

Evaluation of the kinetics of recrystallization based on the scaling 1D cellular automaton by DSC measurement

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Abstract. There are several methods to study the kinetics of the recrystallization process. DSC measurement is one of the most effective measurement methods. Cellular automata simulations are frequently used in computational studies. Both methods give and use kinetic constants. The aim of this study to find the relation between the kinetic constants. Additionally an evaluation method is introduced based on the DSC evaluation and cellular automaton which can compute a more detailed kinetic description of recrystallization.

1. Introduction

The kinetic analysis of recrystallization is one of the most important steps in the simulation studies [1]. There are several methods which give a proportional measure to recrystallized volume fraction [2]. Optical microscopy can show a detailed picture about the recrystallization, but in metals where the recovery plays an important role, we cannot give any information about it. But certain metallographic measurement and evaluation of the kinetics of nucleation and growth can also be analyzed [3]. The results of mechanical testing or resistivity measurement can also provide the recrystallization volume fraction and can additionally show the effect of recovery too [4]. In several cases calorimetry, especially DSC measurement is used to evaluate the changes of recrystallized volume fraction as a function of time and temperature [5]. The analysis of DSC measurement is well studied and developed. The disadvantage of this methods is it doesn't show information about the microstructure and the recovery. But in several cases the kinetic constants from DSC evaluation are enough for simulating property changes.

Cellular automaton is one of the oldest non-conventional computation methods used in simulation of phase transformation processes [6]. This method is successfully applied in the simulation of recrystallization [7,8]. Cellular automaton is a beloved simulation technique in this field, because this can reproduce the stochastic nature of the nucleation and growth processes [9]. The major problem in the application of these simulations is the scaling. The automata work with arbitrary units, but we need natural terms. It is possible to simplify the simulation to 1D as much as possible [10]. The big advantage of 1D is that the number of kinetic constants decreases to the minimal number, but the simulation computes all necessary aspects of the recrystallization [11]. The results in this case is not as sophisticated as a 2D [12] or 3D [13,14] computation and cannot cover all detailed phenomena of course. But the computation become extremely fast.

The 1D cellular automata simulations of recrystallization also use the kinetic constants of nucleation and growth. Using these values, the scaling of the 1D automata becomes an easy optimization process

[15]. Due to the arbitrary units of the automaton the question on the meaning of these numbers is important. These values on one side could be only parameters of the simulation, and are only used by the automata. On the other side, the scaling provides physical meaning for the arbitrary units. Hence the used kinetic constants can have a relation to that kinetic equations which are used by the DSC evaluation. The main aim of the recent study is to examine this question.

Pure copper (OFHC Cu) is used to study the recrystallization process to avoid the disturbing effect of recovery. DSC measurements were performed, and a 1D recrystallization automaton was scaled to the measured results. The kinetic constants from the DSC evaluation and from the automaton will be compared.

2. Materials and methods

Continuously casted OFHC copper slab was heat treated at 400°C for 1h. The thickness of the slab was 10mm. The slab was rolled at room temperature with a Von-Roll laboratory stand to 2mm thickness. DSC samples were machined from the cold rolled sheet. The samples were heated up in a Netsch DSC 204 heat flux DSC equipment at 10K/min, 15K/min, 25K/min and 30K/min.

The DSC curves were analyzed with a self-developed software. The transformed volume fraction was calculated from the peaks according to [15]. The curves were evaluated with Kissinger [16] and Takhor [17] method. The Takhor method calculates the activation energy and rate constant of the recrystallization process. The Ozawa method was used to evaluate the value of the Avrami exponent.

The 1D cellular automaton used was published in [10]. The scaling was performed according to [15]. The scaling was made in the same time unit as the rate constant was calculated. The linear size in this case is an independent variable, and the grain size was not calculated in the recent study. The scaling procedure gives the activation energy of nucleation and growth during the simulated recrystallization process.

