Effects of Two-Step Cold Rolling on Recrystallization Behaviors in ODS Ferritic Steel

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1. Introduction

Oxide-dispersion-strengthened (ODS) ferritic steels are considered to be promising candidates for cladding tubes in advanced fast reactors due to their excellent radiation resistance and better creep strength.1–3) During the manufacturing process of ODS cladding tubes, cold rolling is a necessary step to acquire the final thickness. Nevertheless, the severe work hardening effect makes it impossible to directly get high reduction by one time cold rolling. Therefore, a process containing several cold rolling and intermediate annealing to soften the materials is usually adopted,4,5) during which the microstructure of the materials are severely affected and different types of texture are produced. Analyses of the changes of microstructure and texture during cold rolling and annealing are essential for optimizing the fabrication procedure in order to obtain the materials with high performances.

Till now, only a few works concerning recrystallization behaviors of cold rolled ODS ferritic steels have been reported.6–10) Furthermore, all these works contain only one time cold rolling, so the effects of multiple cold rolling and intermediate annealing are still unknown. Therefore, an experiment with two cold rolling and an intermediate annealing is reported here to investigate the effects of two-step cold rolling on recrystallization behaviors in cold rolled ODS ferritic steel.

2. Experimental Procedure

An ODS ferritic steel with the composition of Fe–15Cr–0.3%C–2W–0.3Y2O3 (mass%) was produced by mechanically alloying a mixture of metal and yttria powders using a high energy ball mill. The resultant powder was subsequently consolidated by hot extrusion and forging at 1150°C to make a 25 mm diameter bar. The steel bar was then annealed at 1150°C for 1 h. A plate in the size of 4 mm x 15 mm x 20 mm was cut from the center of the bar for cold rolling and the rolling direction was parallel to the extrusion direction. Figure 1 shows the processing routes of the ODS plate: after 1st 70% cold rolling, the plate was equally divided into two parts, A and B. Part A was cut into small specimens for the isochronal annealing experiment at different temperatures in air for 0.5 h to investigate the recrystallization behaviors during intermediate annealing. Part B was at first annealed at 1100°C for 0.5 h (intermediate annealing), after which the material should be fully recrystallized according to annealing experimental results of part A, then underwent 2nd cold rolling with 70% reduction. After that, part B was also cut into small specimens and isochronal annealed at different temperatures in air for 0.5 h for final annealing experiment.

A Shimadzu HVM-2 micro hardness tester was used to measure the hardness of as-cold-rolled and annealed specimens, where the load was 4.9 N and 5 points were measured for each specimen. A JOEL JSM-6500 FE-SEM with EBSD system was used to observe the microstructure and to analyze texture of specimens. Texture intensity was described by orientation distribution functions (ODF), which was calculated by the series expansion method using TSL OIM analysis software, and orientations were expressed in form of Euler angles (ψ1, Φ, ψ2) in ODF figures. All the hardness and texture analyses were taken at the surface of rolling plane (ND) of the specimens.

3. Results

3.1 1st cold rolling

Figure 2 shows the change of texture induced by 1st cold rolling, the numbers in ODF figure means the intensity of each contour line. For example in Fig. 2 ODF of hot-
extrusion, the number 7 in the left hand side line means that all the orientation inside this line have texture intensity 7 times stronger than random orientations, and “max 11.2” means the maximum texture intensity in that figure is 11.2 times random. Before 1st cold rolling, the specimen shows homogenous $[110] \parallel RD \alpha$ fibre texture, which is a typical hot extrusion texture. Whereas after 1st cold rolling, the former uniformly distributed $\alpha$ fibre texture converges; orientation with maximum intensity of 10 times random is between $[112] \parallel [110]$ and $[111] \parallel [110]$ while orientations close to $[110] \parallel [110]$ disappear. Besides, $[100] \parallel [110]$ has a intensity of 4 times random and a weak $\gamma$ fibre is also developed.

