

Deformation behavior of electro-deposited pure Fe and its texture evolution during cold-rolling and subsequent annealing

N Sugiura¹ N Yoshinaga¹ and K Ushioda¹

¹Nippon Steel and Sumitomo Metal Corp, Futtsu, JAPAN

E-mail: ushioda.cd8.kohsaku@jp.nssmc.com

Abstract. Electro-deposited pure Fe has a characteristic of having very sharp isotropic ND//<111> fiber texture with a needle-like shaped fine grain elongated to ND. This Fe exhibits a surprisingly high r-value of over 7; however, such a high r-value might not be rationalized only from texture. Careful slip analyses reveal that restricted slips take place in the specific {110} slip planes, which are perpendicular to the sheet surface. Since grain boundaries with columnar structure are also perpendicular to the sheet surface, the slip plane in a certain grain may easily connect to the slip plane in adjacent grains having within ± 30 degree rotation relationship around the common axis of ND//<111>. The operation of such a slip system is considered to cause the width strain much larger than the thickness strain. Furthermore, the texture evolution during cold-rolling and subsequent annealing was investigated using electro-deposited pure Fe as a starting material. Regardless of the amount of cold-rolling reduction, 65% to 90%, {111}<112> cold-rolling texture developed. After recrystallization, {111}<112> texture remained when material is cold-rolled by 65%, while {111}<011> texture developed when materials are cold-rolled by 80% and 90%. From the investigation into the mechanism on the development of recrystallization texture, the oriented nucleation and selective growth theories are concluded to contribute to the evolution of annealing texture.

1. Introduction

Steel products, which have been widely used for various applications, have greatly contributed to society. However, the potential capabilities of steels such as strength, formability and magnetic property, have not been fully exploited. In this sense, steels are very attractive materials having many possibilities.

In this paper, the studies of deep drawability [1] and tensile behavior [2, 3] of the electro-deposited pure Fe are reviewed because of its unique characteristic of having extremely sharp isotropic ND//<111> fiber textures with a fine columnar structure in the thickness direction. This Fe exhibits a surprisingly high r-value and local elongation, which indicates the remaining space for Fe to further improve its properties by controlling texture and structure. Moreover, the study on the texture evolution during cold-rolling and subsequent annealing using the electro-deposited pure Fe as a starting material [4] is reviewed so that we could have an insight into the nature of texture formation mechanism in Fe.

2. Electro-deposited pure Fe

The material used is the electro-deposited pure Fe produced by Toho Zink Co., Ltd., the chemical composition of which is listed in Table 1. The structure and texture close to

Table 1 Chemical composition of electro-deposited pure Fe used (mass %).

C	Si	Mn	P	S	Al	N	O
0.0006	0.0001	0.0001	0.0001	0.0002	0.0002	0.0009	0.0018



Content from this work may be used under the terms of the [Creative Commons Attribution 3.0 licence](https://creativecommons.org/licenses/by/3.0/). Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.

the electrode is different from that in the central region. Therefore, the original material with the thickness of about 2 mm is ground from both surfaces to 0.8 to 1.0 mm thickness. The electro-deposited pure Fe comprised a needle-like shaped fine grain elongated to ND (Fig. 1 (a)). The grain size observed from ND is about few μm to 30 μm . Moreover, the $\{100\}$ pole figure, shown in Fig. 1 (b), shows that this Fe has extremely sharp isotropic ND// $\langle 111 \rangle$ fiber textures. The crystal growth in the electro-deposition process of bcc Fe implies that the Fe atoms are consistently deposited on $\{111\}$ planes, despite this not being a close-packed or low energy surface in bcc metals.

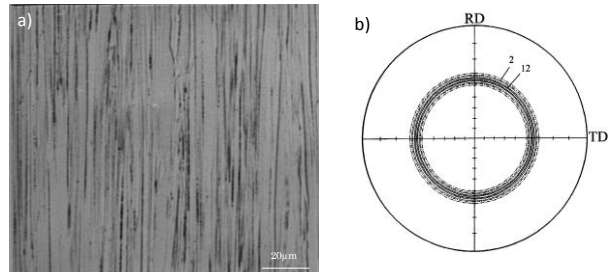


Fig. 1 a) Optical micrograph showing fine columnar structure and b) $\{100\}$ pole figure of electro-deposited pure Fe.

