# Recrystallization Kinetics of Fe-3%Si after Deformation at High Strain Rate and High Temperature

An Fenghui<sup>a</sup> \*<sup>10</sup>, Liu Bo<sup>a</sup>, Zhang Deqin<sup>a</sup>, Liu Jinlong<sup>b</sup>, Sha Yuhui<sup>b</sup>

<sup>a</sup>School of Mechanical and Materials Engineering, Jiujiang University, Jiujiang Jiangxi 332005, China <sup>b</sup>School of Materials Science and Engineering, Northeastern University, Shenyang Liaoning 110819, China

Received: November 21, 2018; Revised: March 08, 2019; Accepted: April 29, 2019

Recrystallization kinetics of Fe-3%Si with initial grain size 296~839 $\mu$ m at the temperature range 1223~1423K, strain range from 0.51 to 0.92, strain rate range 5~80s<sup>-1</sup> was studied by isothermal compression test using Gleeble-2000. The effect of strain, strain rate, initial grain size and annealing temperature on the recrystallization kinetics were discussed. Avrami equation was used to describe the static recrystallization kinetics. Moreover, the effect of initial grain size and annealing temperature on the exponent in Avrami equation was discussed and the reason of the exponent deviating from the theory value is owed to the effect of static recovery. In all the experimental range the static recrystallization kinetics can be well predicted by Avrami equation.

Keywords: Fe-3%Si, recrystallization kinetics, exponent in avrami equation, prediction.

# 1. Introduction

As one of the most important soft magnetic materials, grain oriented silicon steel is widely used in electrical industry. Its excellent magnetic property comes from the sharp Goss texture realized by secondary recrystallization. Homogeneous microstructure is the premise for second recrystallization taking place, and it needs recrystallization taking place in hot rolling process. On the other hand, grain oriented silicon steel is a ferrite material in high temperature range. The investigation about the static recrystallizatin kinetics of austenite steel is sufficient and many models have been set up to predict the recrystallization kinetics in the practical production<sup>1-7</sup>. Although the investigation of static recrystallization about ferrite steel has been carried out early<sup>8,9</sup>, systematic results have not been obtained. Recently, hot deformation in the ferrite range has been paid attention in the relative lower temperature range<sup>10-12</sup>, while the investigation of recrystallization in high deformation temperature range is still scarce. Akta et al.13 have studied the static recrystallization behavior of Fe-3%Si steel in the high temperature range from 1173K to 1373K and strain rate range from 2.5 to 5s<sup>-1</sup>. In the practical production, the strain rate is high in the range from 1 to 120s<sup>-1</sup> and the deformation temperature is in the range 1123K to 1473K. With the increase of deformation strain rate and annealing temperature, the kinetics of static recrystallization will be accelerated. For predicting the recrystallization kinetics in the practice, the static recrystallization kinetics in higher strain rate range at relative high temperature needs to be further discussed.

The aim of present paper is to pay attention to the static recrystallization kinetics of Fe-3%Si after deformation at high strain rate and temperature by Gleeble-2000. The effects of initial grain size, strain, strain rate and annealing temperature on the recrystallization kinetics were discussed. Consequently, Avrami Equation was used to regress and predict the recrystallization kinetics in the present experimental range.

### 2. Material and Experimental

Fe-3%Si alloy used in the present experiment was melted with induction furnace and cast into cylindrical ingot with the chemical compositions (wt.%) of 3.06Si, 0.015C, 0.024Mn, 0.006S, 0.008P, 0.01Cu, 0.01Al, 0.02Ni, 0.03Cr and bal. Fe. The ingot was first homogenized at 1473K for 1h, then hot forged to square billet and further hot rolled to sheets with thickness of 20mm. To obtain medium size grains, the sheet was normalized at 1423K for 10min. For obtaining the smaller size grain, the sheet was further rolled to 12mm at 873K and normalized at 1423K for 10 min. For obtaining the larger size grain, the sheet was normalized at 1523K for 20min. After that the sheets were cut into cylindrical specimens with diameter of 10mm and height of 15mm.

Hot deformation test was carried out on Gleeble-2000 thermo-simulation machine in the temperature range from 1223K to 1423K and strain rate range from 5s<sup>-1</sup> to 80s<sup>-1</sup>. Two end surfaces of specimen were painted with graphite sheet to reduce the friction between specimen and crosshead. During compression, each specimen was heated to the deformation temperature at 10K/s and then held for 90s to eliminate any thermal gradient. After a reduction of 40% or 60%, the specimen was hold for a given time at the deformation temperature isothermally, then rapidly quenched with water controlled by computer. Compression test was performed in argon atmosphere to prevent oxidation. Quenched specimens were sectioned parallel to the compression axis for metallographic analysis and the point count method<sup>14</sup> was used to calculate the recrystallization fraction.

