The Effect of the Grain Size of Divertor's Cooling Pipe on the Capability of High-Frequency Ultrasonic Tests to Evaluate the Quality of the Bond between Divertor's Cooling Pipe and Armor*)

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This study evaluated the effect of the grain size of the divertor's cooling pipe on the capability of high-frequency ultrasonic tests to evaluate the quality of the bonded interface between the divertor's cooling pipe and armor. First, simple oxygen-free copper and copper-chromium-zirconium block samples with different grain sizes were prepared and measured by an ultrasonic microscope with a 35 MHz probe. The results of the measurements confirmed that the non-uniformity of backwall echoes increased with the grain size of the samples. Samples with large grains provided distinctive signals that can be clearly confirmed on the ultrasonic C-scan images. Subsequently, two bonded samples consisting of 2.5 mm oxygen-free copper bonded with a block of pure tungsten that meets the material specifications of tungsten for ITER component which mimicked the basic design of a divertor's cooling pipe and a monoblock, were measured to evaluate their bonded interfaces. One of the bonded samples bonded at a high temperature provided distinctive signals due to the enlargement of the grain of the oxygen-free copper. Results confirmed that the grain enlargement is the reason for reduced defect detection capability of the high-frequency ultrasonic tests as was suggested previously. This study also revealed that the enlargement of grain caused by improper manufacturing would be non-destructively detectable by high-frequency ultrasonic tests.

Keywords: nuclear fusion reactor, non-destructive inspection, bonded interface, diffusion bonding, heat treatment

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1. Introduction

Divertor is a major in-vessel component in a stellarator or a tokamak-type fusion reactor, and its main function is to remove heat and plasma particles from inside the fusion vessel. One of the most promising divertor designs comprises two structural elements: a tungsten (W) monoblock as the plasma-facing material, and a cooling pipe made from copper-chromium-zirconium (CuCrZr) as the material that facilitates the heat removal procedure [1, 2]. According to the current design of the DEMO divertor, the cooling pipe is bonded to the W monoblock through an interlayer made from oxygen-free copper (OF-Cu) to compensate for the large difference between the thermal expansion coefficients of W and CuCrZr [3–5]. Small defects (e.g., cracks and debonding) that are present at the bonded interface not only block the normal heat transfer procedure but also make the divertor prone to serious damages, like breaking or complete detachment owing to the large stress at elevated temperatures [6]. Thus, performing pre-service non-destructive inspections on the divertor, especially to ensure the integrity of the bonds, is an important issue for the safe and economical operation of a fusion reactor.

Ultrasonic testing is generally recognized as an efficient inspection method for bonded interfaces [7, 8]. While the frequency used for ultrasonic testing is usually no higher than 15 MHz, two recent studies have proven that using a higher frequency improves the detection resolution at the bonded interface of the divertor [9, 10]. These studies also revealed that the grain size of OF-Cu and CuCrZr would affect the capability of a high-frequency ultrasonic test to evaluate the integrity of the bonded interface. Ultrasonic attenuation is well known to depend strongly on the grain size [11–14], because a larger grain size leads to a larger scattering of the ultrasonic wave. In the case of high-frequency ultrasonic tests to inspect the bonded interface, grain enlargements would lead not only to the ultrasonic at-

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tenuation but also to distinctive signals from the interface. The most probable reason for this is that the scattered portion of the ultrasonic waves in the target is rather limited because the target is not so thick compared with the size of enlarged grains. This finding implies that high-frequency ultrasonic tests can non-destructively evaluate the deterioration of pipe strength due to the enlargement of the grain sizes of OF-Cu and CuCrZr [15–18].

Based on the above background, this study evaluated the effect of OF-Cu and CuCrZr grain size on the capability of high-frequency ultrasonic testing to inspect the bonded interfaces between the divertor's cooling pipe and armor. Several OF-Cu and CuCrZr blocks with different grain sizes were prepared. High-frequency ultrasonic tests and metallographic tests were performed to evaluate the effect of grain size on high-frequency ultrasonic signals. Subsequent analyses quantified the dependence of the non-uniformity of measured signals on grain size. Other bonded block samples mimicking the basic design of a divertor were fabricated by bonding OF-Cu with a block of pure tungsten that meets the material specifications of tungsten for ITER components (ITER-grade tungsten block) using a hot-press as the earlier study [9]. One of the bonded samples was bonded intentionally at a high temperature to enlarge the grain size of OF-Cu. High-frequency ultrasonic signals caused by the bonded samples supported the validity of the results obtained using the OF-Cu and CuCrZr blocks. They also confirmed that high-frequency ultrasonic tests can non-destructively detect enlarged grains caused by improper bonding.

