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Article

Corrosion Fatigue of Austenitic Stainless Steels for Nuclear Power Engineering

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Abstract: Significant structural steels for nuclear power engineering are chromium-nickel austenitic stainless steels. The presented paper evaluates the kinetics of the fatigue crack growth of AISI 304L and AISI 316L stainless steels in air and in corrosive environments of 3.5% aqueous NaCl solution after the application of solution annealing, stabilizing annealing, and sensitization annealing. Comparisons were made between the fatigue crack growth rate after each heat treatment regime, and a comparison between the fatigue crack growth rate in both types of steels was made. For individual heat treatment regimes, the possibility of the development of intergranular corrosion was also considered. Evaluations resulted in very favourable corrosion fatigue characteristics of the 316L steel. After application of solution and stabilizing annealing at a comparable ΔK level, the fatigue crack growth rate was about one half compared to 304L steel. After sensitization annealing of 316L steel, compared to stabilizing annealing, the increase of crack growth rate during corrosion fatigue was slightly higher. The obtained results complement the existing standardized data on unconventional characteristics of 304L and 316L austenitic stainless steels.

Keywords: austenitic stainless steel; heat treatment; corrosion fatigue; fatigue crack growth rate; intergranular corrosion

1. Introduction

Chromium-nickel austenitic stainless steels are used in nuclear power engineering to a significant extent.

304L and 316L austenitic stainless steels represent important structural materials for the construction of primary circuit components and internal in-building of light water nuclear power plants, and 316L also for building components for nuclear power systems with fast reactors [1].

A certain disadvantage of these types of steel is their relatively low strength level achieved after annealing. To achieve a higher level of strength of these steels, it is necessary to apply appropriate techniques based on the combination of mechanical and thermal processing, which ensure the achievement of a desired level of strength parameters, as well as their stabilization.

From the perspective of a comprehensive evaluation of austenitic steels and nuclear power plants operating conditions, it is also important to study fatigue stress—especially the kinetics of fatigue crack growth, including superposition of the effect of external environment [2,3]. The initiation and stable development of a crack occurs only if the state of stress and environmental and material characteristics reach a critical level [4].

For corrosion fatigue, we cannot think of the fatigue limit, because the corrosion cross-section of the component is shrinking all the time. The fatigue curve with the decreasing tensile stress and with the increasing number of cycles has a steadily downward course, so that even below the fatigue limit fracture occurs. The slower the tension cycles, the greater the possible impact of the corrosion environment on reducing the number of cycles to fracture (i.e., on the service life of the components) [5].

Corrosion fatigue during cyclic stress is characterized by the existence of a threshold value K_{Iscc} in the area of the validity of Paris's Law [6] $da/dN = C\cdot(\Delta K)^m$, where da/dN is the fatigue crack growth rate, ΔK is the stress intensity factor range at the crack tip, and C and m are material constants. At $\Delta K > K_{ISCC}$, the fatigue crack growth rate compares to the growth rate in the air. In this area, the fatigue crack growth rate largely depends on the frequency and cycle asymmetry [7]. Cracks generated during corrosion fatigue are usually transgranular with characteristic branching and are perpendicular to the applied tensile stress [4,8]. Other important factors that affect the rate of fatigue crack growth are, for example, dislocation substructure, deformation induced by phase transformation in the plastic zone adjacent to the top of the fatigue crack, residual stresses, temperature, etc.

2. Materials and Experimental Technique

The fatigue crack growth kinetics were evaluated for the above-mentioned 304L and 316L austenitic stainless steels.

The evaluation was performed using the material taken from 25 mm-thick sheets which were operationally heat treated by solution annealing 1050 °C/4 h/water.

The chemical composition of the studied steels is shown in Table 1.

Table 1. Chemical composition of steels /wt. %.

Steel	C	Mn	Si	P	S	Cr	Ni	Mo
304L	0.03	1.25	0.31	0.033	0.018	18.07	11.74	-
316L	0.03	0.80	0.37	0.033	0.018	17.86	12.60	2.90

The evaluation itself was performed for three variants of heat treatment:

(a) Solution annealing: 1050 °C/1 h/water,
(b) Stabilizing annealing: 1050 °C/1 h/water + 850 °C/4 h/water,
(c) Sensitization annealing: 1050 °C/1 h/water + 600 °C/24 h/water.