3.2 Intermediate annealing

In this experiment, recrystallization temperature of the specimen is defined as the lowest temperature required to make a 80% recrystallization in area fraction after annealing for 0.5h. Figure 3 shows the results of 0.5h isochronal annealing experiment of 70% cold rolled specimens. We can see that, after annealed at 950°C for 0.5h, area fraction of recrystallized grains is more than 80%, although deformed parts still exists [showed in black color in Fig. 3(b)]. Therefore, we consider the recrystallization temperature of intermediate annealing as 950°C. Besides, We can see from Fig. 3(a) that hardness of specimens annealed at temperature from 950 to 1100°C are very similar, which also indicates that recrystallization almost completed after annealing at 950°C for 0.5h. Further decrease of hardness after annealing at 1200 and 1300°C are ascribed to the coarsening of oxide particles, which will be discussed in another paper.

Figure 4 shows the texture of specimens after intermediate annealing at different temperature. We can see that the recrystallized specimens have a sharp $[111] \parallel [112]$ texture, which is a component of $\gamma$ fibre; increasing of annealing temperature do not change the texture. In order to make sure that the recrystallization is fully completed meanwhile avoiding particle coarsening, we choose 1100°C as the intermediate annealing temperature before 2nd cold rolling.

3.3 2nd cold rolling

To investigate the effect of multiple cold rolling, the specimen intermediately annealed at 1100°C for 0.5h was again 70% cold rolled. From Fig. 5 we can see that after 2nd cold rolling a partial $\alpha$ fibre especially $[100] \parallel [110]$ is developed, while $[111] \parallel [112]$ still remains at high intensity.

It is worth noting that, although the as-cold-rolled specimens by both 1st and 2nd cold rolling contains partial $\alpha$ fibre and $\gamma$ fibre, they are quite different; in specimen after 2nd cold rolling, $[100] \parallel [110]$ has the maximum intensity of 10 times random and $[111] \parallel [112]$ has highest intensity along $\gamma$ fibre. On the other hand, in specimen just after the 1st cold rolling (Fig. 2), $[100] \parallel [110]$ has a low intensity of only 4 times random and component of highest intensity along $\gamma$ fibre is $[111] \parallel [110]$.

3.4 Final annealing

After 2nd cold rolling, specimens were again isochronal annealed at different temperature for 0.5h to study the recrystallization behaviors during final annealing. Figures 3(a) and 3(c) show that the recrystallization tem-
perature at final annealing is 1100°C, which is 150°C higher than the recrystallization temperature at intermediate annealing.

The texture of specimens after final annealing is showed in Fig. 6. The specimen finally annealed at 950°C remains cold rolling texture similar as the one just after 2nd cold rolling, which demonstrates that recrystallization is retarded compared with intermediate annealing whose recrystallization temperature is 950°C. The specimen annealed at 1300°C exhibits fully recrystallization texture of γ fibre along which {111}(110) has maximum intensity; this is quite different from the sharp {111}(112) recrystallization texture by intermediate annealing.

4. Discussion

The results of experiments are summarized in Fig. 7. As the results show, the recrystallization temperature at final annealing is 150°C higher than that at intermediate annealing. This should be attributed to the different rolling texture induced by 1st and 2nd cold rolling. The driving force of recrystallization is the stored strain energy (Ed)
in deformed grains accumulated during cold rolling, which depends on the orientations of deformed grains. Former researches\textsuperscript{11,12} show $E_{\{111\}} > E_{\{112\}} > E_{\{001\}}$ for cold rolled ferritic steels, thus $\{110\}$ deformed grains have low stored energy and are difficult to recrystallize.\textsuperscript{13} The intensity of $\{100\}(110)$ in 2nd and 1st cold rolled specimen is 10 times random and 4 times random respectively. Such kind of high $\{100\}(110)$ content makes the specimen after 2nd cold rolling difficult to recrystallize during the following final annealing.
Despite of the possible difference in rolling texture between single crystal and polycrystalline steels, the systematic data from single crystal experiment is a valuable reference to get the basic understanding of rolling texture formation. A comprehensive chart for the typical crystalline rotation during cold rolling in single crystal 3% silicon steels, as shown in Fig. 8, is used to interpret how the different cold rolling texture forms by 1st and 2nd cold rolling. During 1st cold rolling, orientations change according to series III in which crystalline rotation takes place around (011) common axis. Grains with orientations near {110}{110} rotate to {111}{110} and towards {112}{110}, which is most stable. Because of this kind of rotation trend, the uniformly distributed α fibre texture made by hot extrusion converges; orientations close to {110}{110} disappear and maximum appears between {112}{110} and {111}{110}.

