The Recrystallization Behavior of Zirconium by Addition of High Nb,Fe

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1. Introduction

The development of improved zirconium alloys has been extensively carried out in many countries for the last few decades [1]. They have reported that the corrosion resistance of zirconium alloys was determined by the alloying elements as well as manufacturing processes. Among the various elements, Nb and Fe are chosen in the advanced claddings, since the corrosion resistance and mechanical properties in the irradiation environment could be improved by the addition of these two elements.

However, the corrosion behavior and mechanical property is very sensitive by the addition of high Nb and Fe. So, it is necessary for the study on microstructural changes with heat-treatment of high Nb, Fe containing Zr alloy to improve the corrosion and mechanical properties [2]. Especially, recrystallization behavior is very important factor to control the precipitate characteristics and fraction of recrystallization. Because the corrosion is controlled by the precipitates type and the mechanical properties are determined by the fraction of recrystallization. Therefore, this study is focused on the effect of high Nb and Fe addition of zirconium alloy on recrystallization behavior.

2. Methods and Results

Table 1 shows the chemical composition of experimental alloys. The Nb content ranged from 1.0 to 2.0 wt% and the Fe content ranged from 0.2 to 0.4 wt% in the zirconium based alloys.

Table.1: Chemical composition of zirconium based alloys

Composition
Zr-xNb (x = 1.0, 1.5, 2.0)
Zr-2.0Nb-xFe (x = 0.2, 0.4)

The experimental alloys with various Nb and Fe contents were manufactured as following the general manufacturing process as shown in Table 2. The alloys were melted in a vacuum environment (three times), water quenched after β-solution treatment at 1020°C for 15 min, hot rolled after pre-heating at 640°C for 15 min, annealed at 570°C for 3h and then cold rolled of 80%.

The specimens to study recrystallization behavaior were performed on annealing at 400°C - 600°C for 1 and 3 h after 80% cold-worked alloy.

The recrystallization behavior for the annealed Zr-base alloys is evaluated by using the polarized light microscopy, microvickers hardness tester, and transmission electron microscope (TEM).

Table.2: Manufacturing process of zirconium based alloys

Process flow
Melting: Vacuum arc (3 times)
↓
Beta quenching: Into water (1020°C x 15min preheat)
↓
Hot rolling: 60% (640°C x 15min preheat)
↓
Annealing: 570°C x 3h (vacuum)
↓
Cold rolling: 80%
↓
Annealing temperature: 400, 450, 475, 500, 550, 600 Annealing time: 1, 3h

2.1 Microhadness test

The microhardness test was performed by using a Knoop-type indenter (Shimadzu, Model HMV-2) to reduce the partial deviation due to microstructural differences, such as grain boundary and precipitates. The hardness value for one sample is averaged from the ten time tests, and applied load is 0.2 N for 10 sec.

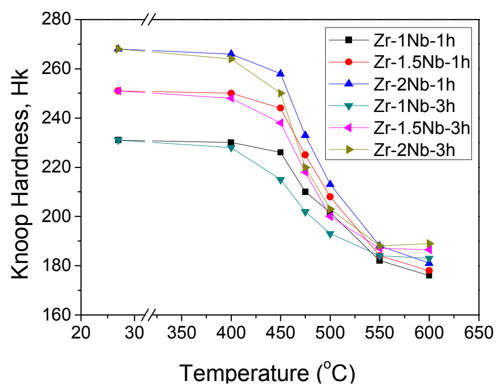

Fig.1 Variation of Knoop hardness the Zr-xNb alloys with annealing time of 1 and 3h.