When evaluating the kinetics of fatigue crack growth according to Paris-Erdogan [8], load cycles in both studied steels had sinusoidal character, and stress ratio $R = 0$ at the selected frequency of 1 and 6 Hz. Testing was carried out both in air and under the superposition effect of the external environment. Aqueous NaCl solution (3.5%) was chosen as a corrosive medium. Evaluation was carried out on flat samples 3 mm thick and 60 mm wide with a central crack of 6 mm in length. Fatigue pre-cracking was used in accordance with ASTM E399. The K level used for pre-cracking each specimen did not exceed two thirds of the starting K-value for the environmental exposure. Kinetics of the fatigue cracks' growth was examined using the INOVA electrohydraulic machine (INOVA Prague Ltd., Prague, Czech Republic). Fracture surfaces were analysed by the Quanta FEG 450 scanning electron microscope (FEI Czech Republic Ltd., Brno, Czech Republic) with the TRIDENT-APEX 4 micro analytical system (EDAX Inc., Mahwah, NJ, USA).

3. Results and Discussion

Figures 1–3 show examples of the basic microstructure of 316L steel after all three variants of annealing. Microstructural analysis was performed using an electrolytic etching in 10% oxalic acid solution. The images suggest the possibility of depletion of chromium at grain boundaries, or annealing twins in the austenite matrix, and they provide basic information on potential susceptibility to the development of intergranular corrosion. Figure 2 shows that the stabilization annealing is followed by discontinuous precipitation of $M_{23}C_6$ carbides, and thus the occurrence of localized areas depleted of chromium. The potential danger of intergranular corrosion in this case is negligible. Figure 3 confirms intense continuous precipitation of $M_{23}C_6$ carbide in long-time sensitized samples, indicating susceptibility to intergranular corrosion. Identical characteristics were also found in 304L steel.

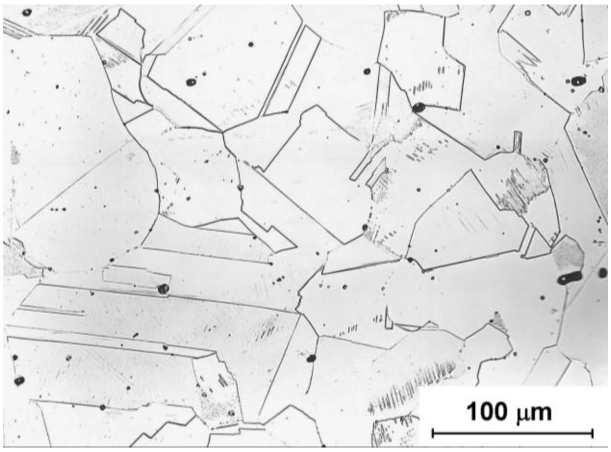

Figure 1. Microstructure of the 316L steel after solution annealing by regime 1050 °C/1 h/water.

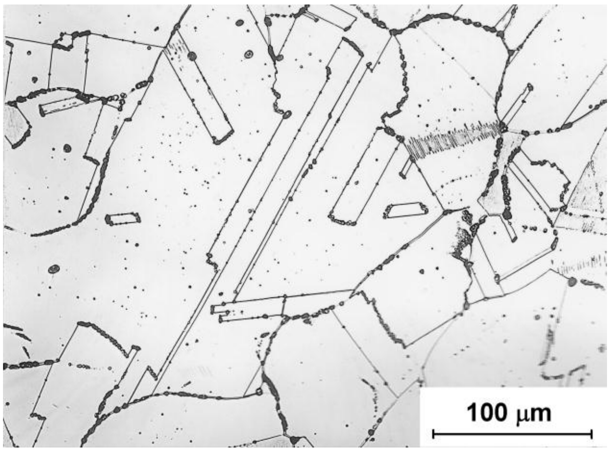

Figure 2. Microstructure of the 316L steel after solution annealing by regime 1050 °C/1 h/water and stabilization annealing by regime 850 °C/4 h/water.