## 3. Results and Discussion

# 3.1. Microstructure and Static recrystallization kinetics curves

Microstructure of deformed samples were analyzed by optical microscopy. The typical microstructure were shown in Fig. 1 corresponds to samples of different annealing time after deformation with strain 0.92 at the strain rate 30s<sup>-1</sup> and temperature 1323K. From Fig. 1(a), it can be seen that the grain boundaries especially the edge or corner are the preferential nucleation sites, and the recrystallization was controlled by the grain growth as shown in Fig. 1 (b) and (c). The recrystallization fractions corresponding to different conditions are shown in Fig. 2 with symbols.

From the experimental results shown in Fig. 2, it can be seen that the kinetics of static recrystallization in the experiment range show the typical sigmoid shape which indicate that the kinetics of static recrystallization isothermally can be described by Avrami Equation<sup>15</sup> that is usually rewritten as<sup>1-7,16</sup>:

$$X_{rex} = 1 - \exp\left[-0.693 \left(\frac{t}{t_{0.5}}\right)^k\right]$$
(2)

In which,  $t_{0.5}$  is the time used for half recrystallization fraction. Eq.(2) was used to fit the experimental results, and the best fitting lines was also shown in Fig. 2. The values of  $t_{0.5}$  and k corresponding to different experimental condition were obtained and shown in Table 1, from which it can be seen that the value of k is obviously affected by initial grain size and annealing temperature, while the strain and strain rate have little effect. According to the theory of Avrami, the value of k is affected by the growth dimensionality of the recrystallized nucleus when the growth rate keeps constant and the nucleation rate is constant or the nucleation is completed due to the higher nucleation rate in the recrystallization processing. The value of k is shown



**Figure 1.** Microstructure of Fe-3%Si steel after deformation with the strain of 0.92 at the strain rate  $30s^{-1}$  and temperature of 1323K and annealed with time (a) 1s ( $X_{rex}=2\%$ ); (b) 5s ( $X_{rex}=51\%$ ); (c) 10s ( $X_{rex}=77\%$ ); (d) 100s ( $X_{rex}=100\%$ ).



**Figure 2.** Effect of initial grain size (a), strain rate (b), strain (c) and temperature (d) on the kinetics of recrystallization. (a)  $d_0=520$  um,  $\epsilon=0.92$ ,  $T_{rex}=1223$ K; (b)  $d_0=520$  um,  $\epsilon=30s^{-1}$ ,  $T_{rex}=1323$ K; (c)  $\epsilon=0.92$ ,  $\epsilon=30s^{-1}$ ,  $T_{rex}=1323$ K; (d)  $d_0=520$  um,  $\epsilon=0.92$ .

$d_0(\mu m)$	3	$\boldsymbol{\varepsilon}^{\boldsymbol{\cdot}}(\mathbf{s}^{-1})$	<b>T</b> (K)	$t_{0.5}(s)$	k
520	0.92	5	1223	83	0.89
520	0.92	30	1323	5.2	1.20
520	0.92	30	1423	1.6	1.75
296	0.92	30	1323	2.4	2.00
839	0.92	30	1323	15	0.97
520	0.51	30	1323	50.6	1.20
520	0.92	80	1223	30	0.93

 
 Table 1. Regression results for time to 50% recrystallization and k in Avrami equation.

Table 2. Ideal exponents in Avrami equation.

Growth dimensionality	Constant nucleation rate	Nucleation is over
1-D	4	3
2-D	3	2
3-D	2	1

in Table 2 with different growth dimensionality. Cahn<sup>17</sup> has extended the theory of Avrami to nucleation on different site and made a conclusion that if the nucleation is over in the early stage of recrystallization, the value of k is 1, 2 and 3 corresponding to the nucleation on grain boundary, grain edge and grain corner.