Fig.1 shows hardness variation with the Nb contents and annealing temperature for 1 and 3 h. The hardness value of Nb-containing Zr alloys is decreased with increasing annealing temperature. And the significant drop of hardness in Nb-containing alloys was shown at the temperature range from 450 to 550℃. At the RT condition, the hardness is increase with addition of Nb content from 1.0 to 2.0 wt%. The increased hardness at initial point is caused by the precipitate hardening in Zr-Nb alloy system because the Nb content in this work is higher than the Nb-solubility in Zr alloy [3]. From the hardness variation in Fig.1 it is known that the high hardness value at initial point is not affected on the recrystallization behavior of Zr-xNb alloys (x= 1.0 to 2.0 wt%). However, the recrystallization temperature is decreased by increasing the annealing time from 1 to 3 h. Fig.2 shows hardness variation with the Fe contents and annealing temperature for 1 and 3 h in Zr-2.0Nb-xFe alloy. The hardness behavior of Fe-containing Zr-based ternary alloys is similar to the Nb-containing Zr binary alloys.

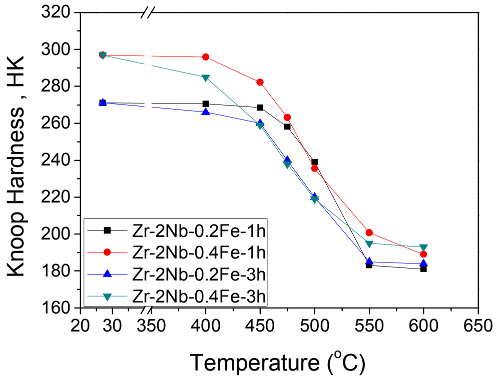

Fig. 2 Variation of Knoop hardness the Zr-Nb-xFe alloys with annealing time of 1 and 3h.

2-2 TEM observation

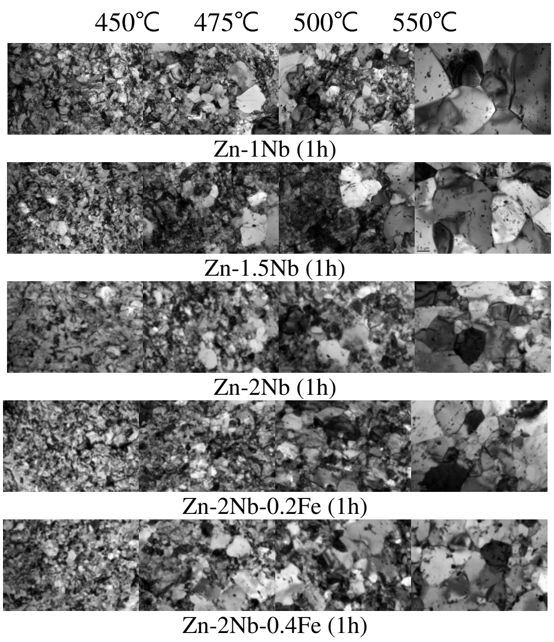

Fig. 3 TEM micrographs of the Zr-xNb, Zr-2Nb-yFe alloys after annealing ranged from 450 - 550°C for 1h.

Fig. 3 shows TEM micrographs of the Z-xNb, Zr-2Nb-xFe alloys after annealing ranged from 450 to 550℃ for 1h. A cold worked microstructure was observed in all alloy compositions after 450℃ annealing for 1h, and the fraction of partial recrystallized grain was increased with increasing annealing temperature up to 500℃, and then fully recrystallized microstructure was observed after 550℃ annealing for 1h. From this result, the recrystalliczation is started from the annealing temperature of 450℃ and is finished at 550℃ annealing. After the recrystallization, the grain size is decreased with increasing Nb and Fe contents [4]. This is caused by the increase of precipitate fraction with increasing Nb and Fe contents of Zr-xNb and Zr-2Nb-xFe alloys. The precipitate is act as a barrier to increase the grain size during annealing.

3. Conclusions

The significant drop of hardness in Nb-containing alloys was shown at the temperature range from 450 to 550℃. The increased hardness at initial point is caused by the precipitate hardening in Zr-Nb alloy system because the Nb content in this work is higher than the Nb-solubility in Zr alloy. The hardness behavior of Fe-containing Zr-based ternary alloys is similar to the Nb-containing Zr binary alloys. After the recrystallization, the grain size is decreased with increasing Nb and Fe contents.

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