3. Deformation behavior of electro-deposited pure Fe

3.1. Tensile property

The tensile properties of the electro-deposited pure Fe are shown in Table 2. The JIS 13B and JIS 5B (cf. Japanese Industrial Standard Z2241) tensile specimens were used to measure the r -values and the other tensile properties, respectively. The strength level of this Fe is similar to that of the conventional IF (interstitial atoms free) steel; however, the r -value is surprisingly as high as 7.1. Moreover, the total elongation is over 68%, in particular, the post uniform elongation is as large as 55%. The appearance of specimen after tensile test is shown in Fig. 2, which reveals that the specimen is necked significantly and fractured with large width reduction together with subtle thickness reduction.

The prediction of the r -value was carried out in order to check the possibility whether such a high r -value comes from the texture; software developed by Van Houtte [5] was employed for this. The calculation was based on both FC (Full Constraint) model and RC (Relaxed Constraint) model, where the condition of RC model is described in Table 3. The r -value calculated is 2.4 to 2.6 in FC model and 2.3 to 3.2 in RC model, which are significantly smaller than that of experimental values such as 4.8 to 7.1. Therefore, it is implied that there exist other reasons than texture for explaining the high r -value. However, it might be possible to predict r -value as high as 7 considering a needle-like shaped fine grain with sharp ND// $\langle 111 \rangle$ orientation assuming proper plasticity model and grain interaction [6-8].

Table 2 Tensile properties of electro-deposited pure Fe.

TS/ MPa	YS/ MPa	T-El(%)	U-El(%)	L-El(%)	r
288	215	68.5	13.0	55.5	7.1

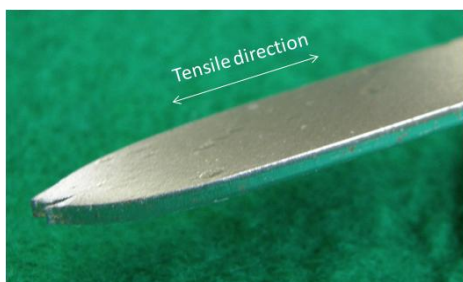


Fig. 2 Appearance of specimen after tensile test.

Table 3 Conditions for calculation of r -value and calculated results.

Relaxed condition	Slip system	r-value calculated
RC1	BCC2	3.16
RC2	BCC1	2.31
RC2	BCC2	2.53
RC3	BCC1	2.56
RC3	BCC2	2.98
RC4	BCC1	2.73
RC4	BCC2	3.17

Condition for Relaxed Constraint model

RC1: e_{13}, e_{23} RC2: e_{12}, e_{21}

RC3: e_{31}, e_{32} RC4: $e_{12}, e_{21}, e_{31}, e_{32}$

Slip system:

BCC1 $\{110\}\langle 111 \rangle + \{112\}\langle 111 \rangle$

BCC2 $\{110\}\langle 111 \rangle + \{112\}\langle 111 \rangle + \{123\}\langle 111 \rangle$

3.2. Deep drawability

In order to verify such a high r -value obtained by the tensile test, a cup drawing test, *i.e.*, TZP test [1, 9], was carried out. The change in the relationship between the stroke and the force for both the first and second draw in the TZP test is described schematically in Fig. 3. Here, the maximum force for first draw and the fracture force in second draw with BHF (Blank Hold Force) are P_m and P_f , respectively. P_f is well known to be related to the r -value. Figure 4 clarifies that the fracture stress obtained experimentally is in good agreement with the one theoretically predicted using the r -value by tensile test [1]. Furthermore, T -value, defined as $(P_f - P_m) / P_f$, was confirmed to correspond to the r -value determined experimentally [1]. Therefore, the extremely high r -value of electro-deposited pure Fe obtained by tensile test may be considered trustable.