3. Results and discussion

Figure 1 shows the DSC curves and the recrystallized volume fraction of the cold rolled OFHC Cu samples. On the curves it is clearly seen that the recrystallization process started around 200°C and finished at 300°C. As the heating rate increases, the start, finish, as well as the peak temperature shifts toward the higher temperature range. Around the peaks it is possible to apply a linear base line to determine the peaks. Based on the integral to the peaks it is possible to determine the transformed volume fraction.

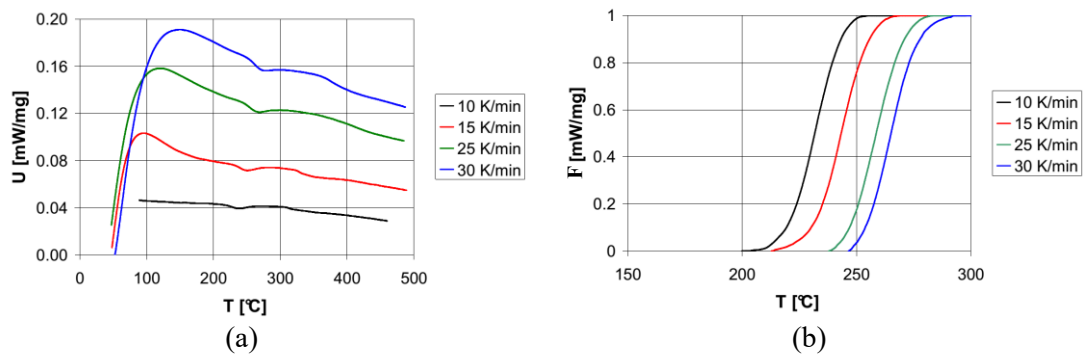


Fig. 1. The DSC curves (a) and the transformed volume fraction (b) as a function of temperature at different heating rates.

There are several thermo-kinetic methods to evaluate the DSC curves. The Kissinger and Takhor method are based on the shift of peak temperature (T_{peak}) as a function of heating rate (v). The basic equations of both methods are shown as eq. 1 and 2. Both methods calculate the activation energy of recrystallization (Q_{rex}) and the rate constant (B_{0rex}).

$$\ln\left(\frac{v}{T_{peak}^2}\right) = -\frac{Q_{rex}}{R} \frac{1}{T_{peak}} + \ln\left(\frac{B_{0rex}R}{Q_{rex}}\right) \quad (1)$$

$$\ln\left(\frac{v}{T_{peak}-T_{start}}\right) = -\frac{Q_{rex}}{R} \frac{1}{T_{peak}} + \ln(B_{0rex}) \quad (2)$$

The symbols R and T_{start} denote the gas constant and the starting temperature at the given measurement in the equations above. Figure 2 shows the left values of the eq. 1 and 2 as a function of reciprocal temperature. The points are laying on a straight line with slope $-Q_{rex}/T_{peak}$. The rate constant can be computed from the intersection of the lines.

