A systematic study has been carried out on Nb-Ti wire of two different kinds of metallurgical structure, to examine the effects of neutron irradiation on the critical density \( J_c \). The samples used were Nb-47.6\% Ti (sample #A) and Nb-59.8\% Ti (sample #B), which were aged at 380°C for 0~104 min and irradiated to \( 1.3 \times 10^{18} \text{n/cm}^2 (E_{\text{n}} > 0.1 \text{MeV}) \). The sample temperature during the irradiation is believed to have been below 70°C. The values of \( J_c \) of both #A and #B aged up to 50 min were found to increase with irradiation. But when aged beyond 100 min, #B had its value of \( J_c \) lowered by the irradiation. The presence of Ti enriched precipitates such as \( \alpha \) and \( \omega \) phases in the samples was surmised from the behavior shown by the critical temperature \( T_c \). The \( T_c \) of #A and #B changed little by irradiation when aged not longer than 100 min, but with aging beyond 500 min, #B showed a decrease in its value of \( T_c \). This decrease indicates that the Ti concentration in the matrix may have increased through radiation-induced breakup of the above-mentioned precipitates, which, in turn, would have brought about the reduction observed in \( J_c \) upon irradiation. It is concluded that superconducting Nb-Ti wire with \( J_c \) enhanced by precipitation does not appear very resistant to neutron irradiation. This underlines the importance of the choice of superconducting materials to be used in fusion reactor magnets.

**KEYWORDS:** niobium-titanium alloys, aging, precipitates, neutron irradiation, critical current density, critical temperature, radiation effect, fusion reactor magnet

**I. INTRODUCTION**

With successful approaches being made toward plasma containment, ever rising expectations are held for the fusion reactor as a future source of energy. Several plans for constructing full-scale fusion reactors have been proposed in recent years, as a practical proposition, from the viewpoint of reactor technology\(^{(1)}\)\(^{-}(5)\). In these plans, a superconducting magnet is proposed for generating the magnetic field required for containing the plasma. There is little doubt that a fusion reactor operating with a steady magnetic field will require superconducting windings, since the power losses in conventional coils would be prohibitively high\(^{(6)}\)\(^{(7)}\). Application of superconducting wires to a fusion reactor magnet involves the following two problems.

(1) High magnetic field and large stored energy
(2) Neutron irradiation effects

In the former area, the difficulty arises from the instability of a “wound” superconductor. The current capacity of a “wound” superconductor is normally lower than that of a “short” superconductor. This problem is outside the scope of this study. In the latter area, radiation effects on superconductors present two aspects.

(1) Flux effect
(2) Fluence effect

The “flux effect” relates to nuclear heating, and appropriate coolant channels must be arranged...
in a large magnet. The "fluence effect" takes the form of changes brought to the superconducting properties of the substance by the cumulative effect of radiation damage.

In this paper we describe an experimental study on the "fluence effect" in some superconductors. Among the properties that determine the superconducting behavior, such as critical temperature $T_c$, critical current density $J_c$ and critical magnetic field $H_{c2}$, the properties most sensitive to neutron irradiation is considered to be $J_c$. In pure and well-annealed superconducting materials, $J_c$ is normally increased by neutron irradiation. A number of authors\(^{(8)-(12)}\) dealing with subject, however, have reported that $J_c$ did not always increase in alloys and compounds. Sugisaki et al.\(^{(11)}\) have irradiated Nb-Zr and Nb-Ti alloys to a neutron fluence of $3.5 \times 10^{18}$ n/cm\(^2\) ($E_n > 0.1$ MeV) at reactor temperature, and observed that $J_c$ increased in the case of Nb-Ti but decreased in Nb-Zr. Cullen et al.\(^{(12)}\) have irradiated Nb$_3$Sn compounds to a fluence of up to $1.4 \times 10^{18}$ n/cm\(^2\) at approximately 50°C. The notable effect observed by Cullen et al. was that the change of $J_c$ due to irradiation depended largely on the initial value of $J_c$: With relatively low initial $J_c$, the value increased with irradiation, whereas it decreased when the initial $J_c$ was high.

