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RELATIONSHIP BETWEEN ANISOTROPY AND CROSS ROLLING PROCESS FOR HIGH PURITY NIOBIUM SHEETS

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Abstract

The anisotropies of niobium sheet with three cross rolling conditions were compared. When the reduction ratios before and after changing the rolling direction were equalized, it is indicating the smallest anisotropy. Moreover, half cells for superconducting cavities were press-formed, and their roundness at the equator part was measured. The difference in anisotropy among the three types of sheets did not significantly affect the press-forming of these half-cells.

INTORODUCTION

The common manufacturing method of superconducting cavities with an elliptical cell shape involves press-forming high-purity niobium sheets into hemispherical half-cells and then joining them together with electron-beam welding. The large-diameter part (referred to as the equatorial part), which serves as the mating surface between the half-cells, requires good roundness. Additionally, for successful press-forming, the niobium sheets must have low anisotropy. Tokyo Denkai produced and supplied 7,800 high-purity niobium sheets for the 1.3 GHz TESLA cavity used in the European-XFEL [1]. Niobium sheets are manufactured by forging and rolling niobium ingots. Cross rolling [2] is employed to reduce anisotropy. While several studies have been conducted on niobium rolling [3-5], examples investigating the influence of cross-rolling parameters on anisotropy are scarce. Therefore, in this study, three types of niobium sheets with varied cross-rolling parameters were prepared to evaluate the differences in anisotropy. Furthermore, the press-forming of the half-cells was conducted to compare their circularities.

EXPERIMENTAL METHOD

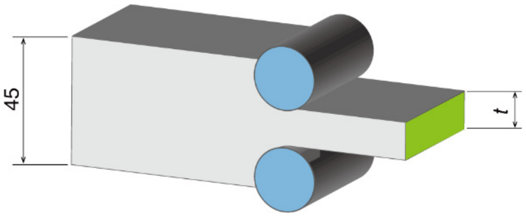
Cross-rolling Method

First, a niobium ingot with a diameter of 245 mm was forged to produce slabs of dimensions 200 × L × t50 mm (where L is approximately 200). After forging, the entire surface was milled to remove the oxidation layers and embedded foreign metals, reducing the thickness to 45 mm. Subsequently, rolling was performed using this material as the starting material. The reduction in sheet thickness owing to rolling is referred to as the rolling reduction ratio, and it can be calculated as follows [2].

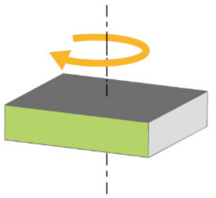
$$\frac{h1 - h2}{h1} \tag{1}$$

Here, $h1$ and $h2$ represent the thicknesses of the sheet before and after rolling, respectively. Rolling from 45 mm to 2.8 mm resulted in a reduction ratio of 94%. As the reduction in thickness per pass was approximately 0.1 mm, over 420 passes were required. During these passes, a method called cross rolling was employed. There are two types of cross-rolling techniques: multistep cross rolling, where the rolling direction changes by 90° after each pass, and two-step cross rolling (TSCR), where the rolling direction changes only once by 90° when the reduction ratio exceeds 50% [2]. Tokyo Denkai, based on empirical evidence of good workability and high yield, adopts the TSCR method as shown in Fig. 1. With this billet, six niobium sheets can be produced.

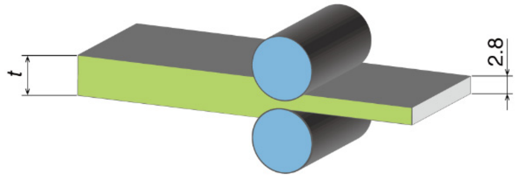
In this study, three different cross-rolling methods with varying thicknesses t when changing the rolling direction, as shown in Table 1, are compared. Method A represents the conditions used until around 2000. Method B represents the current production conditions. Method A has been modified to reduce anisotropy by decreasing t, which effectively increases the reduction ratio of the first rolling pass. Method C represents a new condition where t is further reduced, and the reduction ratios of the first and second rolling passes are equalized.



(i) First step rolling



(ii) Change of rolling direction (90 deg. rotation)



(iii) Second step rolling

Figure 1: Process of cross rolling

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Table 1: Rolling conditions

	A	B	C
Initial thickness [mm]		45	
Reduction ratio [%] $(45 - t) / 45$	36	62	76
Intermediate thickness t [mm]	29	17	11
Reduction ratio [%] $(t - 2.8) / 2.8$	90	84	75
Final thickness [mm]		2.8	

Preparation of Test Specimens

Table 2 presents the chemical composition and RRR values of the niobium ingots. The three types of sheets were manufactured using slabs obtained from the same ingot. From the finished sheets, tensile test specimens were cut using a wire electric discharge machine. The shape of the tensile test specimens conforms to ASTM E8 Subsize specimen (width 6 mm, gauge length 25 mm) standards [6]. These specimens were cut at the angles of 0°, 45°, and 90° with respect to the final rolling direction of the niobium sheet. Subsequently, these specimens underwent vacuum annealing at 800 °C for 3 h after cutting.