During 2nd cold rolling, the sharp {111}{112} texture induced by intermediate annealing is a stable orientation, thus a certain amount of deformed grains remains {111}{112} orientation. Meanwhile, other texture are produced by rotation from {111}{112} to {112}{111} and finally to {100}{110} which is another stable orientation according to series II. The above mechanism causes the different {100}{110} contents by 1st and 2nd cold rolling, which consequently leads to a difference in recrystallization temperature between intermediate and final annealing.

Besides, the different cold rolling texture before intermediate and final annealing also induce different recrystallization texture. During intermediate annealing, the initial rolling texture of weak γ fibre and partial α fibre with high {111}{110} intensity produce a sharp {111}{112} recrystallization texture, whereas during final annealing, initial texture of high {100}{110} and {111}{112} content leads to a recrystallization texture of γ fibre with maximum at {111}{110}. From Fig. 9, we can see that, the orientation relationship between {111}{112} and {111}{110} is a 30° rotation around (111) axis. According to Hutchinson, however, growth selection would not occur because such orientation relationship doesn’t correspond to special high mobility boundaries in bcc metals. Thus, preferential nucleation of {111} grains is considered to be responsible for the development of γ fibre recrystallization texture.

Although it is generally accepted that recrystallized {111} grains nucleate in deformed {111} grains and grow at the expense of deformed {111} grains, argument still exist. Some claim that {111}{110} nuclei form in the most high strained {111}{110} regions and {111}{112} new grains recrystallize from {111}{112} deformed matrix, while others consider that the recrystallized {111}{110} are formed in deformed {111}{112} regions and {111}{112} new grains recrystallize from {111}{110} deformed matrix. Our experiment shows that when the initial cold rolling texture has high {111}{110} intensity, the recrystallization texture will have high {111}{112} intensity (in the case of intermediate annealing). On contrary, when the initial texture consists of high intensity {111}{112} component, the recrystallization texture will have high intensity at {111}{110} (in the case of final annealing). These results indicate that, during recrystallization of our Fe–15Cr ODS ferritic steel, {111}{112} recrystallized grains nucleate in {111}{110} deformed grains and {111}{110} recrystallized grains nucleate in {111}{112} deformed grains.

5. Conclusions

We investigated the effects of two step cold rolling on recrystallization behaviors of 15Cr-ODS ferritic steel, and the following results were obtained.

(1) Specimen undergo 1st cold rolling contains weak {100}{110} and strong {111}{110} texture, while specimen undergo 2nd cold rolling after intermediate recrystallization contains strong {100}{110} and strong {111}{112} texture, which is ascribe to the different routes of crystalline rotation during these two cold rolling.

(2) The recrystallization temperature of intermediate annealing and final annealing are 950 and 1100°C respectively. A retardation of recrystallization is due to the increasing of {100}{110} texture, which has low stored energy and is difficult to recrystallize.

(3) The recrystallization texture of intermediate annealing and final annealing are both γ fibre, but the texture component with highest intensity is {111}{112} and {111}{110} respectively. The difference is due to different initial cold rolling texture induced by 1st and 2nd cold rolling.
REFERENCES