2. Materials and Methods

2.1 Sample preparation

To evaluate the effect of grain size on high-frequency ultrasonic signals, this study prepared heat-treated OF-Cu and CuCrZr block samples assuming that the residual strain due to manufacturing condition is negligible and has no effect on the ultrasonic wave propagation. The sample specifications are summarized in Tables 1 and 2. The OF-Cu and CuCrZr samples measured 30 mm × 30 mm and 30 mm × 20 mm, respectively. Heat treatment was carried out using a furnace, and the samples were air-cooled at room temperature after they were kept at a certain temperature in the furnace. The surfaces of the samples were polished manually using abrasive papers to remove oxidation layers generated by the heat treatment. This study assumed that the residual strain due to polishing affects the ultrasonic testing results, but because the thickness of layer where the residual strain remains should be much smaller than the wavelength of the ultrasonic wave, its effect should be negligible compared with the large reflection at the surface. Tables 1 and 2 include information on the grain sizes, specifically average grain areas, of the samples. In this study, the average grain area is used as a measure of grain size and is evaluated according to the procedure described in Section 2.3. The grain structures were assumed uniform and isotropic. Table 1 shows that the heat treatment enlarged the grains of the Cu samples. The reason why Cu3 had the largest grains is unclear, which does not affect the discussion in this study because the purpose of this study is to evaluate the effect of grain sizes. By contrast, Table 2 reveals that the heat treatment had almost no effect on the average grain sizes of the CuCrZr samples.

Table 1 Specification of OF-Cu samples*1.

ID	Temperature [°C]	Holding time [h]	Ave. Grain area [mm^2]
Cu1	N/A*2	N/A*2	1.3×10^{-3}
Cu2	500	1	2.0×10^{-3}
Cu3	800	3	1.9×10^{-1}
Cu4	900	1	9.5×10^{-2}
Cu5	900	3	1.3×10^{-1}
Cu6	900	6	1.4×10^{-1}

*1 The temperature and holding time indicate the condition of the heat treatment.
*2 Cu1 was not heat-treated.

Table 2 Specification of CuCrZr samples*1.

ID	Temperature [°C]	Holding time [h]	Ave. Grain area [mm^2]
CuCrZr1	N/A*2	N/A*2	3.3×10^{-3}
CuCrZr2	500	3	3.7×10^{-3}
CuCrZr3	900	6	4.7×10^{-3}

*1 The temperature and holding time indicate the condition of the heat treatment.
*2 CuCrZr1 was not heat-treated.

In addition to the OF-Cu and CuCrZr block samples, two bonded W-Cu samples simulating the divertor were manufactured. These samples consisted of a 2.5 mm OF-Cu bonded to an ITER-grade W block by hot-press using a high-temperature vacuum furnace (HP-10X10-CC-23, NEMS Co., Ltd., Saitama, Japan). Two temperatures (i.e., 850°C and 950°C) were used for the bonding to have different grain sizes. Bonding pressures was set to 10 MPa and time to 1 hour. The more specific procedure of the bonding can be found in previous reports by the authors [9, 10].

2.2 High-frequency ultrasonic test

High-frequency ultrasonic testing was performed using an ultrasonic microscope (IS-350, Insight k.k., Tokyo, Japan) with a 35 MHz probe in pulse-echo mode. The vertical position of the probe was adjusted so that the surface echoes have the largest amplitude. The probe scanned a 40 mm × 40 mm area in 0.1 mm pitch with two different amplifications to normalize echoes from inside the sample with respect to the amplitude of the surface echo.

Measured ultrasonic echoes were converted into signals from a certain depth from the surface based on their time-of-flight. The speed of sound used for the conversion was 4660 m/s for all the samples, though in reality the speed would be non-uniform and anisotropic especially when the sample has large grains. The present study assumed that the backwall echoes of OF-Cu and CuCrZr samples are contained in signals whose depths range from 2.5 mm to 3.5 mm. To evaluate the bonded interface of the W-Cu bonded samples, signals from 2.0 mm to 3.0 mm deep were analyzed.