The Lankford value, r, is measured to evaluate the anisotropy of the sheets. r represents how much easier the width direction deforms compared with the thickness direction when uniaxial tensile strain is applied to a metal sheet. The specimens are stretched until the strain reaches 20%, and r is calculated from the strain in the width direction at that time [7]. r is determined from specimens taken at the angles of 0°, 45°, and 90° with respect to the rolling direction, denoted as r_0, r_{45}, and r_{90}, respectively. The average plastic strain ratio $\bar{r}$ is expressed as follows, representing the difficulty of deformation in the thickness direction. A higher $\bar{r}$ indicates better press-formability, allowing for deeper drawing.

$$\bar{r} = \frac{r_0 + 2r_{45} + r_{90}}{4} \tag{2}$$

The planar anisotropy Δr is expressed as follows. A value closer to 0 indicates less anisotropy.

$$\Delta r = \frac{r_0 - 2r_{45} + r_{90}}{2} \tag{3}$$

Press-forming Half Cells

Circular discs with a diameter of 260 mm were cut from the niobium sheets using a wire electric discharge machine, followed by vacuum annealing at 800 °C for 3 h. The press-forming of the half-cells was conducted using these circular discs. The roundness of the equatorial part of the completed half-cells was measured using a three-dimensional measuring machine. The required value for roundness is 0.4 mm.

RESULTS AND DISCUSSION

Specimen Observation

Specimens were cut from the three types of sheets and subjected to vacuum heat treatment at 800 °C for 3 h before observing the crystal orientation distribution using electron backscattering diffraction (EBSD). The observation results are shown in Fig. 2. The RD plane represents the center of the sheet thickness, where recrystallization occurs owing to heat treatment, and nearly circular grains are observed. The average grain sizes calculated using EBSD are shown in Table 3. Although C has the smallest value, the difference is minor. There is no significant difference between the TD and RD planes. When observing the ND plane, both B and C show a prevalence of (111) planes. Yamaguchi *et al.* demonstrated that rolled niobium materials exhibit a predominance of (100) and (111) planes, which transform into (111) planes after recrystallization [8], consistent with similar results obtained here. Overall, no significant differences in crystal distribution were observed owing to variations in the rolling conditions.

Tensile Testing

Tensile tests were conducted using the Shimadzu AG-50kN-X tensile testing machine. The testing speed was set at 1.5 mm/min. The results of the tensile tests are presented in Table 3 and Fig. 3. In all three types of sheets, specimens cut at an angle of 45° with respect to the final rolling direction exhibited higher tensile strength, whereas those cut at 0° and 90° showed nearly equal strength. Additionally, specimen type C demonstrated the smallest difference in strength between the angles of 45° and 0°.

Assessment of Anisotropy

The results of the Lankford value measurements are presented in Table 3 and Fig. 4. Similar to the tensile test results, specimens cut at an angle of 45° with respect to the final rolling direction exhibited higher r values for all three types. Additionally, similar to the tensile strength results, type C showed the smallest difference in r values between the angles of 45° and 0°. The calculated results for $\bar{r}$ and Δr are shown in Fig. 5. It is evident that $\bar{r}$ for type C is the largest, indicating good deep-drawing characteristics. Furthermore, Δr for type C is closest to 0, indicating minimal anisotropy. Based on these findings, it can be concluded that type C sheet is the most suitable for press-forming.