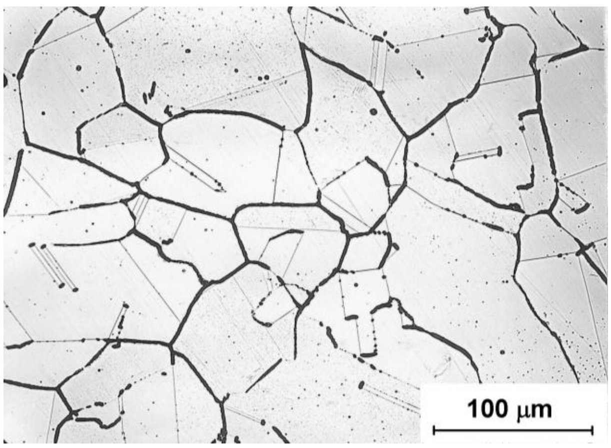

Figure 3. Microstructure of 316L steel after solution annealing by regime 1050 °C/1 h/water and sensitization annealing by regime 600 °C/24 h/water.

Fractographical analysis showed a higher incidence of brittle cleavage disruption on fracture surfaces of corrosion fatigue tests. In many cases, this type of disruption was accompanied by occurrence of intergranular areas, especially at lower levels of ΔK and in the evaluation of corrosion fatigue under superposition environmental effects (see Figure 4). As is known, the negative effect of harmful elements in steel (e.g., phosphorus, sulphur, and generally, other elements of subgroup IV.a to VI.A of the periodic table) lies in their ability to segregate on large-angle grain boundaries, which results in a reduction of the cohesive strength and the formation of low-energy intergranular fractures [9]. Corrosion fatigue is precisely one of the degradation processes in which the formation of intergranular fractures through micro-segregation effect occurs [10].

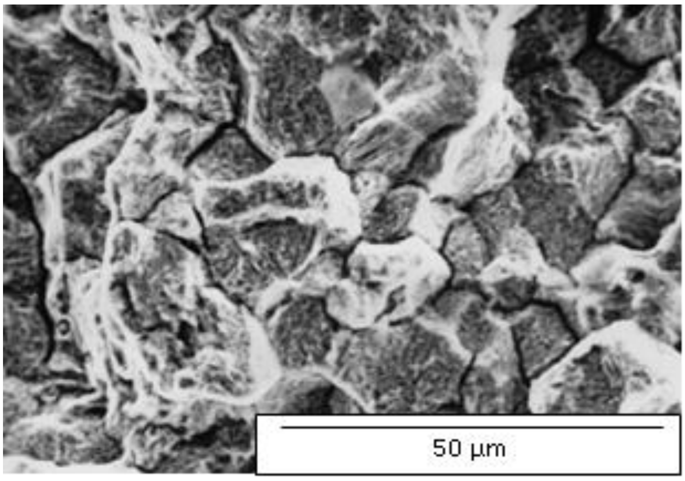

Figure 4. Intergranular brittle fracture of the 316L steel.

Figure 5 summarizes the plotted kinetic dependence of the development of fatigue cracks $\Delta a/\Delta N$ at ΔK of both studied steels after solution annealing.

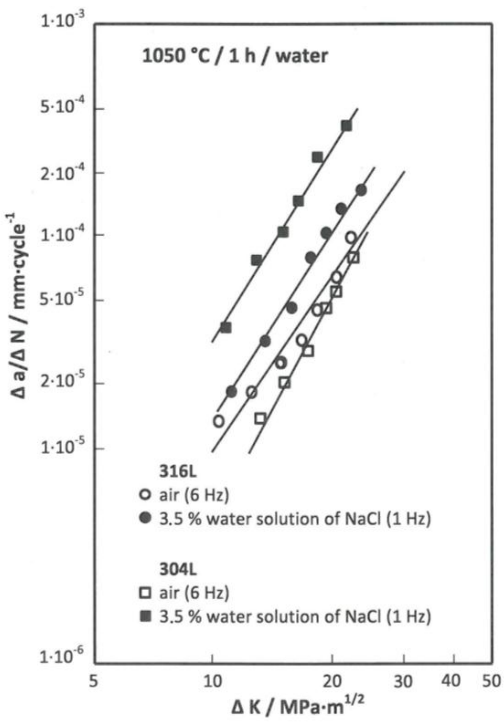

Figure 5. Kinetics of fatigue crack growth for 304L and 316L steels after solution annealing.

