Use of Electromagnetic Acoustic Resonance Method to Detect Micro-Voids Via Evaluation of Ultrasonic Wave Attenuation Coefficient of SUS304 Steel Fabricated by Hot Isostatic Press*

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Creep damage in SUS304 steel samples fabricated by a hot isostatic press (HIP) at 1050°C was evaluated using the electromagnetic acoustic resonance (EMAR), noise energy and ultrasonic spectroscopy (first moment) methods. The coefficients of attenuation of 1.1 to 5.4 MHz electromagnetically excited acoustic waves in the samples were investigated. By reducing diffraction loss and the loss to the electromagnetic acoustic transducer due to transmission of the ultrasonic waves via mechanical contacts, it was possible to detect shear wave attenuation coefficients as low as 2.5×10^{-4} /microsecond at 1.1 MHz. With specimens fabricated at pressure of 170 MPa, the attenuation coefficient increased in proportion to frequency up to 5.4 MHz, whereas with specimens fabricated at pressure lower than 80 MPa the attenuation coefficient increased rapidly above 4 MHz. The void fraction was found to be greater in specimens fabricated under lower pressure, which may be responsible for the markedly higher ultrasonic attenuation in the specimens fabricated at pressure lower than 80 MPa. It was apparent from the experiments that the EMAR method detects creep voids with greater sensitivity than the other methods. Accordingly we hope it will be possible for the EMAR method to be used on real facilities in the future.

Key Words: Ultrasonic Attenuation, Electromagnetic Acoustic Resonance (EMAR), SUS304, Shear Wave, Diffraction Loss, Thermoelastic Dissipation, Hot Isostatic Press, Creep Voids

1. Introduction

Currently, more than half of Japan's thermal power plants have exceeded 100 000 hours of operation, which means that it is highly necessary to correctly ascertain the state of their secular deterioration

and implement appropriate repair. The technology needed for such evaluation is becoming increasingly important. Research by the authors in recent years⁽¹⁾ has shown that in some cases creeping damage occurs inside the welded joints of the high-temperature portions of the plants' boilers, starting from the interior of the walls, and that such damage cannot be evaluated from the outer surfaces alone unless the equipment has already reached the end of its service life; in order to secure reliability there is an increasing need for detection of interior damage that is on the order of microns.

The state of surface deterioration can be ascertained by means of the replica method, creep damage diagnosis systems⁽²⁾, and so forth. But at present such methods can only ascertain fissures on the order of millimeters inside the welded joints; they do not

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permit precise evaluation of creep and other micron-order deterioration. Recently reports have appeared in the literature to the effect that creep damage can be evaluated using the Electromagnetic Acoustic Resonance (EMAR) method⁽³⁾, but this method has only been applied to base materials that are not susceptible to creep voids and has not been used specifically for creep void detection.

It has long been known that these micron-order physical quantities can be determined non-destructively by measuring the ultrasonic attenuation⁽⁵⁾⁻⁽⁷⁾ of metallic materials, since the coefficient of ultrasonic attenuation varies with the crystal grain size, dislocation density and other aspects of such materials. And for some time, piezo-electric elements have been used to measure the attenuation of ultrasonic waves. But in addition to the ultrasonic attenuation inside the specimen being measured, simultaneous attenuation of the probe and within the acoustical coupling agent is also observed. Correcting such extraneous attenuation is problematic, since it is very closely dependent on the pressure with which the probe is applied⁽⁸⁾. By contrast, the EMAR method is able to excite and detect ultrasonic waves without bringing the probe into contact with the specimen^{(9),(10)}. This means that attenuation measurement by means of EMAR can precisely ascertain the coefficient of attenuation inside the specimen, without the need to take account of attenuation of the waves by the probe or inside the acoustical coupling agent—unlike attenuation measurement using a piezo-electric element. However, EMAR measures the attenuation of standing waves, so that in order for it to determine the ultrasonic attenuation inside the specimen, correction must be made for the fact that ultrasonic waves spread throughout the whole of the specimen. Further, although EMAR does permit measurement without contact between the specimen and probe, in actual practice EMAR measurement is carried out with the probe in contact with the specimen, so that attenuation due to such contact is also present.