According to the theory of Avrami and Cahn,  $t_{0.5}$  is a function of the grain growth rate. If the growth dimensionality and the grain growth rate were kept constant during the recrystallization processing, the value of k should be kept constant. In fact, continuous varying of  $t_{0.5}$  in the recrystallization processing due to the effect of other factors including recovery, dynamic recrystallizatin18,19, precipitation19-21 also change the value of k when using Avrami equation to regress the experimental results. For example, if the static recovery occurs prior to static recrystallization which decreases the deformation energy of unrecrystallization region, the grain growth rate would be decreased and the time corresponding to the half recrystallization is prolonged, as shown schematically in Fig. 3. The best fitting lines for part and whole the experimental results were signed with dot and solid lines which were found in aluminium alloy, copper<sup>22</sup> and iron<sup>18</sup>. Humphreys and Hatherly considers that Avrami equation is too simple to quantitatively model the complex recrystallization process. In other words, Avrami equation can be used to model the recrystallization to satisfy the need of practical production, though the physical significance of k will be different to that proposed by Avrami originally.

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Figure 3. Schematically illustrate the effect of the time for half recrystallization on the value of k.

#### 3.2. Prediction of recrystallization kinetics

The time for half recrystallization fraction can be affected by the initial grain size  $(d_0)$ , strain  $(\varepsilon)$ , strain rate  $(\varepsilon)$  and annealing temperature (T), so the time for half recrystallization  $(t_0)$  can be expressed as Eq. (3)<sup>5-7</sup>

$$t_{0.5} = A d_0^1 \boldsymbol{\varepsilon}^m \dot{\boldsymbol{\varepsilon}}^n \exp\left(\frac{Q_{rex}}{RT}\right) \tag{3}$$

Where *A*, *l*, *m* and *n* are material dependent constants, *R* has its usual meaning, and  $Q_{rex}$  is the apparent activation energy for recrystallization. Take nature logarithm, Eq.(3) can be written as

$$\ln(t_{0.5}) = \ln(A) + l\ln(d_0) + m\ln(\varepsilon) + n\ln(\dot{\varepsilon}) + \frac{Q_{rec}}{RT}$$
(4)

The relationship between the time for half recrystallization fraction and the inverse of annealing temperature is shown in Fig. 4, obviously, it has an approximately linear relationship.



Figure 4. Effect of annealing temperature on time to 0.5 fraction of recrystallization.

By multivariate linear regress, the constants in Eq.(4) can be obtained as  $A = 6.5 \times 10^{-14}$ , l = 1.76, m = -3.47, n = -0.38 and  $Q_{rex} = 243.6$  kJ/mol. The static recrystallization activation energy is only slightly higher than 230 kJ/mol reported by Akta et al. for Fe-3%Si steel in the temperature rang from 1173K to 1373K<sup>13</sup>, close to the self diffusion activation energy of 239.3 kJ/mol in high-purity iron<sup>23</sup> and higher than the self-diffusion activation energy of Fe atom in Fe-3%Si alloy ranging from 218 kJ/mol<sup>24</sup> to 220kJ/mol<sup>25</sup>.

As discussed above, the value of k is dependent on the initial grain size and annealing temperature. It is almost independent on the strain and strain rate in the experiment range. Accordingly, the value of k can be expressed as follows:

$$k = Bd_0^f \exp\left(\frac{Q_k}{RT}\right) \tag{5}$$



Figure 5. Relationship of n with initial grain size (a) and annealing temperature (b).





Figure 6. Comparison of the predited value t<sub>0.5</sub> and n with those of experiment.

Fig. 5 shows the relationship of k with the initial grain size and annealing temperature. The parameters in Eq.(5) can be calculated by using the same method as dealing with the parameters in Eq.(3) by the value in Table 1 and The calculated results are  $B = 7.8 \times 10^3$ , f = -0.70 and  $Q_k = -47.5 \text{ kJ/mol.}$ 

From the discussion above, it can be seen that the accuracy of prediction about the static recrystallization kinetics is dependent on the time for half recrystallization and the value of k. For examining the accuracy of the mode to predict the static recrystallization, the value of  $t_{0.5}$  and k obtained by experiment and prediction were shown in Fig. 6, from which it can be seen that the results obtained by prediction are in good agreement with those obtained by experiment.

## 4. Conclusions

Static recrystallization kinetics of Fe-3%Si was studied after deformation at temperature 1123~1423K and strain rate 5~80s<sup>-1</sup> by Gleeble-2000, and the main results obtained as follows:

 In the test range, the kinetics of static recrystallization of Fe-3%Si can be satisfactorily described by Avrami equation and the time for half recrystallization and exponent can be further expressed as

$$t_{0.5} = 6.5 imes 10^{-14} d_0^{1.76} oldsymbol{arepsilon^{-3.47}} \dot{oldsymbol{arepsilon^{-3.47}}} \dot{oldsymbol{arepsilon^{-3.47}}} iggl( rac{243600}{RT} iggr) \ k = 7.8 imes 10^3 d_0^{-0.7} \exp iggl( rac{-47500}{RT} iggr)$$

 The value of k is affected not only by the growth dimensionality of recrystallized nucleation as theory but also by the recovery or precipitation, which is the reason that the value of k obtained by experimental result different to that as theory.