For the 304L steel, after the application of solution annealing, the equation for evaluating corrosion fatigue in the air (1) and the equation in the specified corrosive environment (2) were determined.

$$\Delta a/\Delta N = 1.5 \times 10^{-9}\ (\Delta K)^{3.484} \quad (1)$$

$$\Delta a/\Delta N = 2.84 \times 10^{-8}\ (\Delta K)^{3.058} \quad (2)$$

For the 316L steel, the equation for the evaluation of corrosion fatigue in the air (3) and the equation for evaluation in the chosen environment (4) were determined after solution annealing.

$$\Delta a/\Delta N = 1.67 \times 10^{-8}\ (\Delta K)^{2.7648} \quad (3)$$

$$\Delta a/\Delta N = 1.25 \times 10^{-8}\ (\Delta K)^{3.021} \quad (4)$$

$$\Delta a/\Delta N = 2.7 \times 10^{-9}\ (\Delta K)^{3.456} \quad (5)$$

$$\Delta a/\Delta N = 1.46 \times 10^{-8}\ (\Delta K)^{3.292} \quad (6)$$

For the 316L steel, the kinetic equation for evaluating fatigue properties in the air (7) and the equation for evaluating the corrosion fatigue in the chosen environment (8) were determined after the application of stabilizing annealing.

$$\Delta a/\Delta N = 2.7 \times 10^{-9}\ (\Delta K)^{3.456} \quad (7)$$

$$\Delta a/\Delta N = 1.5 \times 10^{-9}\ (\Delta K)^{3.796} \quad (8)$$

Figure 6 and Equations (5) and (7) show that the kinetic characteristics of both studied steels when evaluated in the air are identical.

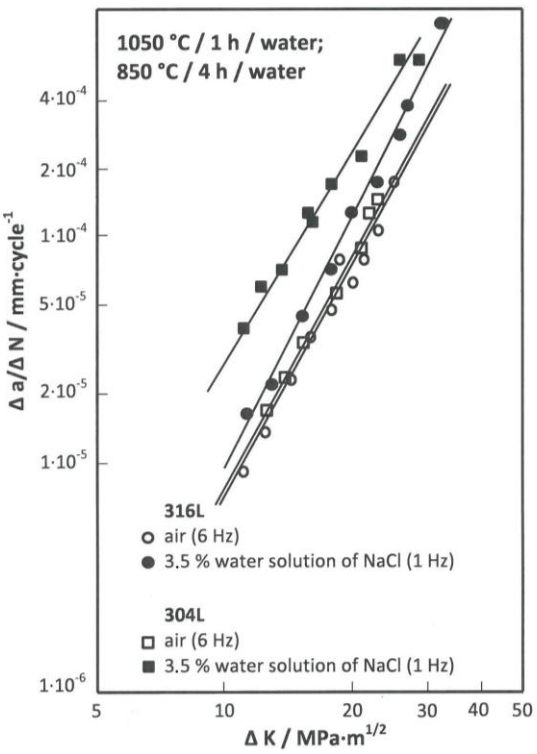

Figure 6. Kinetics of fatigue crack growth for 304L and 316L steels after stabilization annealing.

Kinetics of the development of fatigue cracks in both austenitic steels after the application of sensitization annealing (Figure 7) was also examined. For the 304L steel, kinetics of the fatigue crack growth for the evaluation of fatigue properties in the air after this treatment can be described by Equation (9), and in the chosen environment by Equation (10).

