Prospect for application of compact accelerator-based neutron source to neutron engineering diffraction

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\textbf{A B S T R A C T}

A compact accelerator-based neutron source has been lately discussed on engineering applications such as transmission imaging and small angle scattering as well as reflectometry. However, nobody considers using it for neutron diffraction experiment because of its low neutron flux. In this study, therefore, the neutron diffraction experiments are carried out using Riken Accelerator-driven Compact Neutron Source (RANS), to clarify the capability of the compact neutron source for neutron engineering diffraction. The diffraction pattern from a ferritic steel was successfully measured by suitable arrangement of the optical system to reduce the background noise, and it was confirmed that the recognizable diffraction pattern can be measured by a large sampling volume with 10 mm in cubic for an acceptable measurement time, i.e. 10 min. The minimum resolution of the 110 reflection for RANS is approximately 2.5\% at 8\,\mu m of the proton pulse width, which is insufficient to perform the strain measurement by neutron diffraction. The moderation time width at the wavelength corresponding to the 110 reflection is estimated to be approximately 30\,\mu s, which is the most dominant factor to determine the resolution. Therefore, refinements of the moderator system to decrease the moderation time by decreasing a thickness of the moderator or by applying the decoupler system or application of the angular dispersive neutron diffraction technique are important to improve the resolution of the diffraction experiment using the compact neutron source. In contrast, the texture evolution due to plastic deformation was successfully observed by measuring a change in the diffraction peak intensity by RANS. Furthermore, the volume fraction of the austenitic phase in the dual phase mock specimen was also successfully evaluated by fitting the diffraction pattern using a Rietveld code. Consequently, RANS has been proved to be capable for neutron engineering diffraction aiming for the easy access measurement of the texture and the amount of retained austenite.

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1. Introduction

Neutron diffraction is known as the only method which can measure internal strains inside crystalline materials non-destructively [1,2]. This is also a useful technique to quantitatively measure microstructural factors of metals [3] such as microstrain, texture and dislocation density in bulk-average, which are strongly related to its mechanical properties such as materials strength and deformability. Quantitative evaluation of these microscopic parameters may bring advanced understanding of the mechanical behavior of metals by investigating the relationship with macroscopic behavior. Neutron engineering diffraction has been well established to evaluate such parameters for a design of advanced metals with outstanding properties and for development of the industrial products with high reliability and low environmental impact. Development of advanced high strength steels is one of the most critical issues to meet above social demands in a variety of industrial fields such as an automotive industry, which urgently requires novel technologies to measure the texture evolution and the amount of retained austenite. Such materials engineering studies using neutron diffraction typically require a neutron engineering diffractometer [2,4–10] installed in large experiment facilities such as a research reactor and a large accelerator to obtain high flux neutrons. Therefore, we have only few chances in a year to carry out challenging neutron experiments using their facilities because of highly competition of the beam.

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time. In contrast, an important demand from industrial users is a lot of opportunities to be able to use neutrons anywhere at any time like commercial X-ray equipment available at their own locations.

A compact accelerator-based neutron source (CANS) [11], based on low-energy accelerators and low-energy charged-particle neutron-producing reactions, might be the only way to meet above demand from industry. We have already had seven accelerator-driven compact neutron sources in Japan, and some more plans to build another compact neutron sources are still running [12]. They are expected to be utilized for component developments [13] and materials engineering researches [14–17], as well as medical irradiation [18]. The compact neutron sources for materials engineering are especially designed to be applied for transmission imaging, small angle scattering and reflectometry [14–17], only for cases that speed of measurement is minor consideration. However, nobody considers using CANS for the neutron diffraction experiment because of its low neutron flux. Nonetheless, nobody has been demonstrated actually so far that CANS is really not applicable for neutron diffraction. If we can evaluate microstructural factors easily by neutron diffraction as well as neutron imaging and small angle scattering using a compact neutron source at our own laboratory, it is expected to achieve efficient developments of industrial products and advanced metals more economically. In this study, therefore, the neutron diffraction experiments are carried out using Riken Accelerator-driven Compact Neutron Source (RANS) [16,17], to clarify a capability of the compact neutron source for neutron engineering diffraction aiming for easy access measurement of the texture and the amount of retained austenite. Furthermore, we discuss the optimum optical system, especially in terms of the moderation time, to realize the high resolution neutron diffraction.

