Quantification of recrystallization kinetics in industrially processed 5xxx aluminum alloy sheet

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Abstract. The annealing kinetics of two aluminum alloys AA5xxx is investigated. The volume fraction of recrystallized material, \( V_v \), is determined for a series of partly recrystallized samples both by hardness measurements and by electron back scattering diffraction (EBSD). Because of extended recovery overlapping with recrystallization, in particular in AA5182, it is not straightforward to estimate \( V_v \) from the hardness measurements. Two methods are suggested: one relying solely on the hardness data, and one in which EBSD data are used to estimate the hardness at the onset of recrystallization. Effects of the choice of method and of parameters on the resulting annealing kinetics are analysed, and the potential and limitations of the two methods are discussed.

1. Introduction

The 5xxx alloys were initially developed in the 1930’s in response to demands for sheet materials with higher strengths, good formability, and high levels of corrosion resistance and weldability. In this study two 5xxx aluminum alloys (AA5182 and AA5657) with markedly different microstructures are investigated. In the automobile industry, AA5182-O sheets are used for deep drawn body-parts, including inner door or hood parts [1]. On the other hand, AA5657-H24, a higher base purity alloy, is widely used in bright and anodized sheet products[2].

The chemical compositions of the two alloys are given in table 1. Due to the very low solubility of Si in high Mg alloys, Mg:Si is often present in the microstructure of AA5182 as a major constituent. Large (1-10 \( \mu \)m) constituent phases containing Fe, Mn and Si such as \( \text{Al}_2(\text{Fe, Mn})_3\text{Si} \), \( \text{Al}_5(\text{Fe, Mn}) \) and \( \text{Al}_3\text{Fe} \), which are found in most commercial alloys with >0.05%Fe [2]. During homogenization at 500-530°C, fine Mn-rich dispersoids [3] of about 0.1–0.4 \( \mu \)m are formed in the AA5182 alloy. These particles increase the strength of the alloy and serve to retard recrystallization and grain growth during annealing. While the two alloys selected for the present study represent considerable differences in deformed microstructure, annealing is of key importance for industrial thermomechanical processing for both alloys.

The overall aim of this study is to quantify the recrystallization kinetics. The classic model describing transformation kinetics was proposed by Johnson and Mehl [4], Avrami [5], and Kolmogorov [6] for phase transformations, but is generally also used for recrystallization. In this so-
called JMAK model, the increase of the recrystallized volume fraction ($V_v$) during isothermal annealing is expressed as:

$$V_v = 1 - \exp(-kt^n)$$

$$n = \frac{\log(-\ln(1 - V_v)) - \log k}{\log t}$$

where $t$ is the annealing time, and $k$ and $n$ are two parameters describing the kinetics, where $n$ is often termed the Avrami exponent. If $(-\ln(1 - V_v))$ is plotted versus $t$ on double logarithm scale a straight line should thus be obtained with $n$ as the slope (see equation (2)). In many cases the equations (1-2) are found to fit experimented data points nicely [7-9], while in others no fit, or a fit to more than one straight line can be observed [10, 11]. In the present work we shall use equations (1-2) to analyse the recrystallization kinetics based on $V_v$ values experimentally determined by electron backscatter diffraction (EBSD) and hardness measurements. Even simple analysis of the EBSD results gives a precise measure of $V_v$ [12, 13]. The hardness data reflect all the mechanisms occurring during the annealing, i.e., recovery, recrystallization, grain growth and also precipitation if that takes place during the annealing. The specific aim of this work is to establish methods to deduce $V_v$ from these more complex hardness data.

2. Experimental

2.1. Materials description

Two commercial aluminum alloys, AA5182 and AA5657, in the form of sheets are used for the present study. The two alloys were supplied by Novelis Inc. The AA5182 sheet was first hot rolled to 4 mm, and subsequently cold rolled to 1 mm (75% reduction) on industrial rolling mills. The AA5657 sheet was first hot rolled to 6 mm, then cold rolled to 3.5 mm followed by inter-annealing at 320-345°C for 100 minutes. Finally the sheet was cold rolled to 0.76 mm (78% reduction) followed by 235-240°C batch annealing (recovery treatment) for 3 hours. In this study, specimens were cut from the two alloys sheets and annealed isothermally at 245°C and 300°C respectively for various durations, ranging from 5 minutes to 1440 minutes in an air furnace. Thereby a series of partially recrystallized specimens was obtained.

