pole Figure maps in grey scale. b), d) Neighboring misorientation maps (tolerancevalues: 1°). RD= Rolling Direction, TD= Transverse Direction

and each pair of points is considered in the calculation only one time. In this way, the influence of grain boundary points is somewhat minimized. Also, the last column and the last line of each orientation map are not taken into account.

The estimation of the recrystallized fraction X_v is then very simple. Both deformed and fully recrystallized states are taken as references. The number of points presenting a misorientation greater or equal to a choosen limiting value with their closest neighbors in the deformed sample (N_d) and in the fully recrystallized material (N_r) are first

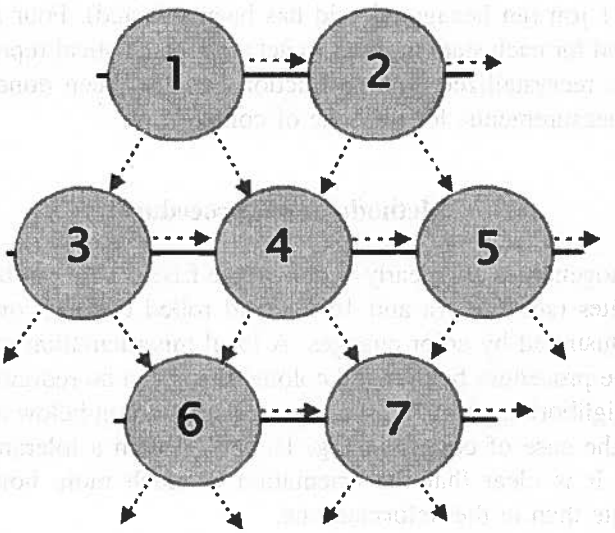

Fig. 2. Method of misorientation calculation for the closest neighbors

calculated. For any partially recrystallized sample, the number of points n fulfilling the described condition — i.e. local misorientation greater or equal to the given limit — is then determined. The recrystallized volume fraction is then equal to:

$$Xv = \frac{n - N_d}{N_r - N_d}. \qquad \text{(Eq. 1)}$$

From Eq. 1, the recrystallized volume fraction can thus be calculated for different partially recrystallized states and the values are directly function of the quality of measurements in the deformed and recrystallized states, as well as of the selected limiting value (Fig. 3). The values of N_d and N_r calculated for the two investigated materials and varying limiting values are shown in Table 1. It is clear that N_d approaches 100% in highly deformed materials when the limiting value is set to the smallest possible value (1°), whereas N_r is still far from 0% in the case of completely recrystallized material (due to the presence of grain boundaries and defects that affect the measurements). This fact justifies the fact that both deformed and recrystallized states have to be taken as references to the same material.

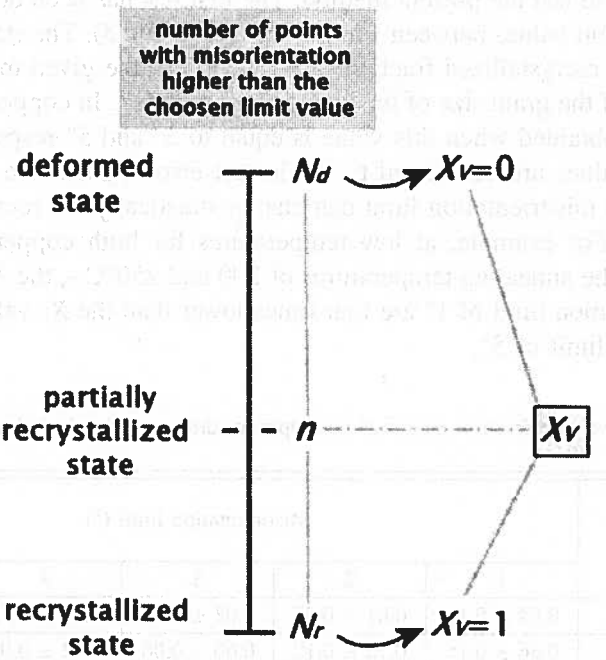

Fig. 3. Estimation of the recrystallized volume fraction from the choosen misorientation limit value

TABLE 1

Calculated N_d and N_r for Copper and Brass for varying limiting misorientation values

Material	Misorientation limiting value (°)	N_d (% of points)	N_r (% of points)
Copper	1	93.05	47.83
Cold rolled, 70%	2	75.48	31.43
	3	60.86	20.19
	4	50.62	19.60
	5	43.69	19.50
CuZn15	1	83.81	41.61
Cold rolled, 80%	2	81.35	38.71
	3	78.86	37.97
	4	76.43	37.87
	5	76.18	37.82

4. Test of the method on EBSD measurements obtained on copper and α-brass

The cold rolled to $\Delta = 70\%$ copper and the CuZn15 to $\Delta = 80\%$ have been selected in order to test the present method. The first test has been done using different misorientation limit values between 1 and 5° (Tables 2 and 3). The statistical dispersion of the calculated recrystallized fractions are function of the given misorientation limit value, and also of the grain size of the investigated material. In copper and CuZn15, the best results are obtained when this value is equal to 3° and 5° respectively, for which the calculated values are connected to the lowest error values. We have to note that the choice of the misorientation limit can change drastically the recrystallized volume fraction values. For example, at low temperatures for both copper and CuZn15 — respectively for the annealing temperatures of 200 and 250°C -, the X_v values obtained with a misorientation limit of 1° are four times lower than the X_v values obtained with a misorientation limit of 5°.