In the present study, we fabricated SUS304 steel samples with artificial voids using a hot isostatic press (HIP)⁽¹¹⁾, and measured the ultrasonic attenuation due to the artificial voids, which represented micron-order deterioration in a real situation, by suppressing the above-mentioned attenuation due to diffusion of the ultrasonic waves and to the probe. The results showed that the sensitivity of EMAR to voids is higher than that of the methods currently used in the field, such as the ultrasonic noise energy method⁽¹²⁾ and ultrasonic spectroscopy (first moment) method⁽¹³⁾; and that therefore EMAR has the potential to be applied for diagnosis of creep damage in

boiler welded joints in the future.

2. Fabrication of Samples

The SUS304 steel samples were fabricated from steel powder by means of treatment in an HIP. The SUS304 steel powder used had average grain size of 10 microns (maximum size 44 microns). The fabricated samples were 40 mm in diameter and 10 mm thick. HIP treatment was at a temperature of 1050°C maintained for 2 hours; during the treatment, pressing pressures of 20, 40, 80 and 170 MPa were applied to samples A, B, C and D respectively. For the EMAR measurement, specimens measuring 10×20×10 mm³ were cut out from the central portion of the samples. The surfaces of the samples were buffed until their irregularity was no more than 1 000 Å (0.1 micron).

Figure 1 shows SEM images of the samples after their surfaces had been etched with aqua regia following HIP treatment and buffing. All of the samples consisted of fine crystal grain with grain size around 10 microns. In sample A, which underwent the lowest-pressure HIP treatment, many voids of several microns in diameter are visible. These voids were also observed prior to etching, and are probably the crevices that are apt to form during HIP treatment. The higher the pressure during HIP treatment, the larger was the size of the voids and the lower their density. When specimens were cut out from the HIP-treated samples and the exposed sample interiors were observed with an SEM, no difference in crystal grain size or void size or density was observed between the samples' surfaces and their interiors.

Table 1 gives the results for void fraction of each sample, as determined by taking a replica from the

Fig. 1 SEM images of SUS304 fabricated by hot isostatic press with pressure of (a) 20 MPa, (b) 40 MPa, (c) 80 MPa and (d) 170 MPa

Table 1 Void area fraction of each sample

Sample NO.	Pressure (MPa)	Void area fraction (%)
A	20	0.21
B	40	0.16
C	80	0.13
D	170	0.04

sample surface, taking an SEM image of the replica, and applying image analysis processing to the resulting image. In each case, the void fractions of 3 sample regions (each 0.3 mm² in area) on the central portion of the replica specimen were calculated, and the average value for the 3 regions was taken as the sample's void fraction. It will be seen that the higher the pressure during HIP treatment, the smaller was the void fraction, which fell from 0.21% in sample A through 0.16% and 0.13% in samples B and C, to 0.04% in sample D.

3. Measurement Using the Various Void Detection Methods

3.1 Measurement using EMAR

Figure 2 shows the structure of the electromagnetic acoustic transducer (EMAT) used for exciting the ultrasonic waves. The EMAT consists of coils wound in a sports track-like formation, plus a permanent magnet. When high frequency current is passed through the coils, eddy current is excited on the surface of the specimen, and interaction between the eddy current and the permanent magnet's magnetic field generates Lorentz force. The Lorentz force acts in a direction parallel to the surface, exciting transverse waves. To receive (detect) wave signals, the EMAT uses the reverse of the above signal transmission process.

Now let us consider the ultrasonic waves excited by the EMAT, which propagate in the direction of the specimen's thickness. These waves are repeatedly reflected on both sides of the specimen, producing multiplex echoes. Scanning of the frequency of these reflected ultrasonic waves showed that when the half-wave length was equal to an integer multiple of the specimen's thickness, all of the reflected waves were in phase. At such times, the multiplex echoes caused resonance which appeared as peaks in the amplitude spectrum. These frequencies are referred to as "resonance frequencies". The n th order resonance frequency fn is given by the following equation, in terms of acoustic velocity c and specimen thickness d :

$$fn = \frac{nc}{2d} \quad (1)$$

The use of resonance frequencies to determine acoustic velocity or specimen thickness is known as

Fig. 2 Lorentz force mechanism for shear-wave excitation by electromagnetic acoustic transducer (EMAT)

the "resonance method". It is a method that is particularly effective for measuring the acoustic velocity of thin specimens, but when it employs a piezo-electric probe (as it has traditionally), it requires complex correction because of the composite resonance modes involving resonance of the probe, acoustical coupling agent and specimen, that will occur. But if it is used with an EMAT, which permits measurement without contact between the probe and specimen, the resonance method can provide resonance spectra of the specimen's interior alone. This combination (resonance method+EMAT) is dubbed the "electromagnetic acoustic resonance" (EMAR) method.