In the present work we have aimed at determining the cause of the degradation observed in $J_c$ with irradiation, and to provide some guiding principles in the choice of superconducting materials to be used in fusion reactor magnets for practical applications. The samples used in our experiments are Nb-Ti superconducting alloys, which have often been used in past experiments on materials intended for use in large magnets of future fusion reactors. We have made a systematic study covering two kinds of Nb-Ti alloys, with particular attention directed to finding the differences in the effect of irradiation arising from differences in metallurgical structure of the samples, which was varied by changing the duration of aging.

## II. EXPERIMENTAL

### 1. Samples

In Table 1 are listed the chemical compositions of the two kinds of samples used in this work. These samples were prepared as follows. One series was a Nb-47.6 % Ti alloy (sample "#A"): The ingot was cast in Ar atmosphere and worked to 50 mm diameter in order to destroy its columnar structure. The material was then subjected to solution treatment 1,000°C for 4 hr in a vacuum of $\sim 10^{-6}$ Torr. The 50 mm rod was cold-worked to 2.5 mm and after cladding with OFC (oxygen free copper) possessing a residual resistivity ratio of about 200, it was drawn to 0.25 mm core and 0.35 mm outside diameter (ratio of reduction in cross-sectional area 99.998%).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Elements (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ti</td>
</tr>
<tr>
<td>#A</td>
<td>31.9</td>
</tr>
<tr>
<td>#B</td>
<td>44.7</td>
</tr>
</tbody>
</table>

The other series was Nb-59.8 % Ti (sample "#B"), for which an arc molten ingot was cast in Ar and worked down to 7 mm diameter. After this, as solution treatment, it was heated at 930°C for 2 hr in $\sim 10^{-6}$ Torr vacuum. The 7 mm bar was then cold-worked to 2.5 mm diameter, and after OFC cladding, it was drawn to 0.25 mm core and 0.35 mm outside diameter (area reduction ratio 99.87%).

Both #A and #B were aged at 380°C, for various periods from 0 to 10\(^4\) min, in $\sim 10^{-6}$ Torr vacuum.

### 2. Critical Current Measurements

The standard four-terminal method was used to determine the current-voltage characteristics of the samples. To obtain low-resistance current contact, the lead wires were soldered to the central part of the sample, separated by a distance of 10 mm. The sample was held on a bakelite plate, which was mounted in a superconducting magnet used as external source of magnetic field for the sample. The magnet system used has previously been described in detail\(^{(11)}\). The current passing through the sample under constant transverse magnetic field was increased at a rate of about 2A/sec, until a predetermined voltage was induced across the sample. The values of
the induced voltage and the transport current were recorded on an XY recorder (YEW 3077), using a microvoltmeter (Toa PM-17A) and a standard resistor (YEW 220). The critical current \( I_c \) was determined from the voltage drop of 5 \( \mu \)V across the sample, and the critical current density \( J_c \) was obtained by dividing the value of \( I_c \) by the cross-sectional area of the sample core.

3. Critical Temperature Measurements

The five samples and the calibrated Ge temperature sensor (Cryocal Inc. CR-500) were each mounted on one side of a right hexagonal cylinder made of Al to ensure measurement under identical temperature conditions. The assembly was then encased in a thick-walled Cu can. The temperature of the sample was controlled by precise adjustment of the Cu can above the liquid He surface. To avoid self-heating, the Ge sensor was used under a current smaller than 20\( \mu \)A across the piece. The voltage induced in the sensor was measured by a digital voltmeter (YEW 2501), and recorded on the X axis of the XY recorder through a D-A converter (National VP-495A). The resistance of the sample was measured by the standard four-terminal method. The current passed through the sample was 20 mA, and potential drop was recorded on the Y axis of the XY recorder. In this manner, a super-normal transition curve was obtained. No thermal hysteresis was observed. The critical temperature was defined as the midpoint of the super-normal transition curve.