2. Accelerator-driven compact neutron source RANS

Fig. 1 shows an entire view of RANS, which consists of a proton accelerator, a neutron production target and instruments for the neutron experiment. Fig. 2 shows a cross-sectional drawing of a shield box around the target. Protons are accelerated with a proton liniac to 7 MeV, and injected to a beryllium (Be) target with 0.3 mm in thickness [19]. A backing of the Be target is a vanadium (V) plate with 4 mm in thickness, cooling with water flowing in a titanium (Ti) cavity with 5 mm in thickness. Fig. 3 shows a neutron spectrum at the camera box, which was simulated by PHITS code [20]. Neutrons with the maximum energy of about 5 MeV are generated via the Be (p,n) reaction, and the flux-peak appears around 1 MeV. The fast neutrons are moderated in a polyethylene moderator with 40 mm in thickness, and the thermal neutrons with approximately 0.01 eV (0.1 nm in wavelength), which is a suitable energy for the diffraction experiment, can be extracted from the moderator surface. A graphite blocks are placed in a box.

![Fig. 1. Entire view of RANS. This is about 15 m in total length, consists of an ion source, a proton accelerator, a target station, a neutron beam pipe and a camera box.](image1)

![Fig. 2. (a) A schematic of the cross-sectional view of the target station of RANS, and (b) an enlarged detail drawing around the beryllium target.](image2)

![Fig. 3. Energy spectrum of RANS at 5 m far away from moderator simulated by PHITS code.](image3)
with 400 mm in cubic, surrounding the moderator, in order to increase the low energy neutron flux along the neutron beamline by gathering them into the moderator inside the neutron and gamma ray shielding layers. A shielding box around the reflector is a large cube shape with 1.8 m in sides and $2 \times 10^4$ kg in weight, which consists of lead (Pb) layers for gamma-ray absorber and borated-polyethylene layers for neutron absorber. In this shielding box, there is a square window with a size of $100 \times 100$ mm$^2$ to pass protons to the Be target from the liniac, whereas the other square window with the same size is placed in the opposite wall of the shield box to take neutrons from the polyethylene moderator. Neutrons with $10^4$ s$^{-1}$ cm$^2$ in flux are provided to the camera box with the size of $725 \times 700 \times 1190$ mm$^3$, installed at approximately 5 m far away from the moderator. This camera box consists of an aluminum frame including polyethylene and boron, for reducing the background noise from outside. We can build a simple optical system inside the camera box, to achieve the purpose of each experiment. Table 1 shows the specifications of the proton liniac and materials around the target for RANS.

### Table 1

| Liniac                        | Particle | Proton |
|------------------------------|----------|--------|
| Energy                       | 7 MeV    |        |
| Current                      | 20–100 µA|        |
| Operating frequency          | 425 MHz  |        |
| Ion injector output energy   | 30 keV   |        |
| Beam pulse repetition range   | 20–200 Hz|        |
| Pulse width range            | 8–200 µs |        |
| Maximum RF duty factor       | 1.30%    |        |

| Materials (around target)    | Target    | Beryllium: $\phi=50$ mm, $z=0.3$ mm |
|------------------------------|-----------|-------------------------------------|
| Backing                      | Vanadium: $\phi=140$ mm, $z=4$ mm |
| Cooling water cavity         | Titanium: $\phi=152$ mm, $z=10$ mm |
| Moderator                    | Polyethylene: $\phi=180$ mm, $z=40$ mm |
| Reflector                    | Graphite: $0.4 \times 0.4 \times 0.4$ m$^3$ |
| Shielding                    | Lead, borated-polyethylene, Iron: $1.8 \times 1.8 \times 1.8$ m$^3$ |