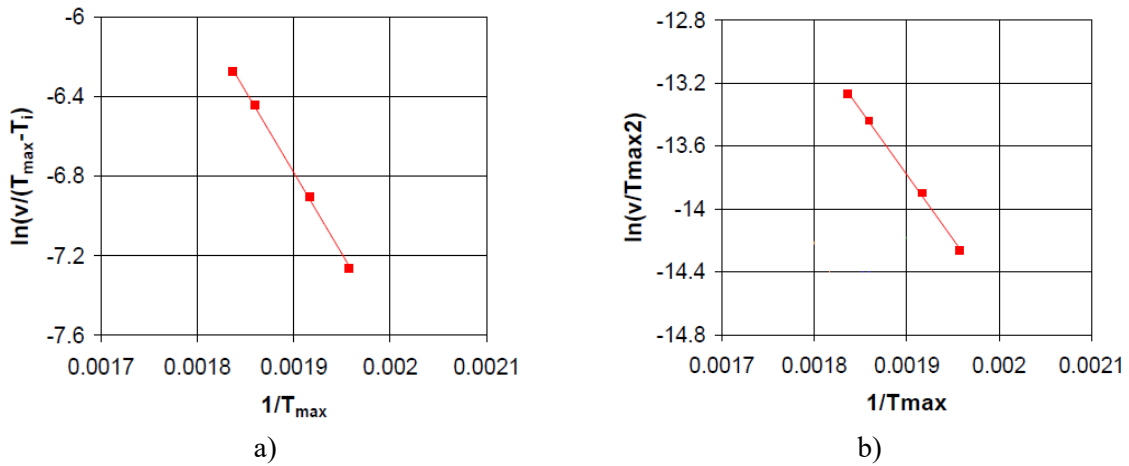


Fig. 2. The determination of the kinetic constants at 80% reduce based on eq. 1 and 2. a): Takhor method, b) Kissinger method.

The analysis gives 58589J/mol and 58523J/mol to the activation energy of recrystallization based on the Kissinger and Takhor method. A small difference exists between the two number. This small difference is enough to cause a large difference in the rate constant. The value of this is 3183.9 1/s and 486.6 1/s based on the Kissinger and Takhor method. Examining the properties of the JMAK equation it becomes evident that both parameters describe the same process.

The Avrami exponent is the missing value. Neither the Kissinger nor the Takhor method can determine this value. There are several approximations to calculate the Avrami exponent. Based on Kissinger's theory the half width of the peaks are proportional to the Avrami exponent. Equation 3 shows this relation. For the calculation of the Avrami exponent the value of the activation energy is necessary. The value from the Kissinger's analysis is used. All peaks give an exponent, the average is calculated. The Avrami exponent based on this analysis is 3.26.

$$n = \frac{2.5T_{peak}^2}{\Delta T_{1/2} \frac{Q_{rex}}{R}} \quad (3)$$

After the detailed kinetic analysis of the curves the [18] cellular automata simulation was scaled with the transformed volume fraction curves based on the method [15]. During the scaling a difference between the curves was defined, and the activation energy of the nucleation and growth were changed by a simplex method. The minimum of the difference between the measured and simulated recrystallized volume fraction was determined based on the mentioned kinetic constants. Figure 3 shows the results of the scaling.

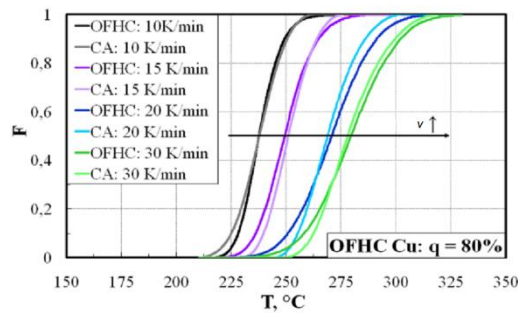


Fig. 3. The recrystallized volume fraction as a function of temperature based on the DSC measurement and scaled cellular automata computation.

The value of activation energy of nucleation (Q_n) is 36627.3 J/mol, and growth (Q_g) is 9890.5 J/mol. Originally these values are used in the automata for simulations studying annealing. But the well-known equation 4 provides a connection between the activation energies.

$$Q_{rex} = Q_n + (n - 1)Q_g \quad (4)$$

Using the values from the scaling and the Avrami exponent from the analysis above, the computed activation energy of recrystallization is 58980 J/mol, which is a bit higher than the Kissinger's or Takhor's value. But these values are suitable to make a detailed study, or simulation without metallographic sample and measurement.

4. Summary

Recrystallization of cold rolled OFHC Cu samples were studied by DSC measurement. The DSC curves were analyzed using Kissinger's and Takhor's theory and the activation energy and rate constant were computed. The Avrami exponent is calculated from the half width of the DSC peaks. Additionally, a recrystallization 1D cellular automaton was scaled with the DSC results. During the scaling the value of the activation energy of nucleation and growth were changed to find the best fit of the measured and simulated transformed volume fraction curves. The activation energies from the kinetic analysis and from the scaled simulation are comparable based on the Avrami exponent. Completing the DSC measurements with these fittings provide a more in-depth kinetic description of the process without information about the microstructure. Of course, the introduced method cannot provide information on grain size, for this reason metallographic examinations are still necessary.

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