Table 2: Chemical compositions and RRR of starting niobium ingot

C	H	N	O	Ta	W	Ti	Fe	Mo	Ni	RRR
<10	<2	<10	<10	14	<10	<10	<10	<10	<10	392

Unit of chemical compositions: wt. ppm

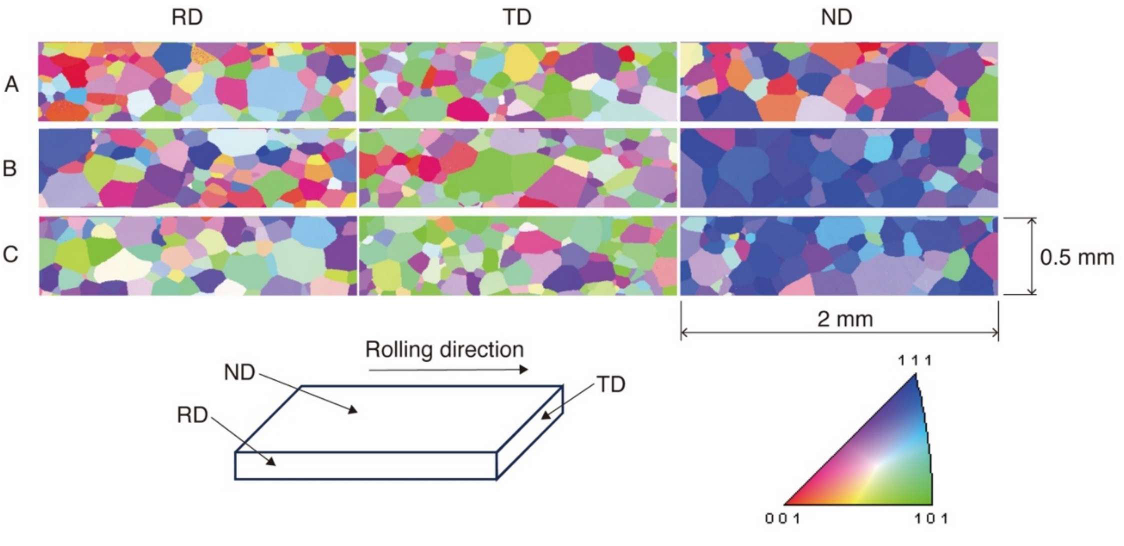

Figure 2: IPF maps of cross rolled specimens

Table 3: Result of measured mechanical properties

	Tensile strength [MPa]			0.2% proof strength [MPa]			Elongation [%]	Hardness [HV1]	Grain size [μm]	Lankford coefficient			$\bar{r}$	Δr	Roundness mm
	0°	45°	90°	0°	45°	90°				0°	45°	90°			
A	156	174	158	48	52	50	60	45	42	1.22	2.06	0.34	1.42	-1.28	0.1
B	149	164	153	50	50	49	54	45	48	0.86	1.32	0.09	0.90	-0.84	0.1
C	158	168	158	50	51	50	53	44	33	1.46	1.85	1.31	1.62	-0.46	0.1

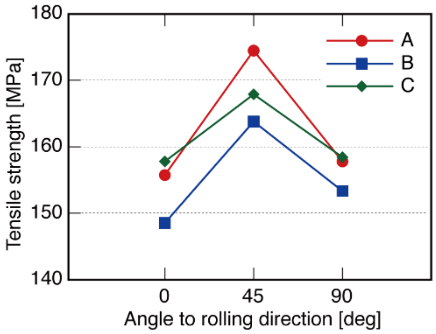

Figure 3: Relationship between tensile strength and angle to rolling direction

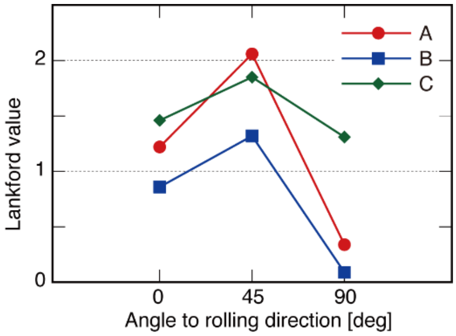

Figure 4: Relationship between Lankford value and angle to rolling direction

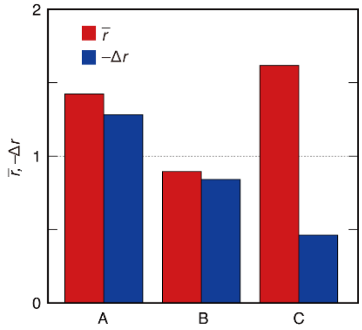

Figure 5: Average Lankford value $\bar{r}$ and planar anisotropy Δr

Comparison of Press-formability

The results of roundness measurements are shown in Table 3. All three types satisfy the required value of 0.4 mm. Although it was expected that the roundness of type C, which has minimal anisotropy, would be smaller, there was no difference observed among the three types. The amount of press-forming for the half-cells is approximately 50 mm and not substantial. It is presumed that the difference in anisotropy among the three types of sheets did not significantly affect the press-forming of these half-cells.

CONCLUSION

Three types of niobium sheets subjected to cross rolling under different conditions were prepared, and their anisotropies were compared. When the reduction ratios before and after changing the rolling direction were equalized, the average plastic strain ratio $\bar{r}$ was the highest, and the in-plane anisotropy Δr was closest to 0, indicating the smallest anisotropy.

Using the three types of niobium sheets with different anisotropies, half-cells for superconducting cavities were press-formed, and the roundness of the equatorial part was measured. The circularities of all three types were 0.1 mm, and no differences were observed. The required value of 0.4 mm was satisfied. The difference in anisotropy among the three types of sheets did not significantly affect the press-forming of these half-cells.

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