3. Optical layout for diffraction experiment

In this study, the perspective of compact accelerator-based neutron sources for engineering applications is discussed based on time-of-flight (TOF) neutron diffraction, which is typically utilized in pulsed neutron sources. Fig. 4 shows the experimental setup for measuring diffraction from steel samples. The incident neutron beam emitted from the moderator surface was formed into a square by a B$_4$C collimator and a B$_4$C slit, installed before a sample at approximately 5 m ($=L_1$) far away from the moderator surface. The aperture size of the slit was adjusted to completely illuminate the whole of the sample mounted on a sample table. The sample

![Fig. 4. (a) The overview photograph and (b) the schematic illustration of the experimental setup for the diffraction experiment.](image-url)
was carefully positioned at the center of the incident neutron beam by using a transmission image captured by a CCD behind the specimen. A neutron detector, which consists of a ZnS(Li) scintillator and a position sensitive photo multiplier tube RPMT, were installed inside the Camera box at 90–150° of the diffraction angle, 2θ. The distance from the sample to the detector, L2, was chosen from the range of 110–500 mm, according to the aim of each experiment. Diffraction patterns were measured based on the time-of-flight (TOF) principle. Fig. 5 shows the energy spectrum scattered from a polyethylene sample, which approximately corresponds to the energy spectrum of the incident neutron beam. Energy range from 0.05 to 0.5 nm in wavelength is utilized to measure the diffraction pattern from the steel in this study.

4. Results and discussion

4.1. Background reduction

For low flux compact neutron source, it is very important to reduce the background noise as much as possible, to detect very low signal of neutron diffraction. In the present study, the neutron detector was installed inside the camera box together with the sample and the slit as well as the CCD, which can be factors raising the background noise. Furthermore, the walls of the camera box surrounding the detector might be also a factor of the background noise. In this study, therefore, the B4C sheets, which can absorb low energy neutrons, covered some components around the detector to reduce the background noise. Fig. 6 shows the result of the background reduction by shielding the scattered neutrons by the B4C sheets. The diffraction angle 2θ of the detector was set to be 150°. The sensitive area of the detector was, at first, covered by the B4C sheet to confirm the influence of the background noise derived from neutrons coming from behind the detector. The result in Fig. 6 shows no background noise appears in the energy range higher than 0.05 nm in wavelength. The extremely high intensity background observed in the wavelength range less than 0.05 nm might be derived from fast neutrons which cannot be stopped by B4C, but it is fortunately out of the suitable energy range for major diffraction peaks from steel. On the other hand, the background noise is increased by removing the B4C sheet form the detector window. This result suggests that the background neutrons come from ahead of the detector. Therefore, the B4C sheets covered the whole components including the walls of the camera box, in the range seen by the detector. As a result, the background noise is dramatically reduced by a factor of 4 in the wavelength range more than 0.05 nm, compared with the result without the B4C sheets.

In the present study, as described above, it is confirmed that the background noise is derived from neutrons scattered by the components and walls of the camera box located in front of the detector window. This background noise can be reduced by covering such scatterers with shielding materials such as the B4C sheets. According to the development concept of RANS, we would not like to use large neutron shields because this is a ‘compact’ neutron source. Therefore, only camera box has been utilized for reducing the background noise derived from the incident optical devices outside the camera box. In the diffraction experiment, however, the background noise derived from the camera box itself could decrease the quality of diffraction data. Therefore, it is suggested for the compact neutron source to carry out the neutron diffraction experiments in the open space without the camera box and unnecessary devices around the sample. Note that it is necessary to shield carefully the background neutrons coming from the target side when measuring neutron diffraction in the open space.