4. Neutron Irradiation

The samples were irradiated in the pneumatic tube or else in the hydraulic facility of the Kyoto University Research Reactor. For the irradiation in pneumatic tube, the samples were enclosed in a polyethylene case and irradiated for 30 min. The flux densities for the various positions on the neutron energy spectrum were: \( 2.75 \times 10^{13} \) (\( E_\alpha \approx 0.025 \text{ eV} \)), \( 1.09 \times 10^{12} \) (\( 0.025 \text{ eV} < E_\alpha \leq 0.1 \text{ MeV} \)), \( 6.0 \times 10^{13} \text{ n/cm}^2/\text{sec} \) (\( E_\alpha > 0.1 \text{ MeV} \)).

For the irradiation in hydraulic facility, samples encased in silica tube were inserted into an Al capsule filled with water. This assembly was irradiated for either 5 hr or 9.5 hr. The flux densities in the facility were: \( 8.05 \times 10^{13} \) (\( E_\alpha \approx 0.025 \text{ eV} \)), \( 5.95 \times 10^{12} \) (\( 0.025 < E_\alpha \leq 0.1 \text{ MeV} \)), \( 3.9 \times 10^{13} \text{ n/cm}^2/\text{sec} \) (\( E_\alpha > 0.1 \text{ MeV} \)).

During irradiation, the sample temperature was not measured on either sample, but it is unlikely to have been much above 70°C.

III. RESULTS

1. Effects of Aging on \( J_c \)

Figure 1 (a) shows the \( J_c \) vs. \( H \) curves for the series of sample $\#A$. It is seen that the aging brings about monotonical improvement in \( J_c \) up to about 5,000 min, but that further aging causes \( J_c \) to fall back. The corresponding curves for the series $\#B$ are shown in Fig. 1 (b). The difference between these two series of samples can be summarized as follows.

In as drawn state, \( J_c \) is smaller for $\#B$ than for $\#A$. It can be considered that both $\#A$ and $\#B$ contain little precipitate, so that the difference must be due to the dislocation structure induced by the cold work, whose extent could be represented by the reduction in area (99.998% for $\#A$ and 99.87% for $\#B$). With aging, however, this relative intensity is reversed between $\#A$ and $\#B$: \( J_c \) becomes higher for $\#B$ than for $\#A$. This reversal takes place with samples aged about 10 min when the magnetic field is low (below 20 kOe), while under high magnetic field (around 50 kOe), $\#B$ overtakes $\#A$ only when aged for 200 min.

2. Relative Change in \( J_c \) as Function of Neutron Fluence

Figure 2 (a) shows how neutron irradiation affects \( J_c \) of $\#A$ at 50.5 kOe. Here, the effect on \( J_c \) is expressed in terms of the difference in \( J_c \) measured before and after irradiation divided by the value of \( J_c \) before irradiation. The \( J_c \) of $\#A$ decreased with irradiation to \( \times 10^{16} \text{ n/cm}^2 \), but increase with further irradiation to \( 6 \times 10^{17} \) and \( 1.3 \times 10^{18} \text{ n/cm}^2 \). The corresponding changes observed with $\#B$ under similar conditions is shown in Fig. 2 (b). In this case, neutron irradiation tended to increase \( J_c \) when the sample was not aged for more than 50 min, while beyond 100 min aging the effect of irradiation is seen to have been reversed.
IV. DISCUSSIONS

1. Effect of Aging
To show more clearly the effect on $J_c$ brought by aging at 380°C, Figs. 1 (a) and (b) are replotted in Figs. 3 (a) and (b), in terms of the value $J_c H$ ("bulk pinning force") as function of magnetic field intensity $H$. These plots reveal peaks around 30 kOe on the samples #A aged beyond 5,000 min. In the case of #B the corresponding

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Fig. 1 Critical current density vs. applied magnetic field—before irradiation

(a) Nb-47.6% Ti (Sample #A)
(b) Nb-59.8% Ti (Sample #B)

Fig. 2 Effect of neutron irradiation on $J_c$ at 50.5 kOe (Figures correspond to aging time in minutes.)