2.3 Metallographic test

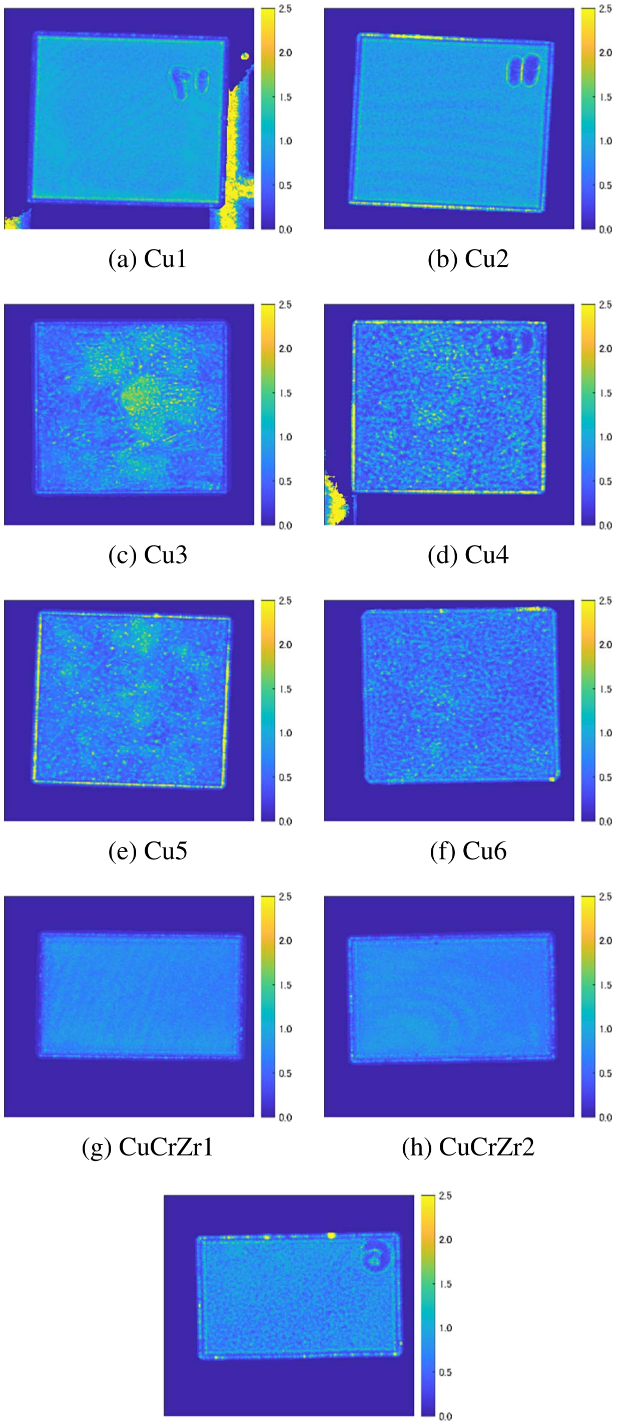
Metallographic tests were performed to evaluate to what extent grains were enlarged and whether the bonded interfaces contained flaws. This study used average grain area as a measure of grain size following ASTM E112-13 (2021) to quantify the size of grains. To confirm the presence of flaws at the interface, the two W-Cu bonded samples were sectioned and observed by a digital microscope.

3. Results and Discussion

Figure 1 presents the backwall echoes of the OF-Cu and CuCrZr samples. The color in the figure represents the normalized amplitude of echoes in percentages. Strong signals outside the sample in Figs. 1 (a) and (d) are due to a jig situated under the sample so that the backwall echoes can be clearly distinguished from the echoes from the bottom of the tank of the instrument. Some of the samples had their tentative ID marked on the surface, which can be clearly confirmed in the figure. The figure shows that the enlarged grain resulted in fluctuations in pixel intensities in the resulting image. In other words, the enlargement of grain size makes the backwall echo non-uniform.