4.2. Diffraction pattern

The neutron diffraction pattern of a ferritic steel, which is a typical material for engineering diffraction, was measured using RANS in this study. An annealed ferritic commercial steel (JIS SM400A) with 10 mm in cubic was provided to the experiment. The distance from the sample to the detector, L2 was set to be 120 mm, in order to obtain higher counting statistics, and the diffraction angle 2θ of the detector was set to be 150°.

Fig. 7 shows the diffraction pattern of the ferritic steel specimen, taken for 10 min, which is given as a function of wavelength. This diffraction patterns were calibrated by time-focusing to the center of the detector. Furthermore, the intensity was normalized by the approximate energy spectrum of the incident beam obtained by a polyethylene sample shown in Fig. 5. As shown in Fig. 7, several distinct diffraction peaks can be found in the wavelength range from 0.1 to 0.5 nm, and they can be indexed by the body-centered-cubic (BCC) structure. This result indicates that it is possible to take the recognizable diffraction pattern by the large sampling volume with 10 mm in cubic for an acceptable measurement time, i.e. 10 min. The measurement efficiency may improve by a factor of 10 by using the position sensitive detector and
by increasing the output power of the proton accelerator, suggesting one diffraction pattern can be taken for one minute.

4.3. Engineering applications

To know texture and the amount of retained austenite in steel is important thing for the assessments of the deformability as well as the material strength. Neutron diffraction is an important probe to assess these parameters in bulk-average. Therefore, the compact neutron source can be a useful technology because we can evaluate these parameters easily at our own laboratory, not only at the large neutron experimental facilities.

Texture is an important parameter related to the deformability of material, which is commonly measured by X-ray diffraction or neutron diffraction [21]. We investigated here the possibility of the texture measurement using a compact neutron source. The sample used here was commercial ferritic steel (JIS JSC440W), which was plastically deformed by approximately 20% in tension along the rolling direction. A rectangular-shaped specimen with approximately $10 \times 10 \times 30 \text{ mm}^3$ was prepared by following procedure. The small pieces of the plate with a size of $10 \times 10 \times 1.2 \text{ mm}^3$ were, at first, taken from a tensile-deformed specimen by shear-cutting, and then assembled together into the rectangular shape, preserving the orientation of the pieces. $2\theta$ and $L_2$ were set to be $90^\circ$ and $470 \text{ mm}$, respectively. The diffraction patterns were taken for 90 min to get sufficient intensity. Fig. 8 shows the diffraction pattern in the loading direction of the deformed specimen, in comparison with that of the undeformed specimen. It can be seen in Fig. 8 that the as-received specimen may originally have strong texture even before tensile deformation because the intensity of the 110 reflection is much higher than other peaks. After applying tensile deformation by 20%, the 110 reflection is preferentially oriented more to the loading axis. The intensity of the 110 reflection is obviously increased by 2.2 times, while that of the 200 and 211 reflections that are perpendicular relation to the 110 reflection is decreased by 0.5 to 0.7 times. This is a typical texture evolution for the BCC structure, caused by tensile plastic deformation [22]. This result suggests that the texture evolution due to the plastic deformation can be observed by RANS. TOF neutron diffraction is an advantage for efficient texture measurement by measuring multiple neutron TOF histograms in various directions of the pole figure, simultaneously measured by a wide area detector [23]. For RANS based on TOF neutron diffraction, therefore, it is also expected to measure the texture of material for relatively short time by using similar technique.