| Table 1 | Chemical compositions of the AA5182 and AA5657 alloys. |
|---------|------------------------------------------------------|
| Alloy   | Mg  | Mn  | Fe  | Si  | Cu  | Cr  | Al   |
| AA5182  | 4.50| 0.26| 0.16| 0.09| 0.05| 0.04| Bal. |
| AA5657  | 0.6-1.0| 0.03| 0.10| 0.08| 0.10| 0.00| Bal. |

2.2. Hardness measurements

The Vickers microhardness was determined using a HMV-G microhardness instrument. The specimens were prepared by mechanical grinding to 4000 grit SiC abrasive paper. The hardness in the rolling plane containing the rolling direction (RD) and the transverse direction (TD) was measured applying a load of 50 g for 10 seconds.

2.3. EBSD measurements

A TESCAN MIRA3 scanning electron microscope equipped with an Oxford Instruments Channel 5.0 EBSD system was used to characterize the microstructures. The microscope was operated at 20 kV. The longitudinal plane containing the rolling direction (RD) and the normal direction (ND) was inspected. EBSD specimens were prepared by mechanical grinding and subsequent polishing with a standard colloidal alumina suspension solution with mean particle size of 30 nm. The EBSD specimens were finally electropolished in a 10% perchloric acid and ethanol solution at 12°C for 10-15 seconds with an electric potential difference of 20 V. The step sizes for EBSD scanning were chosen.
to be 0.5 µm and 1 µm for the AA5182 and AA5657 alloys, respectively. Based on EBSD maps, the recrystallized volume fraction (\(V_v\)) was calculated by the equation:

\[ V_v = \frac{\sum \lambda_i}{L} \]

(3)

where \(\lambda_i\) is the recrystallized grain intercept length, i.e., the chord length of grain \(i\) intersected by a test line where \(L\) is the total test line length. Fifteen lines along ND with total line lengths of 3600 µm and 10000 µm were used for each EBSD map for the AA5182 and AA5657 alloys, respectively.

3. Results and discussion

In the deformed state AA5182 has a Vickers hardness of \(~117\) HV which, during annealing, decreases to \(~79\) HV for the almost fully recrystallized state. The results are shown in figure 1(a). Initially the hardness decreases at a slow rate from 5 minutes to 60 minutes, then at a higher rate. The hardness evolution for AA5657 is shown in figure 1(b). Compared to AA5182, it is clear that the hardness drop before initiation of recrystallization is less significant (this is partly due to the already recovered structure in the initial state), and the total reduction in hardness is only \(~20\) HV, whereas in AA5182 it is \(~40\) HV.

Figure 1. Vickers microhardness vs. time during isothermal annealing of (a) AA5182 annealed at 245°C and (b) AA5657 annealed at 300°C.

Figure 2. IPF maps from EBSD characterization of partially recrystallized AA5182 annealed at 245°C for (a) 60 minutes (\(V_v = 0.05\)); (b) 160 minutes (\(V_v = 0.26\)); (c) 1440 minutes (\(V_v = 0.96\)).

Figure 3. IPF maps from EBSD characterization of partially recrystallized AA5657 annealed at 300°C for (a) 30 minutes (\(V_v = 0.05\)); (b) 80 minutes (\(V_v = 0.52\)); (c) 240 minutes (\(V_v = 0.95\)).
Images of the microstructures of the two alloys as observed by EBSD are shown in figures 2 and 3. The recrystallized grains are almost equiaxed with an average size of ~10 µm. In the AA5657 alloy the recrystallized grains are elongated along RD with an average size of ~60 µm (see figure 3). After 240 minutes annealing, some grain growth has occurred even though the microstructure is not fully recrystallized.

The volume fraction of recrystallized material, \( V_v \), is readily calculated from the EBSD data based on equation (3). The most common way to quantify recrystallization is to fit the \( V_v \) data by the JMAK equation (1). The results, shown in figure 4, show 2 distinct stages, i.e., one \( n \) value cannot describe the present data well for either of the alloys. A similar result is also seen for recrystallization of other materials, e.g., copper, iron and steel [11, 14, 15]. In the following, we shall analyse the dominant stage, i.e., the part with the highest \( n \) value.

From the hardness data, \( V_v \) can be estimated by the following equation:

\[
V_v = \frac{HV \_0 - HV}{HV \_0 - HV \_R}
\]

where \( HV \) is the hardness at a certain annealing time, \( HV \_0 \) is the hardness after recovery and \( HV \_R \) is the hardness when the sample is fully recrystallized. A particular challenge here is how to determine \( HV \_0 \), in particular for the AA5182 alloy in which the transition from recovery to recrystallization is very subtle. Actually, for this alloy it is also not straightforward to determine exactly the value of \( HV \_R \).