TABLE 2

Recrystallized volume fraction estimated in copper for different misorientation limiting values

annealing temperature (°C)	Misorientation limit (°)				
	1	2	3	4	5
200	0.04 ± 0.11	0.01 ± 0.09	0.02 ± 0.15	0.04 ± 0.12	0.20 ± 0.12
230	0.66 ± 0.15	0.72 ± 0.12	0.60 ± 0.06	0.73 ± 0.02	0.60 ± 0.09
240	0.78 ± 0.11	0.87 ± 0.05	0.71 ± 0.03	0.67 ± 0.05	0.62 ± 0.09
260	0.86 ± 0.04	0.97 ± 0.05	0.77 ± 0.06	0.70 ± 0.08	0.61 ± 0.10
290	0.95 ± 0.11	0.99 ± 0.03	0.78 ± 0.02	0.71 ± 0.04	0.63 ± 0.04

TABLE 3
Recrystallized volume fraction estimated in CuZn15 for different misorientation limiting values

annealing temperature (°C)	Misorientation limit (°)				
	1	2	3	4	5
250	0.05 ± 0.21	0.07 ± 0.21	0.10 ± 0.21	0.14 ± 0.21	0.19 ± 0.22
270	0.07 ± 0.27	0.13 ± 0.35	0.18 ± 0.38	0.22 ± 0.39	0.26 ± 0.15
290	0.05 ± 0.09	0.09 ± 0.10	0.13 ± 0.09	0.17 ± 0.09	0.21 ± 0.08
310	0.35 ± 0.07	0.42 ± 0.04	0.44 ± 0.03	0.46 ± 0.02	0.47 ± 0.02
320	0.46 ± 0.07	0.51 ± 0.03	0.52 ± 0.02	0.52 ± 0.01	0.51 ± 0.01
340	0.43 ± 0.07	0.58 ± 0.05	0.60 ± 0.06	0.61 ± 0.06	0.61 ± 0.11
360	0.41 ± 0.15	0.53 ± 0.07	0.56 ± 0.01	0.57 ± 0.03	0.57 ± 0.04
400	0.58 ± 0.09	0.65 ± 0.09	0.66 ± 0.09	0.66 ± 0.09	0.67 ± 0.09

The X_v values obtained from the present method are directly compared with the one calculated from Vickers micro-hardness measurements and the quality index distribution analysis done with the same EBSD data set [8] (Fig. 4).

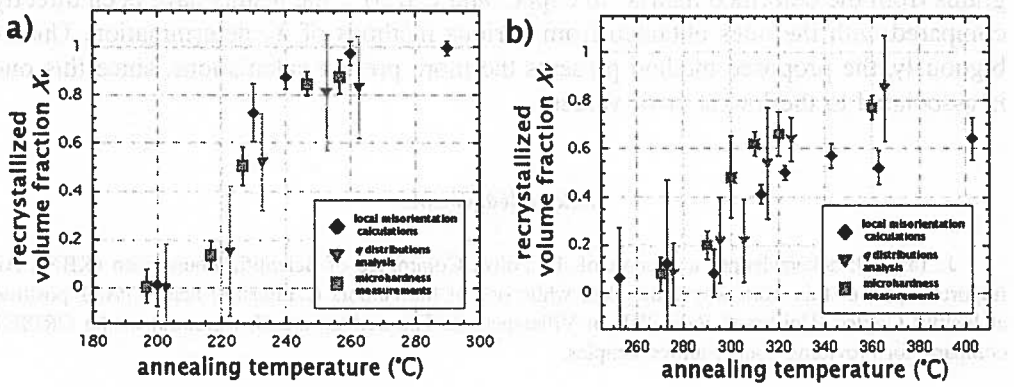

Fig. 4. Comparison of recrysallized volume fractions estimated by the present method, from microhardness measurements and q distributions analysis. a) Copper. b) CuZn15

For both materials and with the present method, the recrystallized volume fraction values are well estimated. The local misorientation calculations give lower error bars than the two other methods.

5. Discussion

The proposed method gives some relatively good recrystallized volume fraction values, under some strict conditions. First of all, the misorientation limit value associated to the lowest error bars has to be determinedcarefully. Indeed, it has been explicited that different values gives important deviations in the calculation of X_v values. The test done on copper and CuZn15 shows that the selected value depends on both material features and grain size. On second hand, it exists a relation between the grain size, the frequency of grain boundaries and the choosen EBSD step size. Of course, if the average grain size is relatively small in the deformed or in the recrystallized state and if only few measured points are "inside" a grain, the misorientation calculation between neighbouring points the grain boundaries would take into account mainly. As a direct consequence, the error values associated to the X_v would be more important. This effect can be reduced with the SEM/EBSD parameters, on account that the smaller average grain size, the smaller the EBSD step size. Obviously, the limit of the present method depends mainly of the limit of the SEM/EBSD used.

6. Conclusions

A new estimation method of the recrystallized volume fraction has been proposed in this paper taking into account complete set of EBSD data. The misorientation calculations between neighbouring points is used in order to separate the recrystallized grains from the deformed matrix. In copper and CuZn15, the results have been directly compared with the ones obtained from various methods of X_v determination. Unambiguously, the proposed method presents the more precise calculations, since this one is associated to the lowest error values.

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