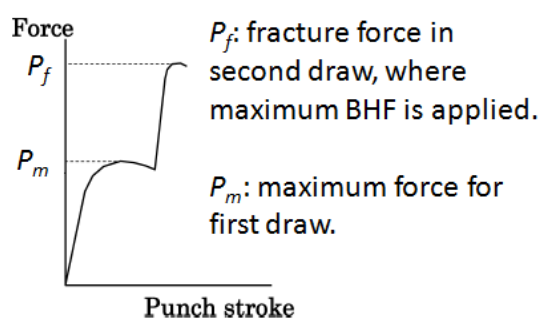


Fig. 3 Schematic illustration showing the change in stroke-force curve during TZP test.

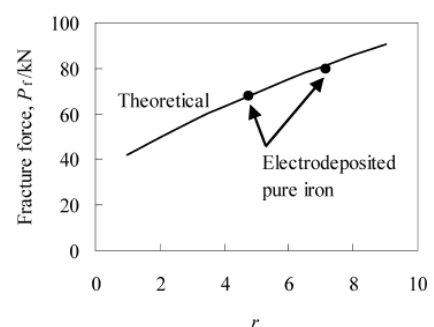


Fig. 4 Relationship between fracture force and r -value in TZP test.

3.3. Analysis of tensile deformation behavior

For the purpose of elucidating the nature of surprisingly high- r value of the electro-deposited pure Fe, the tensile behavior was analyzed in detail [2, 3].

Figure 5 shows the optical micrograph indicating the slip pattern observed from ND after 8% stretching. Many slip patterns aligning 30 to 60 degree from tensile direction were observed. EBSD-IQ map of the same stretched specimen is shown in Fig. 6, where the black contrasts reveal the boundaries of grains of the electro-deposited Fe with complicated shape, while the straight striations observed slightly within the grains marked as the red line correspond to the slip line. Crystal orientation analysis indicates that these striations are $\{110\}$ slip planes perpendicular to the top surface. The red lines in the figure also show the three $\langle 112 \rangle$ directions parallel to the surface as schematically illustrated in Fig. 7. Moreover, the observed slip line is confirmed to be parallel to one of the three $\langle 112 \rangle$ directions.

Electro-deposited pure Fe is inferred to have an extremely high r -value owing to the operation of $\{110\}$ slip system perpendicular to the surface. In the case of the slip system of $\{112\}$ and $\{123\}$, there

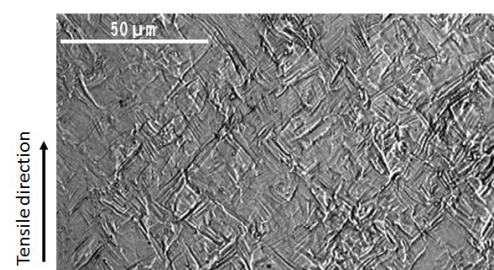


Fig. 5 Optical micrograph showing slip pattern observed from ND after 8% stretching.

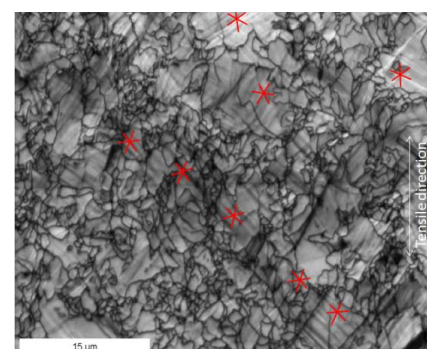


Fig. 6 EBSD-IQ map showing grain boundaries and slip lines observed from ND after 8% stretching.

is only one slip direction of $\langle 111 \rangle$ which is parallel to the thickness direction. This implies that Schmid factors for these slip systems equal to zero. Therefore, the operation of $\{112\}$ and $\{123\}$ slip system becomes impossible. Meanwhile, there are six $\{110\}$ slip planes with two different $\langle 111 \rangle$ slip directions, which leads to 12 slip systems. In the case of sharp ND// $\langle 111 \rangle$ texture, the three $\{110\}$ planes are perpendicular to the surface (Type A), while the other three $\{110\}$ planes incline 35 degree to the surface (Type B). Schmidt factors for these slip systems were calculated, and turned out that two slip systems belonging to Type A have the same maximum value as two slip systems in Type B. Despite the fact that these four slip systems are expected to be active, only the two slip systems belonging to Type A were confirmed to be active, the reason of which is discussed later.

