

# Evaluation of stress corrosion cracks in metals

# by linear and nonlinear ultrasound

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# ABSTRACT

Nondestructive crack depth measurement is necessary for the evaluation of material strength in aged nuclear power plants. If a stress corrosion crack (SCC) is closed because of residual stress or by oxide film, the crack could be underestimated or overlooked. Thus far, to simulate SCCs generated in nuclear power plants, various formation methods were used. However, the difference in closure behavior between the formation methods has yet to be examined. Here we formed two kinds of SCCs in the specimens with a starting notch and a deep fatigue crack, respectively. Then we imaged them using linear phased array (PA) and subharmonic phased array for crack evaluation (SPACE). By comparing these results and the optically-observed cross section, we discussed the formation-method dependence of crack closure behavior.

# 1. INTRODUCTION

Fatigue cracks and stress corrosion cracks (SCCs)[1] have been generated in the important structure of nuclear power plants. Fatigue cracks are generated by repetitive load, whereas SCCs are generated by three factors which are material, tensile stress and corrosive environment. For instance, it has been known that, in recirculation pipe of actual plants, SCCs have been generated in heat affected zone loaded by residual stress in high temperature pressurized water.

To simulate them strictly, SCCs should be formed in an autoclave which can endure high temperature pressurized water. This is not easy because of high cost and long time. Thus far, various SCC formation methods have been used. For example, the method to extend SCC from the tip of a short fatigue crack has been reported by Brown [2] and Shoji [3]. The short fatigue crack was used only to generate SCC readily for examination of growth rate and growth threshold. On the other hand, the mechanism of the SCC generation has also been studied as follows. SCC extension is generally considered to be parallel to the direction of the plane of maximum tensile stress.[4] It has also been reported that SCC is not a mechanical fracture phenomenon directly defined by stress states but a dissolution phenomenon defined by the texture of materials, such as slip band, and the environment.[1] This relates to the fact that SCC generation originates in the dissolution of a path activated by a slip band in the high shear stress area around the crack tip.[5] However, the difference in extension mechanism and closure behavior between the formation methods has yet to be clarified, although this might strongly affect the measurement accuracy.

Here we formed two kinds of SCCs. One from a starting notch and another from a deep fatigue crack, respectively, with different stress fields around their tips. Then we imaged the SCCs and examined their closure behavior using SPACE. Finally, we discuss the difference between formation methods by the comparison of ultrasonic measurement results and optical observation of the cross sections.

### 2. LINEAR ULTRASONIC PHASED ARRAY (PA) AND SUBHARMONIC PHASED ARRAY FOR CRACK EVALUATION (SPACE)

To ensure the safety and reliability of nuclear power plants, the accurate measurement of crack depths is required for the evaluation of material strength of the structures. Crack depths can be measured by ultrasound if they are open, since the ultrasound is strongly scattered by the crack tip. However, ultrasonic inspection can sometimes underestimate and overlook fatigue cracks because some of them are closed owing to residual stress.[6] On the other hand, it has been known that SCC is open because it is generated and propagated only by tensile stress. For example, it has been reported that SCC depths were measured with standard deviation of less than 2 mm by laser ultrasound.[7] However, ultrasound is transmitted through the SCCs that are closed by oxide films. These crack closure could result in catastrophic accidents such as radiation leaks because the closed cracks may be propagated by large external stresses. To solve this problem, nonlinear ultrasound is the most promising means of evaluating closed cracks. Nonlinear ultrasound is based on the detection of nonlinear components, e.g., superharmonic waves (2f, 3f,...)[8,9] or subharmonic waves (f/2, f/3,...)[10,11], generated by the interaction of largeamplitude ultrasound with closed cracks, where f is the inputwave frequency. This phenomenon has been referred to as contact acoustic nonlinearity (CAN),[9] and a related measurement is often called nonlinear elastic wave spectroscopy (NEWS).[12] Among nonlinear components, subharmonic waves are specifically useful because of their excellent selectivity for closed cracks and high temporal resolution.[10,11] Thus far, we have developed a novel imaging method, namely, the subharmonic phased array for crack evaluation (SPACE)[10,11], on the basis of subharmonic waves and a phased array algorithm. We have demonstrated its performance in closed fatigue cracks and the closed cracks generated in material manufacturing process.

