

Texture Variation by Annealing at 550°C in Zr-2.5%Nb Pressure Tube Material

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1. Introduction

Zr-2.5%Nb alloy, which is excellent in high-temperature strength, corrosion resistance, and neutron absorption cross-sectional area, is used for pressure tube material in pressurized heavy water reactor (PHWR). Since the primary water flows through the pressure tube during the operation of PHWR, some of the hydrogen produced by corrosion process with Zr is absorbed into the pressure tube [1].

The pressure tube materials containing hydrogen precipitate hydrides, when the hydrogen concentration exceeds the solubility of hydrogen during cooling process of pressure tube. The precipitation of hydride lowers the fracture toughness of the pressure tube materials. In addition, hydrogen in the pressure tube material tends to be precipitated at the stress concentration site at the crack tip and causes crack initiation and propagation by the delayed hydride cracking mechanism [1].

It is known that Zr alloys shows a change in texture during recrystallization heat treatment at below $\alpha+\beta$ temperature [2]. The change of texture during recrystallization treatment after cold work in Zr alloy was studied. However, there is a limitation to trace the texture variation systematically after recrystallization it was not possible to analyze successfully [3].

Meanwhile, the pressure tube material was so well developed that it was possible to systematically analyze a change in the texture during recrystallization. In this study, the reason for change of texture was analyzed using recrystallized specimen at 550 °C for 240 hours, and the mechanism causing the change of texture was identified.

2. Experimental

The pressure tube material used in the experiment is Zr-2.5% Nb alloy. The chemical composition is shown in Table 1. After the Zr-2.5% Nb alloy pressure tube was heat-treated at 500 °C up to 3,000H and at 550 °C for 240H, the texture variation was analyzed using XRD and EBSD (electron back scattered diffraction). The specimen was annealed in a vacuum sealed tubing.

Table 1. Chemical composition of Zr-2.5%Nb (wt%).

Zr	Nb	Fe	O	N	P
97.4	2.5	0.05	0.097	0.007	0.003

Texture variation with the strain before and after a recrystallization is examined by the inverse pole figure measurement using a Cu-K α X-ray and EBSD. The XRD specimen surface is prepared by swabbing with 8%HF+45%H₂O solution. The surface preparation of EBSD specimen is electro-chemically polished by 10% per chloric acid and methyl alcohol solution.

3. Results and Discussions

Figure 1 shows the comparison of the inverse pole figure figure with XRD method before and after annealing after 550°C-240H heat treatment. This inverse pole figure is measured in the plane perpendicular to the longitudinal direction of a pressure pipe. Before the heat treatment, the (10 $\bar{1}$ 0) plane is placed vertically in the longitudinal direction of the pressure tube, whereas after the heat treatment, the (21 $\bar{3}$ 1) plane is mainly placed in the longitudinal direction of the pressure tube. Since the crystals were grown tens of times in the heat treatment process, it can be seen that twin action occurred in recrystallization. The peculiarity of twinning is that the crystal placed in a certain position disappears and the crystal is concentrated in another specific position.

Along with slip, twins are an important deformation mechanism and are known to act when deforming in a non-slip direction in Zr alloys. In the present study, deformation did not occur but only heat treatment. This is an unexpected result. It has been known that changes in texture occur during cold working and/or recrystallization heat treatment of Zr alloys, but the cause is not well explained yet.

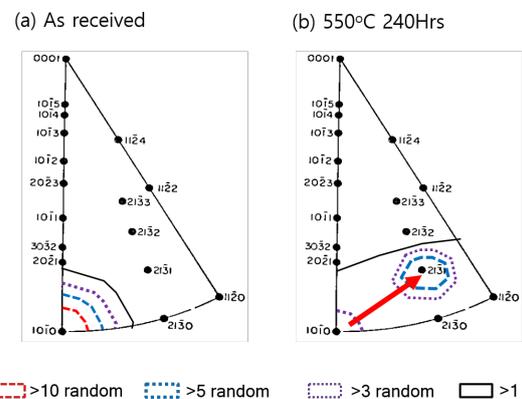


Fig. 1. Texture variation by annealing at 550°C for 240H treatment in Zr-2.5%Nb pressure tube material.

Figure 2 shows the comparison of the inverse pole figure by EBSD analysis at the radial, transverse, longitudinal normal planes after 550°C-240H. Inverse pole figure examined by EBSD in longitudinal normal plane (Fig. 2 (c)) is very consistent with that analyzed by the XRD method (Fig. 1 (b)). In other words, the analysis results by XRD and EBSD are similar. It can also be seen that grains are concentrated around (10 $\bar{1}$ 0) before annealing and (21 $\bar{3}$ 1) after annealing in the inverse pole figure.

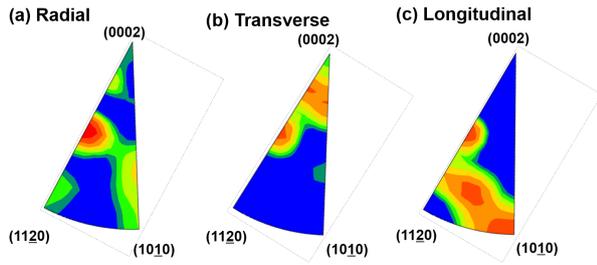


Fig. 2. Inverse pole figure after annealing 550°C for 240Hours treatment in in Zr-2.5%Nb pressure tube material

Figure 3 schematically shows the twin deformation mechanism that occurs when tensile and tensile stresses are applied to the crystal direction in a Zr alloy of HCP crystal structure [6]. It has been explained that when tension is applied in a direction close to the c axis of the HCP crystal, tensile twin deformation occurs, and tension is applied in a direction slightly away from the c axis, but tension twin deformation occurs.