In a grain with ND// $\langle 111 \rangle$ orientation like the electro-deposited pure Fe, there exist three slip planes belonging to Type A. Therefore, it is expected that a neighboring grain with ND// $\langle 111 \rangle$ orientation always has a slip system within ± 30 degree rotation relationship around the common ND// $\langle 111 \rangle$ axis with the original grain. Therefore, the crossing line of two Type A slip planes in the neighboring grains lie on the grain boundary schematically as shown in Fig. 8, because grains are elongated to the thickness direction. Consequently, it is inferred that the plastic deformation in the horizontal directions takes place smoothly because the slip planes are connected to every neighboring grain resulting in the easy transmission of internal stress through grain boundary (Fig. 8). Moreover, this kind of deformation suggests a large width strain compared to a very small thickness strain when the specimen is subjected to the tensile deformation. Meanwhile, the Type B slip systems have a twist relationship in each adjacent grain, which indicate the difficulty in internal stress transmission due to a lack of continuity in slip systems.

Furthermore, the electro-deposited pure Fe, having fine ND// $\langle 111 \rangle$ columnar structure in the thickness direction, is considered to have higher flow stress in the thickness direction as compared to the width direction. Therefore, a large difference in flow stress between thickness and width directions is expected, which also brings about a high r-value. The grain boundary sliding due to the fine columnar structure is reported to be responsible for the high r-value [10], but it is speculated difficult for grain boundary sliding to occur during tensile deformation at room temperature. The evidence of slip line within grain is another reason for the speculation.

4. Recrystallization and texture evolution of electro-deposited pure Fe

The recrystallization behavior and texture evolution in the process of cold-rolling and annealing were investigated using electro-deposited pure Fe as a starting material [4]. The

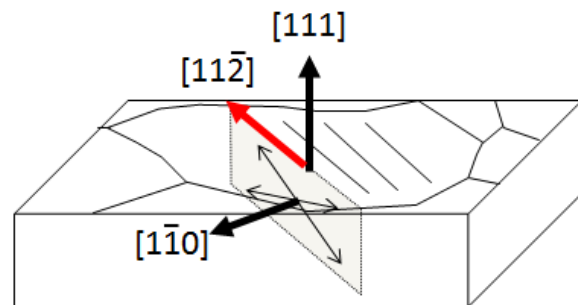


Fig. 7 Schematic illustration showing slip system and slip line at surface.

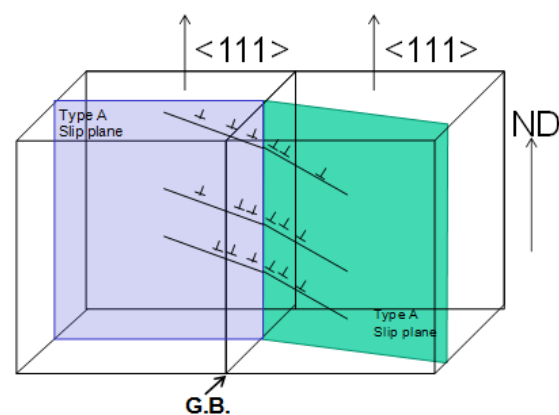


Fig. 8 Schematic illustration of Type A slip systems in neighboring two grains with common ND// $\langle 111 \rangle$ axis, suggesting easy transmission of internal stress through grain boundary.

focus was placed on the influence of cold-rolling reduction, which was changed from 65 and 80% to 90%. There are many previous studies using hot-rolled bands as starting materials; however, no study has been conducted using electro-deposited pure Fe with sharp isotropic ND//<111> texture as a starting material.