Figure 1 shows the schematic diagrams of linear ultrasonic phased array (PA) for open-crack imaging and nonlinear ultrasonic phased array for closed-crack imaging, SPACE developed by us. Phased array can steer ultrasound to specific directions and can focus it to a specific point by exciting each

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element of array transducer following proper delay law. Generally, PA utilizes an array transducer excited by low-voltage pulses. This can image open cracks. In SPACE, a LiNbO3 single-crystal transmitter with a polyimide wedge is used to generate intense ultrasound, and a phased array sensor is used as a receiver for focusing on reception.[13] By inputting intense ultrasound, the scattering of fundamental and subharmonic waves occurs at the open and closed parts of cracks, respectively. The scattered waves received by the array sensor are digitally filtered at fundamental and subharmonic frequencies. Then, they are phase-matched following the delay laws. Fundamental image (FA) and subharmonic image (SA) can indicate the open and closed parts of cracks, respectively.[11] Here both PA and FA can image open cracks, whereas the following points are different. PA has a high spatial resolution because of focussing both on transmission and reception, and it is easy to mechanically scan. The present implementation of SPACE does not have dead zone caused by the effect of excitation signal because the transmitter and the receiver are separated, but the mechanical scan is not easier than PA because of using two transducers.



Fig.1 Schematic diagrams of experiment to image cracks:(a) Linear phased array and (b) Nonlinear phased array (SPACE).

### 3. SCC specimens

### 3.1 Formation of SCCs from starting notches

We formed SCCs from starting notches in specimens which are butt weld composed of weld metal made of Nibased alloy, commercially available as Inconel alloy 600, and a base material made of an austenitic stainless steel type 316L. As shown in Fig.2(a), we formed SCCs from starting notches which are perpendicular to weld lines. To control the SCC depths, we applied a following method. First, we dug a volume with a depth slightly larger than target depth in weld metal by electric discharge machining (EDM). Then, the volume was molded with another Inconel alloy 600 with higher carbon content where SCC could be generated readily. The starting notches of 0.2 mm width and 0.5 mm depth were fabricated at the center of the molded parts by EDM. Their tips are not very sharp in contrast to those of deep fatigue cracks. Subsequently, the thermally-expanded specimens were welded into a jig, and then they were cooled to room temperature. Thus a tensile stresses in the direction perpendicular to each starting notches were loaded because of thermal contraction. The tensile stresses were measured to be 250-300 MPa on strain gauges bonded on surfaces of the specimens. Under this condition, the local areas with starting notches were exposed in tetrathionate solution which has been often used for SCC formation. Thus, we formed SCCs with different depths from the starting notches. After forming the SCCs, the staring notches were eliminated.

# 3.2 Formation of SCC from a deep fatigue crack

The method to extend SCCs from the tip of short fatigue crack has been reported by Brown [2] and Shoji [3]. The short fatigue cracks were used only to generate SCC readily for examination of growth rate and growth threshold. Here we proposed a method to fabricate a deep SCC for a short time. First, we formed a fatigue crack with a depth of approximately 10 mm in a sensitized austenitic stainless steel specimen (SUS304 sensitized at 600°C for 4 h) by a threepoint bending fatigue test with maximum and minimum stress intensity factors  $K_{\text{max}}=28$  MPa·m<sup>1/2</sup> and  $K_{\text{min}}=0.6$ MPa $\cdot$ m<sup>1/2</sup>. Subsequently, we extended an SCC from the tip of the deep fatigue crack using an SCC apparatus, as shown in Fig.2(b). This apparatus was designed to immerse the entire fatigue crack specimen in a cell with a corrosive environment under a static bending load with a maximum of 100 kN. The temperature of the corrosive environment and the load are controlled with a thermostat and a hydraulic pump, respectively. The extension of SCC on the specimen surface can be monitored in situ visually. In this study, the corrosive environment was a solution of 30 wt % MgCl<sub>2</sub> at 90°C, and a nominal bending stress of 124 MPa was applied to the crack for 650h. Thus, we formed an SCC more than 10 mm depth for relatively short time.



Fig.2 SCC testing: (a) INCONEL 600 weld metal with starter notch subjected to tensile stress in tetrathionate solution and (b) SUS304 with fatigue crack subjected to 3-point bending stress in  $MgCl_2$  solution.