With the EMAR method, resonance waves are excited, then the excitation source is stopped and the variation in the resonance waves' intensity over the subsequent time period is measured; measurement of this kind permits determination of the attenuation coefficient of the ultrasonic waves. The following 4 factors are liable to cause energy loss in attenuation measurement using EMAR:

- (1) Attenuation inside the specimen (solid)
- (2) Attenuation due to contact between the specimen and the EMAT
- (3) Electrical-mechanical connection between specimen and EMAT
- (4) Diffraction of ultrasonic waves

(1) is attenuation of ultrasonic waves in the specimen (solid) interior due to a micron-order energy loss factor. This is the attenuation that was the object of measurement in the present study. Further details will be given in 4. "Discussion" below.

(2) is ultrasonic wave attenuation due to mechanical contacting between the specimen and the EMAT. Although in principle the EMAR method permits excitation and detection of ultrasonic waves without any contact between the specimen and EMAT, in actual use the latter has to be spatially supported while it is performing measurement, and the practice is to provide such support by fastening

the EMAT onto the specimen. As a result, ultrasonic attenuation due to the contact of the specimen with the EMAT is inevitable. Even so, the EMAR method does not require the acoustic connection between the probe and specimen that the piezo-electric element requires; consequently the degree of ultrasonic attenuation due to contact is much smaller with EMAT than with the piezo-electric element method.

(3), ultrasonic attenuation due to electrical-mechanical connection, is the reverse process of the ultrasonic wave excitation that the EMAT performs. Accordingly (3) is used for detection of ultrasonic waves (despite being a process that causes attenuation of the waves). The coefficient a_e of attenuation due to electrical-mechanical connection was determined by Ogi et al.⁽⁴⁾ to be given by the following equation:

$$a_e = \frac{sB_0^2}{2r} \quad (2)$$

where B_0 is the magnetic flux density, r is the density of the specimen, and s is its electrical conductivity. Assuming $B_0=1.0$ and substituting the SUS304 steel values of $s=1.4 \times 10^6$ (S/m) and $r=7900$ kg/cm³, we have $a_e=1.0 \times 10^{-4}$ (1/ μ sec).

(4), loss due to diffraction, is caused by diffusion throughout the specimen of the resonance waves excited by the EMAT. The size of such loss is dependent on both the effective area of the EMAT and the area of the specimen. Ogi et al. determined the size of the loss due to diffraction by means of calculations using the finite-element method, and carried out correction for such loss. According to their calculations, when converted to an attenuation coefficient the loss due to diffraction is a high-order value of 1×10^{-2} (/microsecond)⁽¹¹⁾. With the SUS304 steel used in the present study, the coefficient figure obtained for the micron-order specimen-internal attenuation under investigation was a low-order value of 1×10^{-4} (/microsecond), as will be discussed later. That made it problematic to numerically correct the attenuation curves (which incorporated diffraction loss) for diffraction effects so as to determine specimen-internal loss. Accordingly, in order to reduce loss due to diffraction the specimens were cut from the samples to a size such that their surface area that would contact with the EMAT was practically the same as the EMAT's effective area.

3.2 Measurement using the ultrasonic noise energy method⁽¹²⁾

Details of the measurement method, etc., can be found in Ref.(12). The apparatus used in the present study consisted of a 50 MHz pulser-receiver, a 20-40 MHz frequency-band power spectrum analyzer, a digital oscilloscope, a broadband probe and a personal

computer. The probe was vertical type (15 MHz) and was used to measure the central portions of the specimens, via commercially-available machine oil as the contacting medium.