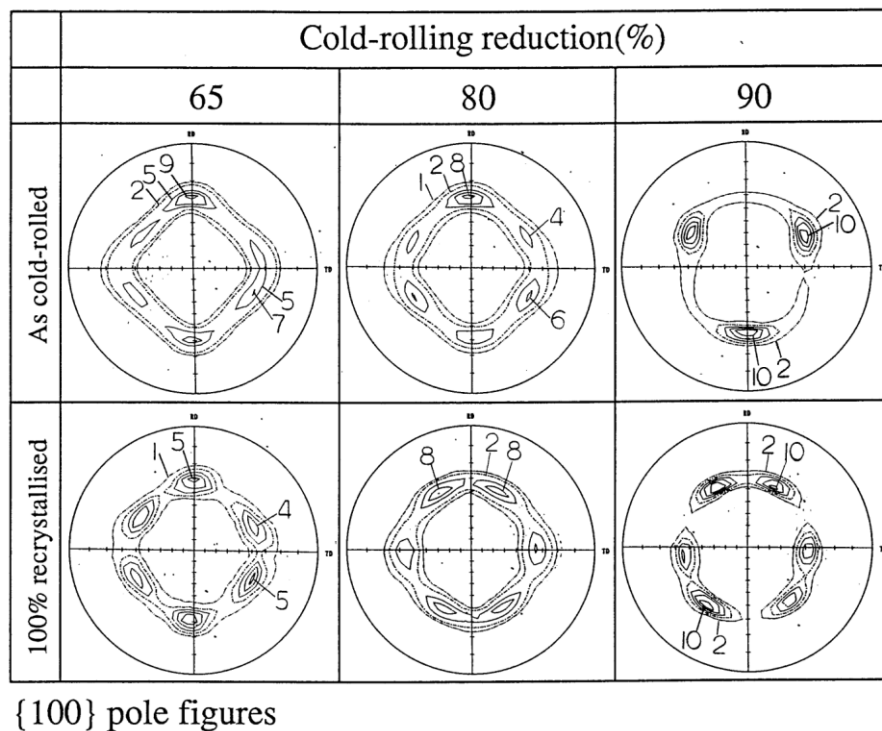


Fig. 9 $\{100\}$ pole figures of cold-rolled and 100% recrystallized sheets as a function of cold-rolling reduction.

Recrystallization commences and completes during iso-thermal annealing at 400 °C of 65% and 80% cold-rolled electro-deposited pure Fe. This very low recrystallization temperature is another characteristic of electro-deposited pure Fe. Figure 9 shows $\{100\}$ pole figures of cold-rolled and 100% recrystallized sheets as a function of cold-rolling reduction. It is generally known that $\{111\}<011>$ texture rather than $\{111\}<112>$ texture relatively increases with increasing cold-rolling reduction; however, it is worthy to note that $\{111\}<112>$ orientation becomes major in this study. Furthermore, $\{111\}<112>$ orientation remains after recrystallization when cold-rolling reduction is 65%; whereas, when the reduction increases to 80%, $\{111\}<011>$ orientation becomes major and this tendency is strengthened by higher cold-rolling reduction by 90%.

In order to investigate the mechanism of the influence of cold-rolling reduction on texture evolution, the orientation of recrystallized nuclei was measured by EBSD, where the fraction recrystallized is about 5%. The ODFs determined by EBSD data for recrystallized grains are shown in Fig.10. The major orientation of recrystallized nuclei turned out

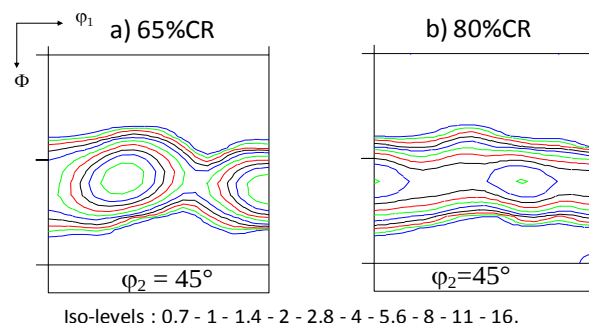


Fig. 10 ODFs of recrystallized grains determined by EBSD data. Fraction recrystallized is 5%, irrespective of cold-rolling reduction of a) 65% and b) 80%..

to be $\{111\}\langle 112 \rangle$ in the case of 65% reduction; whereas, in the case of 80% reduction the intensity of $\{111\}\langle 011 \rangle$ orientation is relatively high with the scattered γ -fiber textures. Therefore, it is obvious that the orientation of nuclei significantly influences the texture after the completion of recrystallization. However, the mechanism is not clearly rationalized only from the orientation nucleation theory.