#### 4. Experimental results

# 4.1 Imaging of SCCs extended from starting notches

To examine the closure behavior, first, we imaged SCCs extended from starting notches using PA and SPACE. Here we used an array censor with a center frequency of 5 MHz, 32 elements at a maximum and an element pitch of 0.5mm for PA. In SPACE measurement, LN transmitter with a center frequency of 7 MHz and that array sensor were used. As an example, imaging results of an SCC are shown in Fig.3(a). In PA and FA, images of SCC were obtained and the crack depths were almost the same although there are difference of images caused by different propagation paths in weld metal with strong anisotropy. In contrast, the SCC was not imaged in SA. These results suggest that the SCC was dominantly open. This trend was also shown in differentdepth SCCs. Thus, the crack depths were measured by PA which has same propagation paths in transmission and reception.

### 4.2 SCC formed from the tip of deep fatigue crack

To precisely image the SCC extended from the tip of deep fatigue crack, we applied SPACE at three positions differ in the crack-length direction. The input signal was a three-cycle burst of a 7 MHz sinusoidal wave with 10.3  $m_{p-p}$  amplitude, where the displacement amplitude was measured at crack positions in another specimen cut from the same material by laser interferometry.[10] We used 64 elements in an array

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transducer with a center frequency of 5 MHz to receive both fundamental (7 MHz) and subharmonic (3.5MHz) waves. We succeeded in visualizing various crack images at each position. The distribution of the crack response in the images was not only in the vertical direction but also in the horizontal direction. This result shows that the SCC did not linearly extend but was complexly branched and had open and closed parts. As an example, fundamental and subharmonic images are shown in Fig.3(b). Note that the crack tips were deeper in the subharmonic images than in the fundamental images. It was similar at all positions. This suggests that the tips of the SCC were closed.



Fig.3 Imaging SCCs by SPACE :(a) SCC extended from starting notch and (b) SCC extended from the tip of deep fatigue crack.

#### 5. Optical observation of cross section and examination of measurement accuracy

#### 5.1 SCCs formed from starting notches

To discuss the measurement accuracy, we compared the depths measured by PA and reference crack depths, as shown in Fig.4. An optically-observed cross section is shown in Fig.5(a).As reference crack depths are optically-observed crack depths on the cross section or target crack depths. Consequently, all the SCCs were measured without large underestimation.Thus, the SCCs formed from starting notches could be measured with sufficient measurement accuracy by PA.

# 5.2 SCC extended from the tip of deep fatigue crack

To optically examine the depths and distributions of the SCC and to compare them with the SPACE images, the specimen was sliced in seven thin plates with a 25 mm<sup>2</sup> area around the crack. The cross sections were polished, and etched in 10 wt % oxalic acid for 30 s. The SCC was complexly branched from the tip of the fatigue crack. Here, we examined the measurement accuracy of SPACE quantitatively. The relationship between optically and SPACE-measured crack depths is shown in Fig.4. The crack depths in the fundamental images were underestimated for all positions, whereas the subharmonic images succeeded in preventing the underestimation.



Fig.4 Mesurement accuracy.

### 6. Discussion of crack closure behavior depending on the formation methods with starting notches and deep fatigue crack

We found that the SCCs formed from the starting notches were open. This agrees with the previous results [5]. In contrast, we found that SCC extended from the tip of deep fatigue crack was partly closed. Although two SCCs were formed in different chemical solutions, it has not been reported that the closure behavior differs depending on the chemical solutions for the acceleration test. Here we discussed the closure behavior with respect to stress condition around SCC origin with starting notches and deep fatigue crack.

In Fig.5(b), interestingly, the SCC extension directions were primarily oblique to the fatigue crack extension directions. This result can be explained by assuming that the SCC generation originates in the dissolution of a path activated by a slip band in the high shear stress area around the crack tip.[5] Although SCC extension is generally considered to be parallel to the direction of the plane of maximum tensile stress,[4] this direction does not agree with the present observation. Thus, the present finding is consistent with the idea[1] that SCC is not a mechanical fracture phenomenon defined by the texture of materials, such as slip band, and the environment.