(a) Sample #A
(b) Sample #B

Relative change $\Delta J_c/J_c$, where $\Delta J_c$ is the difference in $J_c$ before and after irradiation.
peaks make their appearance after only 10 min of aging, first around 20 kOe shifting to a new position around between 200 and 500 min of aging. This shift of the peak is accompanied by a transient setback in the overall level of the bulk pinning force.

Ricketts et al.\(^{(13)}\) indicated that the \(J_cH\) vs. \(H\) curves of Nb-Ti alloys containing precipitates such as \(\alpha\) and \(\omega\), have peaks when the magnetic field is lower than 50 kOe. Samples #A aged beyond 5,000 min, and #B beyond 10 min may contain these precipitates. If we assume that the 30 kOe peak observed in #A aged beyond 5,000 min and in #B beyond 500 min corresponds to the existence of “stable” \(\alpha\) phase precipitates, the #B samples aged between 10 and 200 min should contain “metastable” \(\omega\) phase precipitates.

To further examine the character of the precipitation in #A and #B, the effect of aging on \(T_c\) was plotted for both kinds of samples (Fig. 4). In the case of #A, \(T_c\) shows little change with aging up to the 10,000 min, but #B revealed a distinct increase of \(T_c\) with aging.

Hulm & Blaugher\(^{(14)}\) have reported that the value of \(T_c\) of a solution-treated Nb-Ti alloy system was mainly governed by the Ti concentration, and that the \(T_c\) vs. Ti concentration curve had a peak around 40% Ti, which is lower than the Ti concentration in either #A or #B. If Ti-rich precipitates—such as \(\alpha\) and \(\omega\)—are increased, the Ti concentration of the matrix will decrease and \(T_c\) will rise. The increase of \(T_c\) seen for #B would indicate that Ti-rich precipitates are produced in #B by the aging. The \(T_c\) is lower in samples aged for 500 min than for 200 min, and the \(T_c\) vs. aging time curve of #B has two peaks. The existence of these two peaks may mean a further substantiation of the surmise that two different kinds of precipitates are produced in #B by aging.
The foregoing discussions would indicate that the metallurgical structures of #A and #B varied with changing duration of aging in the following manner.

1. When aged for periods not exceeding 500 min, #A is free of precipitates, at least from evidence so far obtained. Beyond 5,000 min the samples may include a small amount of precipitates.

2. In as drawn state, #B contains little precipitate, but some amount of precipitate is produced by aging up to 50 min. In samples aged beyond 100 min, the presence of precipitates is evident. The precipitates produced by aging up to 200 min may be different from those produced by aging beyond 500 min.

2. Effect of Irradiation

The relative change in $J_c$ at 50.5 kOe after neutron irradiation to a fluence of $1.3 \times 10^{18} \text{n/cm}^2$ is presented in Fig. 5, together with the value of $J_c$ before irradiation, under the same field of 50.5 kOe. In the case of #A, the change is always in the positive direction except with 10 min aging. The direction of change is reversed in #B, except when aged for periods shorter than 50 min. As already suggested, #B aged beyond 100 min should contain precipitates. Based on the above discussions, the present experimental results would indicate that superconducting Nb-Ti wire with the $J_c$ enhanced by the precipitates deteriorates with neutron irradiation.

The effect on $T_c$ brought by irradiation to a fluence of $1.3 \times 10^{18} \text{n/cm}^2$ is presented in Fig. 6. It is seen that the effect is almost nil for #A for all periods of aging, which is also the case with #B up to 100 min aging. Beyond 500 min, however, irradiation tends to reduce $T_c$, which would indicate that the precipitates are disintegrated by the irradiation, and which serves to increase the Ti concentration in the matrix.

V. Conclusion

The $J_c$ enhanced by the Ti-enriched precipitates of superconducting Nb-Ti wire is reduced by neutron irradiation. This is due to breakup of the precipitates by the irradiation. This underlines the importance of the choice of superconducting materials in the application of Nb-Ti
alloys to fusion reactor magnets.

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