#### 5. References

- Sellars CM. Modelling microstructural development during hot rolling. *Materials Science and Technology*. 1990;6(11):1072-1081.
- Siwecki T. Modelling of Microstructure Evolution during Recrystallization Controlled Rolling. *ISIJ International*. 1992;32(3):368-376.
- Hodgson PD, Gibbs RK. A Mathematical Model to Predict the Mechanical Properties of Hot Rolled C-Mn and Microalloyed Steels. *ISLJ International*. 1992;32(12):1329-1338.
- Kwon O. A Technology for the Prediction and Control of Microstructural Changes and Mechanical Properties in Steel. *ISIJ International*. 1992;32(3):350-358.
- Serajzadeh S, Karimi Taheri A. An investigation of the silicon role on austenite recrystallization. *Materials Letters*. 2002;56(6):984-989.
- Serajzadeh S, Karimi Taheri A. An investigation on the effect of carbon and silicon on flow behavior of steel. *Materials & Design*. 2002;23(3):271-276.
- Bianchi JH, Karjalainen LP. Modelling of dynamic and metadynamic recrystallisation during bar rolling of a medium carbon spring steel. *Journal of Materials Processing Technology*. 2005;160(3):267-277.
- Glover G, Sellars CM. Static recrystallization after hot deformation of α iron. *Metallurgical Transactions*. 1972;3(8):2271-2280.
- Cetlin PR, Yue S, Jonas JJ, Maccagno TM. Influence of strain rate on interpass softening. *Metallurgical Transactions* A. 1993;24(7):1543-1553.
- Sakai T, Saito Y, Hirano K, Kato K. Deformation and Recrystallization Behavior of Low Carbon Steel in High Speed Hot Rolling. *Transactions of the Iron and Steel Institute of Japan.* 1988;28(12):1028-1035.
- Cetlin PR, Yue S, Jonas JJ. Simulated Rod Rolling of Interstitial Free Steels. *ISIJ International*. 1993;33(4):488-497.

- Akbari GH, Sellars CM, Whiteman JA. Static restoration processes in warm rolled interstitial free steel. *Materials Science* and Technology. 2002;18(8):885-891.
- Akta S, Richardson GJ, Sellars CM. Hot Deformation and Recrystallization of 3% Silicon Steel Part 1: Microstructure, Flow Stress and Recrystallization Characteristics. *ISIJ International*. 2005;45(11):1666-1675.
- 14. Rossi PLO, Sellars CM. Quantitative metallography of recrystallization. *Acta Materialia*. 1997;45(1):137-148.
- Avrami M. Kinetics of Phase Change. I General Theory. *The Journal of Chemical Physics*. 1939;7(12):1103-1112.
- Sellars CM, Whiteman JA. Recrystallization and grain growth in hot rolling. *Metal Science*. 1979;13(3-4):187-194.
- Cahn JW. The kinetics of grain boundary nucleated reactions. Acta Metallurgica. 1956;4(5):449-459.
- Sandström R, Lagneborg R. A model for static recrystallization after hot deformation. Acta Metallurgica. 1975;23(4):481-488.

- Laasraoui A, Jonas JJ. Recrystallization of austenite after deformation at high temperatures and strain rates-Analysis and modeling. *Metallurgical Transactions* A. 1991;22(1):151-160.
- Medina SF, Mancilla JE. Determination of Static Recrystallization Critical Temperature of Austenite in Microalloyed Steels. *ISIJ International*. 1993;33(12):1257-1264.
- Medina SF, Mancilla JE. Influence of Alloying Elements in Solution on Static Recrystallization Kinetics of Hot Deformed Steels. *ISIJ International*. 1996;36(8):1063-1069.
- Humphreys FJ, Hatherly M. Recrystallization and Related Annealing Phenomena. Oxford: Elsevier; 2012.
- Buffington FS, Hirano K, Cohen M. Self diffusion in iron. Acta Metallurgica. 1961;9(5):434-439.
- Mills B, Walker GK, Leak GM. The diffusion of iron in 3% silicon iron. The Philosophical Magazine: A Journal of Theoretical Experimental and Applied Physics. 1965;12(119):939-942.
- Young WS, Mykura H. Mass transfer diffusion measurements in Fe-3.8% Si crystals. Acta Metallurgica. 1965;13(5):449-452.