Retained austenite in steel is an important parameter related to the toughness of material, which is commonly measured on the material surface by X-ray diffraction [24]. However, it is actually necessary to measure this parameter as a bulk-average since it may have through-thickness variation. Therefore, neutron diffraction technique is useful to assess the amount of retained austenite for the development of the advanced steels. We investigated here the possibility of the measurement of the amount of retained austenite by using a compact neutron source. The dual phase mock specimen was prepared by stacking small cubes of the ferritic steel (JIS SM400A) with the cylindrical austenitic stainless steel plates (JIS SUS316), to control the volume fraction of the austenite to be 19.1%. The diffraction pattern from the dual phase specimen was measured by the detector installed at $2\theta = 140^\circ$ and $L_2 = 140 \text{ mm}$. The specimen was continuously rotated during measurement in order to reduce the influence of texture. Fig. 9 shows the measured diffraction pattern, taken for 60 minutes, which is smoothed by a Binominal smoothing. Diffraction peaks derived from the ferritic and austenitic steels are recognizable, and the volume fraction of
the austenitic phase is predicted to be $170 \pm 3.5\%$ by fitting the diffraction pattern using Z-Rietveld code [25]. This value agrees with the actual value 19.1\% within the error bar, and it is expected to improve the measurement accuracy more by optimizing the measurement condition.

### 4.4. Effect of moderation time on instrument resolution

As shown in Fig. 7, the peak width of each reflection associated with the resolution seems to be larger compared with that of typical neutron engineering diffractometers. In the present study, the resolution was determined by normalizing the full width half maximum (FWHM) of the reflection by its peak position. The peak position and FWHM were obtained by Gaussian fitting. Typical resolution for the engineering diffractometer in the large neutron facility, i.e. TAKUMI in J-PARC/MLF [9], is optimized to be 0.2–0.4\%, in order to perform accurate strain measurement by neutron diffraction. In contrast, the resolution of the 110 reflection in Fig. 7 obtained by RANS is calculated to be approximately 3.2\%, which is lower resolution than typical engineering diffractometer. This result indicates that much higher resolution is required to perform the strain measurement using RANS. Therefore, we discuss here the optimum optical system, especially in terms of the moderation time, to realize the high resolution of TOF neutron diffraction in a compact environment.

The dispersion of the wavelength is related to the dispersion of the flight time of neutrons, which is determined by the neutron pulse width. The neutron pulse width depends on the proton pulse width, $t_p$ and the moderation time, $t_{\text{mod}}$. Furthermore, the dispersion of the diffraction angle is affected by a difference between the estimated neutron path and the real neutron path. We define ‘$z$’ as an effective sample width, which can be seen from the detector. 20 and $L_2$ were set to be 90° and 500 mm, respectively, and it took 50 min to obtain one diffraction pattern.

At first, a change in the resolution was experimentally measured as a function of $t_p$. The proton pulse width, $t_p$ was manually changed from 190 to 8 μs, and the resolution of the 110 reflection was calculated for each $t_p$. Note that the sample width, $z$ was constant at 10 mm in this measurement. It can be found in Fig. 10 that the resolution is almost linearly increased with an increase of $t_p$. The reason why the resolution at $t_p=20$ μs is higher than that in Fig. 7 is a predominant effect of smaller solid angle of the detector due to longer distance from the sample to the detector. Secondary, the effective sample width, $z$ was changed by changing the distance between two samples as shown in an insertion of Fig. 11, and a change in the resolution was experimentally measured as a function of $z$. Note that the proton pulse was constant at 20 μs in this measurement. As shown in Fig. 11, the resolution is also linearly increased with an increase of $z$. According to the results in Figs. 10 and 11, the minimum resolution is predicted to be about 2.5\%, which is still insufficient for the strain measurement by neutron diffraction. Consequently, a way to improve the resolution is investigated below by focusing on the moderation time.