To quantify the non-uniformity of backwall echoes due to the increase in grain size, Fig. 2 shows the relationship between average grain area and standard deviation of the amplitude of the normalized backwall echoes. The figure clearly shows that the non-uniformity tends to increase with grain growth. In reality, however, it would not be reasonable to assume that the samples prepared in this study had a constant grain size as shown in Fig. 3. Whereas analyzing the results shown in Fig. 1 based not on the average but on the distribution of grain sizes would enable a more quantitative discussion on this matter, further analyses were avoided because how ultrasonic waves are scattered in the target is outside the scope of this study.

Figure 4 shows ultrasonic echoes from the bonded interfaces of the two W-Cu bonded samples. Because of the difference in the thicknesses of the target and acoustic impedances, the results shown in Fig. 3 are not quantitatively comparable with those in Fig. 1. However, the sample bonded at the higher temperature provides a C-scan image similar to the one from the block samples with large grain sizes. Figure 5 presents the histograms of the normalized amplitudes of ultrasonic echoes from the bonded interface shown in Fig. 4. The echoes due to the sample with the higher bonding temperature led to smaller mean and larger standard deviation, which is attributed to ultrasonic scattering because of the enlarged grains. Subsequent metallographic test confirmed that the grain of the OF-Cu of the bonded sample enlarged, as shown in Fig. 6. In addition, the metallographic test confirmed that there was no flaw such as delamination at the bonded interfaces of the two bonded samples.



(a) Cu1 (b) Cu2 (c) Cu3 (d) Cu4 (e) Cu5 (f) Cu6 (g) CuCrZr1 (h) CuCrZr2 (i) CuCrZr3

Fig. 1 Ultrasonic C-scan images of backwall echoes.

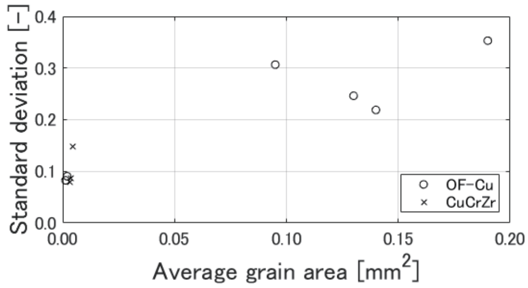
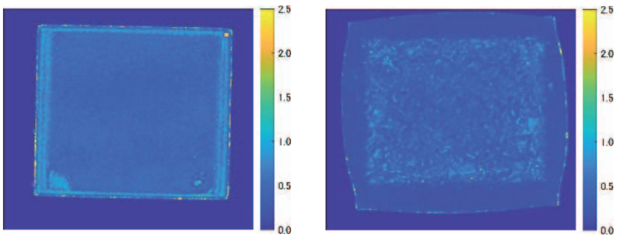


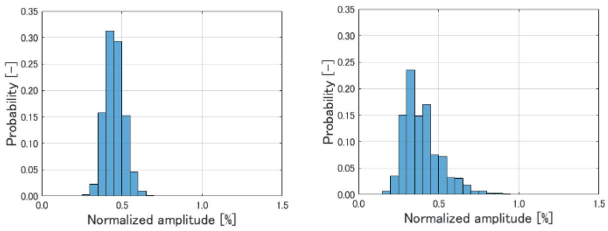
Fig. 2 Relationship between average grain area and standard deviation of backwall echoes.

Fig. 3 Grain structure of sample Cu3 with a wide grain size distribution.



(a) 850°C (b) 950°C

Fig. 4 Ultrasonic C-scan images of the bonded interfaces of the W-Cu bonded samples.



(a) 850°C (b) 950°C

Fig. 5 Histograms of the ultrasonic signals shown in Fig. 4.

To discuss the effect of enlarged grain size on the capability of high-frequency ultrasonic test to inspect the bonded interface, Fig. 7 presents the ultrasonic signals from another W-Cu bonded sample used in the earlier study of the authors [9]. The sample had an artificial groove on the W surface to introduce artificial delamination on the bonded interface, which can be clearly confirmed at the center of Fig. 7 (a). Because the bonding temperature of the sample was 900°C, the non-uniformity of the signals from the bonded interface would be somewhere between those of Figs. 4 (a) and (b). Because the bonding temperature of the sample was 900°C, the non-uniformity of the signals from the bonded interface would be somewhere between those of Figs. 4 (a) and (b). Figure 7 (b) shows the histogram of the normalized amplitude of ultrasonic echoes due to the delamination. A comparison of Fig. 7 (b) with Figs. 5 (a) and (b) implies that the signal change caused by enlarged grains would mask the signals due to a flaw at the bonded interface. However, it would be preferable that one can non-destructively evaluate the grain enlargement because, in reality, both bonded interfaces containing large flaws and improperly bonded interfaces with large grain sizes need to be excluded.