3.3 Measurement using the ultrasonic spectroscopy method⁽¹³⁾

Details of the measurement method, etc., can be found in Ref.(13). The apparatus used in the present study is listed below. The method of soaking with broadband ultrasonic waves was employed, and time domain waveforms in the measurement area were measured using a suitably minute spacing. Then the backscattering wave signals appearing between the surface echo and bottom echo (in particular the first moment signals) were analyzed so as to evaluate the creep damage.

Probe: Point-focusing type longitudinal wave probe (nominal frequency 10 MHz, nominal focal length 3 inches, actual focal length 104 mm, transducer diameter 0.375 inches) Measurement area: Central portion of specimens

Spacing of measurements: 0.25 mm

Analysis parameter: First moment = $\left(\int S(f) \cdot f df \right) / \left(\int S(f) df \right)$, where S is size of backscattering waves and f is frequency

4. Experimental Results

Figure 3 shows resonance spectra obtained by scanning 10×20 mm² specimens from sample D at excitation frequencies of 1 to 5 MHz. The bullets in the figure indicate the peaks, which appeared at regular time intervals. These were resonance peaks produced by the longitudinal waves advancing in the direction of the specimen's thickness. The acoustic velocity as determined from the peaks' interval was 3150 m/sec.

Figure 4 shows attenuation curves measured at resonance frequency of 4.2 MHz. Figure 4(a) is the curve of the attenuation coefficients measured by the EMAT on the central portion of sample D prior to cutting, when its dimensions were diameter 40 mm $\times$ thickness 10 mm. Figure 4(b) shows the curve measured for a 10 mm $\times$ 20 mm² specimen cut out of the central portion of sample D. In both cases resonance waves were excited by a single 20 microsecond burst of 4.2 MHz sine waves. Figures 4(a) and 4(b) both plot ultrasonic wave intensity against time elapsed after stopping of wave excitation; the intensity is integrated for a time gate of 20 microseconds in Fig. 4(a) and for one of 50 microseconds in Fig. 4(b).

In Fig. 4(a) the signal is attenuated sharply and intensity falls to zero after approximately 180

Fig. 3 Resonance pattern of electromagnetically excited ultrasonic wave on SUS304 plate with thickness of 10 mm fabricated by HIP with pressure of 170 MPa

microseconds, but following that the signal reappears. The sharp attenuation is due to transverse diffusion of the resonance waves excited by the EMAT, while the reappearance of the signal is thought to be caused by waves reflected at the edges of the specimen returning to the central portion.

The attenuation of the ultrasonic waves can be expressed by the following exponential function, using an attenuation coefficient a :

$$I(t) = 10 \exp(-at) \quad (3)$$

The solid line drawn in Fig. 4(a) represents the result of applying Eq. (3) to the initial steeply-falling portion of the attenuation curve. The attenuation coefficient thereby obtained was 1.4×10^{-2} /microsecond. The attenuation coefficient derived from Fig. 4(b) on the other hand was 2.7×10^{-3} /microsecond. The difference between the two coefficients may be assumed to be due to apparent attenuation resulting from diffraction of the ultrasonic waves. In the remainder of the experiments discussed here, measurement was performed on specimens cut to a size of $10 \times 20 \text{ mm}^2$ in order to reduce the effects of diffraction.

Figure 4(c) shows the result of ultrasonic

Fig. 4 Ring down curves of ultrasonic wave in SUS304 with the shape of (a) $\Phi 40 \times 10$ (b) $10 \times 20 \times 10$ and (c) $10 \times 20 \times 10$ measured by using EMAT with effective area of $10 \times 14 \text{ mm}$

attenuation measurement in which paper spacers $1 \times 10 \text{ mm}$ in area and 0.1 mm thick were interposed between the EMAT and the specimen in such a way as to leave the specimen's central portion clear of the EMAT; this was in order to reduce attenuation due to contacting between the EMAT and the specimen. By contrast, the spectra in Figs. 4(a) and 4(b) were obtained from measurement in which the specimens were fastened to the top of the EMAT by double-sided adhesive tape. In Fig. 4(c) the attenuation coefficient is much smaller than in the other 2 cases, being just 4×10^{-4} /microsecond.