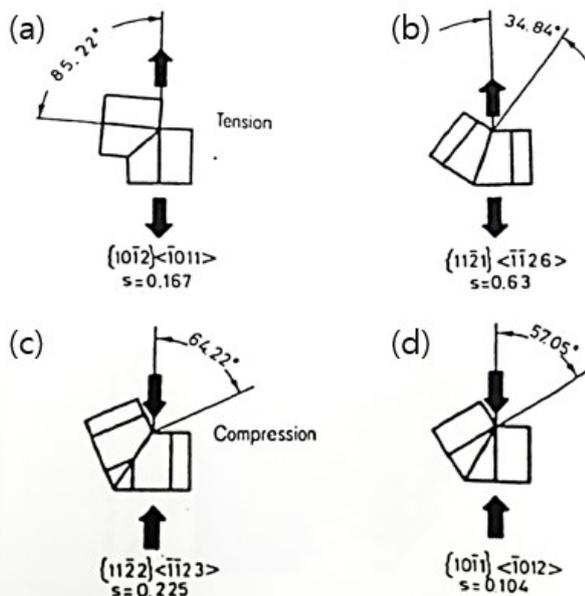


Fig. 3. Twinning modes in α -Zirconium [6].

Figure 4 illustrates the process of identifying the rotation angle of (10 $\bar{1}$ 0) pole before and after

recrystallization treatment using the inverse pole figure and the Wulff net. This figure illustrates how the {11 $\bar{2}$ 1} $\langle \bar{1}\bar{1}26 \rangle$ twin operated, this system is tension twin. This diagram looks a bit complicated, but the operation of {11 $\bar{2}$ 1} $\langle \bar{1}\bar{1}26 \rangle$ twin is well explained. It can be seen that the twin shear occurs along the direction of [11 $\bar{2}$ 0] direction because the twinning direction depends on the specific twin plane and twin direction.

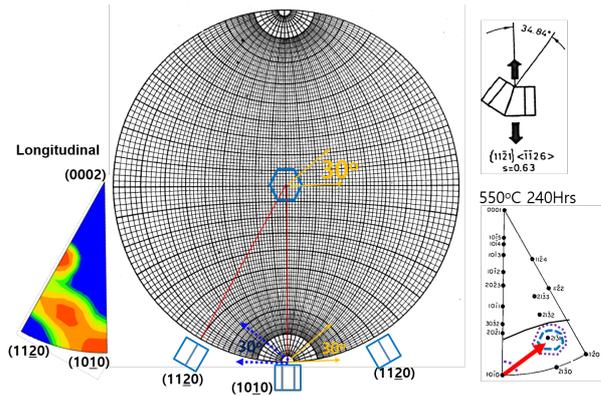


Fig. 4. Schematic explanation of operation of {11 $\bar{2}$ 1} $\langle \bar{1}\bar{1}26 \rangle$ twinning system during annealing at 550°C for 240Hours in Zr-2.5%Nb pressure tube material.

Figure 5 shows how the Schmid factor for each deformation mechanism changes with HCP crystal and the direction of stress. It can be seen that the Schmid factor for {11 $\bar{2}$ 1} $\langle \bar{1}\bar{1}26 \rangle$ twin is the largest in the direction of the c-axis and the tensile stress at about 65°. This means that the c-axis and tensile stress were about 65° during the recrystallization of the pressure tube material

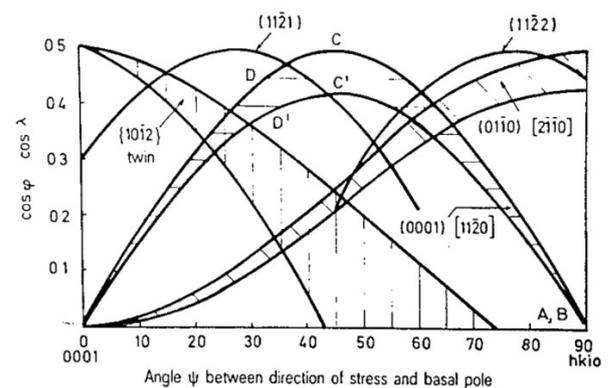


Fig. 5. Orientation dependence of Schmid factor for various slip and twinning systems in Zirconium [6].

4. Conclusions

1. During the heat treatment of Zr-2.5% Nb pressure tube material, recrystallization causes at 550°C for 240H change in texture significantly.
2. The analysis of reason for the texture variation before

and after recrystallization using Wulff net and inverse pole figure showed that showed the operation of tensile $\{11\bar{2}1\} \langle 1126 \rangle$ twin.

3. Texture variation examined by both XRD and EBSD method provided a consistent result.

Acknowledgments

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REFERENCES

- [1] G.J. Field, J. T. Dunn, B. A. Cheadle, AECL-8335, 1984.
- [2] F. Gerspach, N. Bozzlolo, and F. Wagner, Scripta Materialia 60 (2009) 203.
- [3] M. H. Li, M. Ma, W. C. Liu, F. Q. Yang, Journal of Nuclear Materials 433 (2013), pp. 6.
- [4] W. M. Evans, R. F. Gessner, and J. G. Goodwin: Metal. Trans., 1972, 3, 2879.
- [5] J. E. Winegar: Measurement of Crystallographic Texture at Chalk River Nuclear Laboratories, Atomic Energy of Canada Limited Report, AECL-5626 (1977).
- [6] E. Tenckhoff: ASTM special technical publication (STP) 966, 1988, 3.