Furthermore, the tendency of crack extension length also differed between the fatigue crack and the SCC. The distribution of the fatigue crack depths had a maximum in the central part. This is reasonable since the central and edge parts are under plane strain and plane stress conditions, respectively.[14] In contrast, the SCC extension lengths were slightly larger in the edge parts than in the central part. This also suggests that the SCC extension was concerned with the shear stress field around the crack tip, since the shear stress is higher in the edge parts than in the central part. Thus, in contrast to starting notches of which the tips are not very sharp, the SCC extended from the tip of deep fatigue crack might be strongly influenced by the shear stress field around the tip of deep fatigue crack.



Fig.5 Optically observations of SCC on cross section:(a) SCC extended from starting notch and (b) SCC extended from the tip of deep fatigue crack .

### 7. Conclusions

To examine the extension and closure behavior of SCC depending on the formation methods, we formed two SCCs extended from starting notches and a deep fatigue crack. Then we measured the SCCs using SPACE. Consequently, we found that the SCCs extended from starting notches were dominantly open. On the other hand, we found that SCC extended from a deep fatigue crack was interestingly partly closed. To discuss the difference, we focused on the influence on the stress condition around the SCC origin. By comparing the SPACE-measured images and optically-observed cross section, we found that the closure behavior might be influenced by the shear stress field around the tip of deep fatigue crack. In future, we would examine the extension and closure behavior in the specimens made of the same material with starting notches and deep fatigue crack, in the same corrosive environment.

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### References

[1] M. Takemoto, *Genba Gijutsusha notameno Oryoku Fu-shokuware* (Stress Corrosion Cracking for Engineers) (Nihon Yosha Kogaku Kenkyujo, Osaka, 2007) p.1 [in Japanese]

[2] B. F. Brown, C. D. Beachem, "A study of the stress factor in corrosion cracking by use of the pre-cracked cantilever beam specimen", Corrosion Science, vol.5, pp.745-750 (1965).

[3] Z. Lu, T. Shoji, Y. Takeda, A. Kai, Y. Ito, "Effects of loading mode and temperature on stress corrosion crack growth rates of a cold-worked type 316L stainless steel in oxygenated pure water", Corrosion, vol.63, pp.1021-1032 (2007)

[4] T. P. Hoar, "Stress-Corrosion Cracking", Corrosion, vol.19, pp.331t-338t (1963)

[5] T. J. Smith, R. W. Staehle, "Role of Slip Step Emergence in the Early Stages of Stress Corrosion Cracking in Face Centered Iron-Nickel-Chromium Alloys", Corrosion, vol.23, pp.117-129 (1967)

[6] W. Elber, "Fatigue crack closure under cyclic tension",

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Engng. Fract. Mech., vol.2, no.1, pp.37-45, July 1970.
[7] Makoto Ochiai, "Laser-Ultrasonics and its Applications to Non destructiveTesting", HIHAKAIKENSA, vol.57, No.1 jan. (2008)

[8] O. Buck, W. L. Morris, and J. M. Richardson, "Acoustic harmonic generation at unbonded interfaces and fatigue cracks", Appl. Phys. Lett., 33, pp.371-373, 1978.

[9] I. Solodov and C. A. Vu, "Non-linear SAW reflection: experimental evidence and NDE applications Ultrasonics", Acoust. Phys., vol.39, pp.476-479, 1993.

[10] M. Akino, T. Mihara and K. Yamanaka, "Fatigue crack closure analysis using nonlinear ultrasound", Rev. Prog. QNDE, vol.23, pp.1256-1263 (2004)

[11] Y. Ohara, S. Yamamoto, T. Mihara and K. Yamanaka, "Ultrasonic Evaluation of Closed Cracks Using Subharmonic Phased Array", Jpn. J. Appl. Phys., vol.47, no.5, pp.3908-3915, May 2008.

[12] K. E. –A. V. D. Abeele, P. A. Johnson, and A. Sutin, "Nonlinear Elastic Wave Spectroscopy (NEWS) Techniques to Discern Material Damage, Part I: Nonlinear Wave Modulation Spectroscopy (NWMS)", Res. Nondestr. Eval. vol.12, pp.17-30 (2000)

[13] T. L. Szabo, Diagnostic Ultrasound Imaging: Inside Out (Academic Press, New York, 2004) pp.171-203

[14] H. Okamura, *Senkei Hakai Rikigaku Nyumon* (Introduction of Linear Fracture Mechanics) (Baifukan, Tokyo, 1976) [in Japanese]