![Figure 4. Avrami plot of the evolution of the recrystallized volume fraction (\( V_v \)) based on EBSD data for both alloys.](image)

![Figure 5. Avrami plot of the evolution of the recrystallized volume fraction (\( V_v \)) determined by a series of \( HV \_0 \) and \( HV \_R \) values based on hardness data for both alloys.](image)

To illustrate how large an effect the choice of the \( HV \_0 \) and \( HV \_R \) values has on the analysis of the recrystallization kinetics, the results for a series of \( HV \_0 \) and \( HV \_R \) values are plotted in figure 5. The \( HV \_0 \) and \( HV \_R \) values are shown by dashed lines in figure 1. Even though these values are not very different, they have a huge effect on the \( n \) values, in particular for the AA5657 alloy in which the \( n \) value varies from 4.9 to 2.7. This clearly illustrates how critical it is to properly determine the two
hardness values of $HV_0$ and $HV_R$ is, if hardness data are to be used to quantify recrystallization kinetics. Two methods to avoid such problem are suggested in the following.

**Method I.** In this method it is assumed that recovery is largely complete when recrystallization starts, and the $Vv$ values determined by EBSD are used to fit $HV_0$ and $HV_R$ by linear fitting. The fitted values together with the calculated $Vv(hardness)$ by equation (4) are given in figure 6. It is obvious that this method gives excellent agreement between $Vv(EBSD)$ and $Vv(hardness)$. However, the approach is impractical because of the many EBSD measurements needed. We therefore also tried to fit $HV_0$ and $HV_R$ only using 3 EBSD data points, corresponding to $Vv$ of 0.05, 0.26, 0.96 and 0.05, 0.52, 0.95 for AA512 and AA5657 respectively. Quite good agreements with the much more complete fitting approach are found. This is illustrated by Avrami plots in figure 6. The good agreement suggests that appropriate values for $HV_0$ and $HV_R$ can be obtained based on only 3 fitting points as long as these are well distributed over the recrystallization interval.

**Figure 6.** Avrami plot of the evolution of the recrystallized volume fraction ($Vv$) based on hardness data by 2 series of $HV_0$ and $HV_R$ values fitted from all EBSD points and 3 EBSD points respectively.

**Figure 7.** (a) An estimate of a ‘continued’ $HV_{recovery}$ curve and (b) Avrami plot of the evolution of recrystallized volume fraction ($Vv$) of the AA5182 alloy based on ‘continued’ recovery.

**Method II.** For an alloy such as AA5182 it is not clear if recovery continues during recrystallization in the not yet recrystallized regions. Concurrent recovery and recrystallization has, for example, been observed in iron [16] and in copper [17, 18]. For the present AA5182 alloy, we find that the initial drop in hardness is related only to recovery. When the hardness values are plotted as a function of the log annealing time, a straight line is observed to fit these initial points well (see figure 7(a)). Assuming that recovery continues to follow this line while recrystallization takes place, $Vv$ may be determined by the equation (5), where $HV_{recovery}$ is determined from the straight line extrapolated to the relevant annealing times. The Avrami plot is shown in figure 7(b). The agreement with method I is excellent.

$$Vv = \frac{HV_{recovery}-HV}{HV_{recovery}-HV_R}$$  (5)
4. Conclusions and outlook

The annealing kinetics of two alloys of the 5xxx series have been analysed by EBSD and hardness measurements. Two methods have been proposed by which the hardness data can be converted to give an adequate description of the recrystallization kinetics. It is found that:

- The recrystallization kinetics cannot be described by a single Avrami exponent for either of the two alloys.
- When analysed based on hardness data, the resulting Avrami exponents depend critically on the choice of transition points from recovery to recrystallization.
- Both methods suggested for conversion of the hardness values to $Vv$ values are adequate. Method I requires normalization based on EBSD maps for at least 3 recrystallization conditions, while method II relies solely on the hardness measurements but requires many data points to be measured in the annealing time interval when only recovery (and not recrystallization) takes place.

It is of interest to notice that the resulting Avrami exponents for the alloys are significantly different: 1.6 in the AA5182 alloy and 2.7 in the AA5657 alloy. This is likely to be because of different nucleation and growth mechanisms, which are related to the difference in deformation microstructures and particle contents in the two alloys. A further investigation of this is planned for a next publication.

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