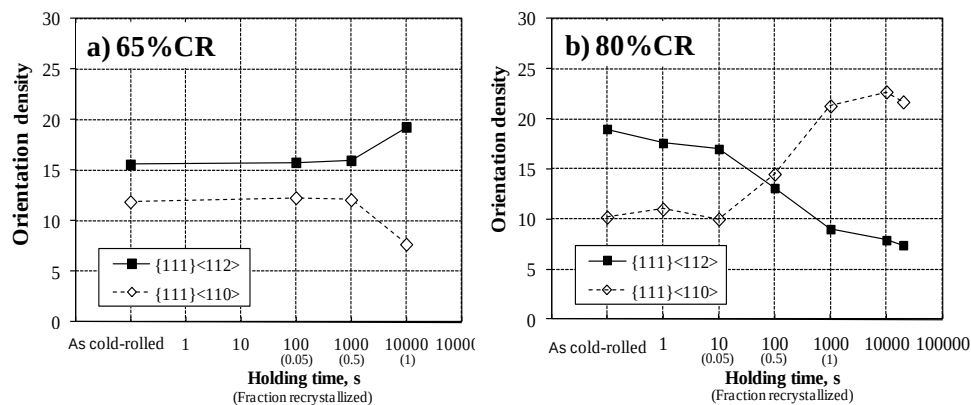
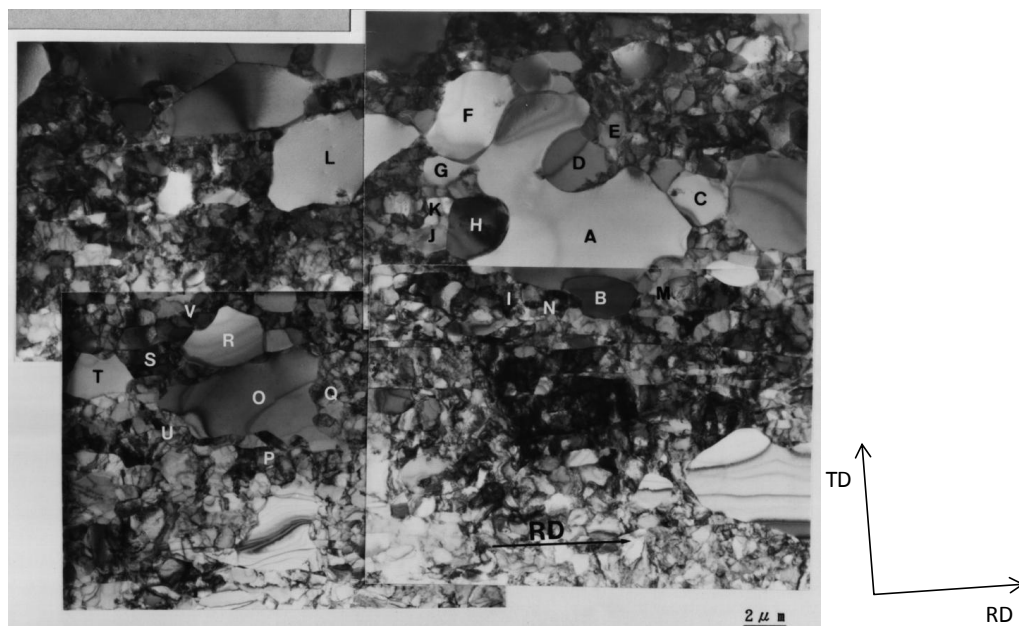


Fig.11 Changes in texture with evolution of recrystallization during annealing at 400 °C for a) 65% and b) 80% cold-rolled materials.



near $\{111\}\langle 110 \rangle$ grains : A, B, G, H, J, K, L, N, O, S, T, U, V

near $\{111\}\langle 112 \rangle$ grains : E, I, P, Q

Fig. 12 TEM micrograph showing recrystallized grains with near $\{111\}\langle 011 \rangle$ and $\{111\}\langle 112 \rangle$ orientations. Cold-rolling reduction: 80%. Fraction recrystallized: 50%.