The moderation time for RANS is estimated to be 30 μs in the standard deviation by fitting the plots in Figs. 10 and 11 with following equations:

$$\sigma = \sqrt{\sigma_{\text{mod}}^2 + \sigma_p^2 + \sigma_z^2}$$  

$$\sigma_p \propto t_p$$  

$$\sigma_z \propto z$$

where $\sigma$ is a dispersion of the diffraction peak, determined by three factors, i.e. a moderation time width, $t_{\text{mod}}$, a proton pulse width, $p$, and a sample width, $z$, which are given by the experiments. The standard deviation of a square wave is given by 0.3 times of the width of that. According to the radiation transport Monte-Carlo simulation, the moderation time necessary to obtain 10–20 meV was calculated to be 30 μs, which is equivalent to that of the experimental estimation. Note that the energy range utilized for this simulation was 0.2–0.3 nm in wavelength, corresponding to the 110 reflection. This moderation time width is much longer than 8 μs of the shortest proton pulse, and it is equivalent to approximately 1.8\% of the resolution at the 110 reflection. This result indicates that it is difficult to achieve the resolution to be 1.8% or less even if optimizing the optical layout, since the moderation time is predominantly effective to determine the resolution for the diffraction measurement using RANS. Therefore, it is necessary to optimize the shape and the material of the moderator to obtain the short moderation time, in order to improve the resolution for RANS. According to Fig. 12 showing the simulation results of the timing distribution of neutron emission at the moderator surface, the resolution is expected to be improved by a factor of 2 by decreasing a thickness of the moderator from current 40 to 20 mm. In addition, the application of the decoupler system may improve the resolution as well. These refinements may work better for the strain measurement using the compact neutron source, while it is conversely a disadvantage for
10 min. The resolution of the diffraction pattern is insufficient to the TOF method, which inherently requires long flight paths for achieving a reasonable resolution level. In contrast, angular dispersive neutron diffraction is the other notable technique to improve resolution in a compact environment. Monochromatic beam obtained by a monochromator is expected to bring a reasonable resolution and lower background noise for the diffraction experiment, which may realize the strain measurement by neutron diffraction with compact accelerator-driven neutron sources.

5. Conclusions

In this study, we discussed on engineering applications of a compact accelerator-driven neutron source based on TOF neutron diffraction. However, the discussion about the perspective of compact neutron sources for engineering applications should not be limited to the TOF method, which inherently requires long flight paths for achieving a reasonable resolution level. In contrast, angular dispersive neutron diffraction is the other notable technique to improve resolution in a compact environment. Monochromatic beam obtained by a monochromator is expected to bring a reasonable resolution and lower background noise for the diffraction experiment, which may realize the strain measurement by neutron diffraction with compact accelerator-driven neutron sources.

5. Conclusions

In this study, the neutron diffraction experiments were carried out using Riken accelerator-driven compact neutron source, RANS, and we suggest some guidelines to realize neutron engineering diffraction using the compact accelerator-based neutron source. The diffraction pattern of a ferritic steel was successfully measured by the TOF method in the wavelength range from 0.1 to 0.5 mm using RANS, and it was clarified that the recognizable diffraction pattern can be measured by the large sampling volume with 10 mm in cubic for an acceptable measurement time, i.e. 10 min. The resolution of the diffraction pattern is insufficient to carry out the strain measurement by neutron diffraction with RANS. However, the refinements of the moderator for decreasing the moderation time by optimizing a thickness of the moderator or by applying the decoupler system may improve the resolution. Furthermore, angular dispersive neutron diffraction can be one important technique to improve the resolution in a compact environment. These ideas for improving the resolution may expand applications of compact neutron sources to materials engineering studies. In contrast, the texture evolution due to plastic deformation was successfully observed by measuring a change in the diffraction peak intensity by RANS. Furthermore, the volume fraction of the austenitic phase in the dual phase mock specimen was also successfully evaluated by fitting the diffraction pattern with a Rietveld code. Consequently, RANS has been proved to be capable for neutron engineering diffraction aiming for the easy access measurement of texture and the amount of retained austenite.

In the future scene of the materials development in industry, the compact neutron source will play an important role for screening samples showing unique trend from many samples fabricated by various procedures. These unique samples, selected by the compact neutron source at laboratory, are provided to the rigorous experiment in the large neutron facilities, and obtained results are feed back to the materials development. This might be a future development cycle of industrial products using the compact neutron source and the large neutron facility.

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