Figure 5 shows the measurements of the ultrasonic attenuation coefficient of sample D. Specifically, Figs. 5(a)–(e), show the results for specimens measured using 1.1, 2.2, 3.6, 4.2 and 5.4 MHz waves respectively. It will be seen that the higher the frequency of the waves, the more rapidly they were attenuated.

Oscillation with a period of 180 microseconds can be observed in the attenuation curve in Fig. 5(a); this swell in the waves is thought to have occurred because certain waves distinct from the (approximately) 1.1 MHz transverse waves advancing in the thickness direction resonated and caused interference.

Figure 6 plots the attenuation coefficient of sample D against frequency. The solid dots indicate the experimental results, while the hollow dots represent the same values corrected for electrical-mechanical connection loss of 1×10^{-4} /microsecond using Eq. (2). As can be seen, the uncorrected attenuation coefficients already vary almost linearly with respect to frequency, and after correction for electrical-mechanical loss the coefficients arrange themselves neatly on a straight line passing through the graph's origin.

Figure 7 gives the attenuation curves measured for sample A, which underwent HIP treatment at

pressure of 40 MPa. A comparison of Fig. 7(c) with Fig. 4(c) above will show that ultrasonic waves were attenuated more rapidly in sample A than in sample D. Figure 8 plots the various attenuation coefficients of the 4 samples against frequency. This graph shows that the difference among the attenuation coefficients of the 4 samples is small at frequencies of 4 MHz and

Fig. 5 Ring down curves of ultrasonic wave in sample D with the shape $10 \times 20 \times 10 \text{ mm}^3$ measured by using EMAT with effective area of $10 \times 14 \text{ mm}$

Fig. 6 Frequency dependence of attenuation coefficient of sample D

Fig. 7 Ring down curves of ultrasonic wave in sample A with the shape $10 \times 20 \times 10 \text{ mm}^3$ measured by using EMAT with effective area of $10 \times 14 \text{ mm}$

Fig. 8 Frequency dependence of attenuation coefficient of samples A, B, C and D

Fig. 9 Relation between atten. coeff. and void area fraction in samples A, B, C and D

below but becomes large at frequencies above 4 MHz; and also that samples that underwent HIP treatment at relatively low pressure had higher attenuation coefficients.

Figure 9 illustrates the relation between void fraction and attenuation coefficient for parameters of 4.2, 4.4 and 4.7 MHz. The void fraction increases as the attenuation coefficient increases, and 4.7 MHz produces the best approximation to a linear relation between the two. Further, the multiple correlation coefficient R^2 is 0.9 or higher for each of the frequencies, indicating a strong correlation.

Figure 10 shows the relation between void fraction and attenuation coefficient with the EMAR (4.7 MHz), ultrasonic noise energy and ultrasonic spectroscopy (first moment) methods taken as the parameters. The vertical axis is normalized for sample D (whose void fraction was 0.04%). Both the EMAR and noise energy methods yield a strong positive correlation between attenuation coefficient and void fraction (0.8 or higher), but with EMAR the approximation to a linear relation between the two is 1.5 times better than with the energy noise method.

Fig. 10 Comprison of EMAR method with other ones at different creep damage in samples A, B, C and D

With the spectroscopy method there was practically no variation in attenuation coefficient with void fraction.

5. Discussion

The most likely causes of attenuation of ultrasonic waves inside a solid are thermoelastic effects, scattering by voids, scattering at grain boundaries, and scattering due to dislocations:

$$a = a_{t-e} + a_{\text{void}} + a_{GB} + a_{dis} \quad (4)$$

Attenuation due to thermoelastic effects a_{t-e} constitutes wave absorption that is proportional to frequency f :⁽¹⁴⁾

$$a_{t-e(f)} = a \times f \quad (5)$$

where a is a constant.

Scattering due to voids is known to increase in proportion to the square of the void radius. The dependence of such scattering on frequency however cannot be expressed so simply as a power function; the calculations of Maeda et al. have shown that such scattering is small at low frequencies but increases sharply from about 5 MHz upward⁽¹⁵⁾.