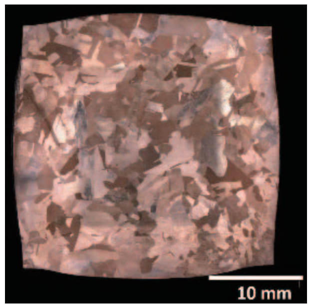
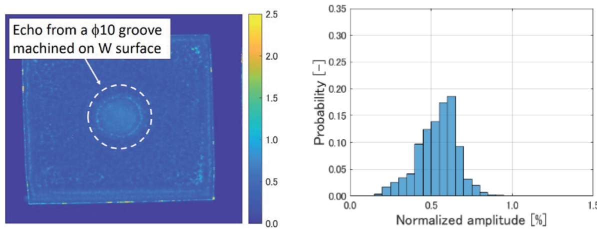


Fig. 6 Cu grains of the W-Cu bonded sample bonded at 950°C.



(a) Ultrasonic image (b) Histogram

Fig. 7 Ultrasonic signals due to a bonded interface with a ϕ10 mm artificial groove ((a) Ultrasonic C-scan image, (b) Histogram of ultrasonic signals in the area surrounded by the broken circle in Fig. 6 (a)) [9].

4. Conclusion

This study evaluated the influence of grain size of divertor's cooling pipe on the capability of high-frequency ultrasonic tests to evaluate the quality of the bond between divertor's cooling pipe and armor. Several OF-Cu and CuCrZr block samples with different grain sizes were prepared. High-frequency ultrasonic tests were performed using a 35 MHz ultrasonic probe in the pulse echo mode. The test confirmed that the enlargement of grain sizes increases the non-uniformity of signals, which can be easily confirmed by ultrasonic C-scan images. This was supported by the results obtained from the bonded W-Cu samples mimicking the bond between the divertor's cooling pipe and armor. Although the effect of grain growth on the defect detection sensitivity was not directly analyzed in this study, the current results confirm that grain enlargement is actually the reason of reduced signal-to-noise ratio of high-frequency ultrasonic tests to detect tiny flaws at the interface, as was suggested in our earlier report [9]. In contrast, this study also demonstrated that the enlargement of grain size, which has a negative effect on the structural integrity, can be non-destructively detected using high-frequency ultrasonic test.

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[1] G. Mazzone *et al.*, Fusion Eng. Des. **157**, 111656 (2020).
[2] G. Pintsuk *et al.*, Fusion Eng. Des. **174**, 112994 (2022).
[3] J.H. You *et al.*, Fusion Eng. Des. **175**, 113010 (2022).
[4] J.H. You *et al.*, J. Nucl. Mater. **544**, 152670 (2021).
[5] M. Richou *et al.*, Physica Scripta **2017**, 014022 (2017).
[6] J.H. You, *et al.*, Fusion Eng. Des. **164**, 112203 (2021).
[7] S. Roccella *et al.*, Fusion Eng. Des. **146**, 2356 (2019).
[8] G. Dose *et al.*, Fusion Eng. Des. **146**, 870 (2019).
[9] N. Yusa *et al.*, Plasma Fusion Res. **17**, 2405013 (2022).
[10] N. Yusa *et al.*, Fusion Eng. Des. **187**, 113367 (2023).
[11] S. Hirsekorn, J. Acoust. Soc. Am. **72**, 1021 (1982).
[12] E.P. Papadakis, J. Acoust. Soc. Am. **33**, 1616 (1961).
[13] M. Norouzian *et al.*, Ultrasonics **102**, 106032 (2020).
[14] A. Van Pamel *et al.*, J. Acoust. Soc. Am. **143(4)**, 2394 (2018).
[15] A.P. Arguelles and J.A. Turner, J. Acoust. Soc. Am. **141**, 4347 (2017).
[16] E. Tejado *et al.*, J. Nucl. Mater. **498**, 468 (2018).
[17] M. Wirtz *et al.*, Nucl. Mater. Energy **12**, 148 (2017).
[18] E. Tejado, Mater. Sci. Eng. A **712**, 738 (2018).