The texture change with the evolution of recrystallization is significantly different between the two materials cold-rolled by 65% and 80% reduction, as shown in Fig 11 a) and b). Namely, in the case of the material cold-rolled by 65% reduction the intensity of $\{111\}\langle 112 \rangle$ slightly increases while that of $\{111\}\langle 011 \rangle$ decreases. This indicates that the nuclei with $\{111\}\langle 112 \rangle$ orientation grows into the matrix with near $\{111\}\langle 112 \rangle$ orientation. Meanwhile, in

the case of the material cold-rolled by 80% reduction the intensity of $\{111\}\langle 011 \rangle$ orientation increases intensively while that of $\{111\}\langle 112 \rangle$ decreases significantly. This indicates that the nuclei with $\{111\}\langle 011 \rangle$ orientation grows selectively into the matrix with $\{111\}\langle 112 \rangle$ orientation. In general, the recrystallization texture is determined by the product of nucleation and growth of each oriented grain. It was confirmed that the increase in cold-rolling reduction intensifies the rolling texture centering on $\{111\}\langle 112 \rangle$ orientation together with a rather homogenous deformation structure. Therefore, the selective interface migration for the nuclei with $\{111\}\langle 011 \rangle$ orientation into the $\{111\}\langle 112 \rangle$ matrix plays an important role when the cold-rolling reduction becomes over 80%. On the contrary, when the reduction is 65%, the development of stable cold-rolling texture, $\{111\}\langle 112 \rangle$ orientation, is not sufficient and the deformation structure is rather heterogeneous. Consequently, the nucleation and growth of recrystallized grains with $\{111\}\langle 112 \rangle$ orientation is inferred to take place. The TEM micrograph of the specimen cold-rolled by 80% followed by partial recrystallization annealing (50% fraction recrystallized), shown in Fig.12, supports the above speculation in the sense that the recrystallized grains with $\{111\}\langle 011 \rangle$ orientation are much larger than that with $\{111\}\langle 112 \rangle$ orientation.

5. Conclusion

The electro-deposited pure Fe has the fine columnar structure elongated in the thickness direction with isotropic sharp ND// $\langle 111 \rangle$ texture. It exhibits a surprisingly high r-value over 7 and extremely large local elongation. These properties are thought to be caused by the columnar structure with isotropic sharp ND// $\langle 111 \rangle$ texture, which leads to the large width strain as compared with the very small thickness strain because of the operation of $\{110\}$ slip systems perpendicular to the surface during tensile test. Recrystallization proceeds at 400 °C in the process of cold-rolling and subsequent annealing, when the electro-deposited pure Fe is used as a starting material. With increase in cold-rolling reduction, $\{111\}\langle 112 \rangle$ cold-rolling texture develops, while annealing texture changes from $\{111\}\langle 112 \rangle$ to $\{111\}\langle 011 \rangle$. The oriented nucleation and selective growth theory may explain the mechanism on these textures evolution. Thus, steels are considered to have potentials to further improve their properties by controlling microstructures and textures.

Acknowledgement

We are very grateful to Prof. A. Rollet, Carnegie Mellon University, for his valuable comments on the prediction of r-value.

References

- [1] Yoshinaga N, Sugiura N, Hiwatashi S, Ushioda K and Kada O 2008 *ISIJ Int.* **48** 667
- [2] Sugiura N and Yoshinaga N 2013 *J. Japan Inst. Metal Mater.* **77** 361
- [3] Sugiura N and Yoshinaga N 2014 *Advanced Materials Research* **922** 740
- [4] Yoshinaga N, Vanderschuren D, Kestens L, Ushioda K and Dilewijns J 1998 *ISIJ Int.* **38** 610
- [5] Van Houtte P 1988 *Texture and Microstructure* **8-9** 313
- [6] Daniel D and Jonas JJ 1990 *Metall. Trans. A* **21A** 331
- [7] Delannay L, Van Houtte P, Van Bael A and Vanderschueren D 2000 *Modelling and Simul. in Mater. Sci. Eng.* **8** 413
- [8] Rollet A 2014 Private communication
- [9] Siebel E and Beisswanger 1955 *Tiefziehen* (Carl Hanser Verlag)
- [10] Motegi T, Masuyama S, Iida K, Suzuki T, Sato H and Tomota Y 2002 *Tetsu-to-Hagane* **88** 801