Attenuation at grain boundaries is proportional to the 4th power of the frequency and is given by the following equation:

$$a_{GB(f)} = S \times D^3 \times f^4 \quad (6)$$

where S represents the scattering factor and D is the average grain size. Bhatia and Moore⁽¹⁶⁾ estimated the value of S for iron to be 1.29×10^{-10} microsecond³/micrometer³. If we substitute this value for iron in Eq. (6) (in place of an SUS304 steel value), and also insert 10 microns as the grain size value (derived from the results in Fig.1), we will obtain the figure of 8×10^{-5} /microsecond at 5 MHz for $a_{GB(f)}$ (coefficient of attenuation due to grain boundary scattering). This is 1 decimal place smaller than the attenuation coefficient of 8×10^{-4} /microsecond observed for sample D; it therefore seems safe to assume that the

effects of grain boundary scattering on attenuation can be ignored in the present study.

Attenuation due to dislocations $a_{dis(f)}$ is proportional to the square of the frequency and is given by the following equation⁽¹⁷⁾:

$$a_{dis(f)} = \frac{16GBb^2}{\pi^4 C^2} \times \lambda L^4 f^2 \quad (7)$$

where G is the modulus of transverse elasticity, B is the friction coefficient, λ is the dislocation density, L is the dislocation loop length, b is the Burgers vector for the dislocations, and C is the tension generated by dislocations.

It is apparent from Fig. 7 that sample D's attenuation coefficient was proportional to frequency; this dependence on frequency would indicate that attenuation was due to thermoelastic effects in this sample. In the other 3 samples by contrast, attenuation increased markedly in the high frequency range, probably because of scattering due to dislocations and voids. Incidentally, the fact that attenuation in sample D was proportional to frequency presumably means that attenuation due to dislocations can be ignored for that sample. Moreover, since all 4 samples were fabricated by means of HIP treatment, sample D is unlikely to have had more dislocations than the others, and therefore we may ignore attenuation due to dislocations in the others as well. Table 1 demonstrates that the lower the pressure used for HIP treatment of a sample, the greater was the sample's void fraction. From this it may be inferred that the reason why samples that underwent HIP treatment at lower pressures exhibited greater ultrasonic attenuation was that there was more scattering due to voids in such samples.

Additionally, the fact that the EMAR method yielded a better approximation to a linear relation (between attenuation and void fraction) than the energy noise and spectroscopy methods shows that it has greater sensitivity to voids. We may infer that the reason for such greater sensitivity is that whereas the other 2 methods only use information signals from 1 burst of waves of the same frequency (that is, scattered and reflected wave intensity information occurring during a single burst of waves of a particular frequency directed into the specimen), EMAR collects information signals over a longer period during which ultrasonic waves of the same frequency move back and forth many times inside the specimen, so that with EMAR even very faint signals will be amplified and contribute to determining the attenuation coefficient.

6. Conclusion

The attenuation coefficients of samples of SUS304 steel fabricated by the HIP method were measured

using EMAR at frequencies ranging from 1.1 to 5.4 MHz. It was found that an effective way of reducing attenuation due to diffraction was to use specimens cut from the samples to a size equal to the effective area of the EMAT. Further, in order to reduce attenuation due to contact between the EMAT and the specimen, the specimen was mounted on the EMAT with paper spacers interposed in such a way that the central portion of the specimen was clear of the EMAT. Thanks to this approach it was possible to measure a low attenuation coefficient value of 2.5×10^{-4} /microsecond for 1.1 MHz transverse waves in the sample that underwent HIP treatment at 170 MPa. And application of correction for attenuation due to electrical-mechanical connection yielded attenuation coefficients that increased in proportion to the frequency.

When the various methods were applied to HIP-processed specimens with various different void fractions, it was found that at frequencies above 4 MHz specimens with larger void fractions had higher attenuation coefficients, with a strong correlation between the two. At the same time it was established in the experiments that EMAR has a greater sensitivity to voids than the ultrasonic noise energy and ultrasonic spectroscopy methods currently in use.

The particular method of eliminating the effects of diffraction that was used in the present study—namely, using specimen strips cut from the samples—can not be used on real equipment. Nevertheless, we believe that with a little initiative it should be possible to apply the essential principles of such a method in measurement of real equipment. For instance, an EMAT with a large surface area could be used to excite ultrasonic waves on a broad portion of the target, together with a small EMAT to detect the ultrasonic attenuation of a narrow central portion within the excited portion.

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