$$\Delta a/\Delta N = 3.28 \times 10^{-9}\ (\Delta K)^{3.530} \tag{9}$$

$$\Delta a/\Delta N = 4.86 \times 10^{-8}\ (\Delta K)^{2.724} \tag{10}$$

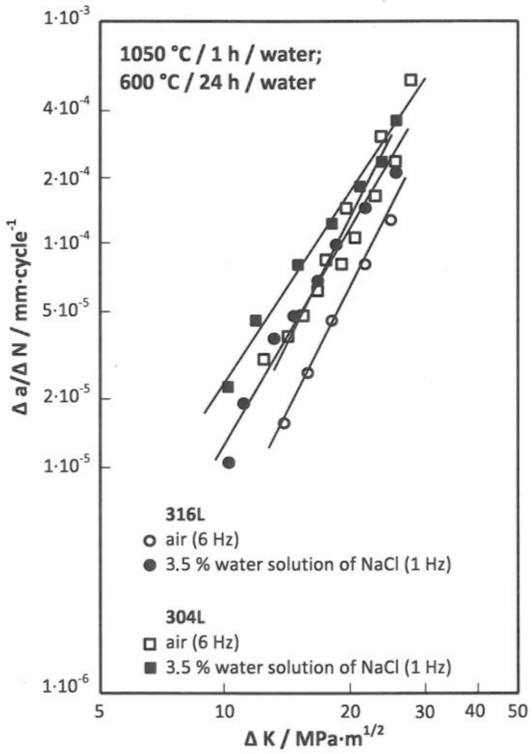

Figure 7. Kinetics of fatigue crack growth for 304L and 316L steels after sensitization annealing.

For the 316L steel, kinetic equations for evaluating the corrosion fatigue in the air (11) and for evaluating the fatigue characteristics in the selected corrosive environments (12) were determined after the application of sensitization annealing.

$$\Delta a/\Delta N = 1.78 \times 10^{-9}\ (\Delta K)^{3.505} \tag{11}$$

$$\Delta a/\Delta N = 8.95 \times 10^{-9}\ (\Delta K)^{3.170} \tag{12}$$

The comparison of the kinetic dependences shown in Figures 5–7 indicates that in the 304L steel, susceptibility to corrosion fatigue in the corrosive environment is significantly reflected. For example, after the application of annealing solution, the rate of fatigue crack growth increased at the level $\Delta K = 15$ MPa·m$^{1/2}$ due to the standard state by about 3/4 of the order, and at $\Delta K = 20$ MPa·m$^{1/2}$ from the value of 5×10^{-5} mm/cycle corresponding to a standard condition to about 3.5×10^{-4} mm/cycle. In the other two variants of heat treatment, the stated increase in speed decreased with respect to the standard condition. The decreased difference between the compared levels of fatigue crack growth rate is related to the fact that for the standard evaluation conditions after application of solution annealing, very low rate of fatigue crack growth was achieved, while for standard conditions, after application of stabilizing annealing or sensitization annealing, the rate of fatigue crack growth was higher.

Figures 5–7 indicate favourable characteristics of corrosion fatigue in the 316L steel. After the application of solution annealing, the increase in growth rate for this steel compared to 304L steel at a comparable level of ΔK is roughly one half. Similar is true after the application of stabilizing annealing (Figure 3). After sensitization annealing, as compared to stabilizing annealing, the increase the rate of crack growth during corrosion fatigue was slightly higher (Figure 4).

Results of the fatigue crack growth rate are in accordance with the general conclusions [11] that in the field of high rate, the sudden final fracture is not strongly affected by corrosive environment. In the field of low and medium fatigue crack growth rate, an increase in rate and lower threshold values can be observed due to the presence of a corrosive environment.

4. Conclusions

In the context of the presented work, the kinetics of the fatigue crack growth of 304L and 316L austenitic stainless steels in air and under corrosion fatigue in 3.5% aqueous NaCl solution was evaluated after three modes of heat treatment; namely, after solution annealing, stabilizing annealing, and sensitization annealing at different frequencies of loading.

Evaluations resulted in very favourable corrosion fatigue characteristics of the 316L steel. After the application of solution and stabilizing annealing at a comparable ΔK level, the rate of fatigue crack growth compared to 304L steel was about one half. After sensitization annealing of 316L steel, in comparison with stabilizing annealing, the increase of crack growth rate during the corrosion fatigue was slightly higher.

The obtained results complement the existing standardized data of unconventional characteristics of 304L and 316L austenitic stainless steels [12]. Knowledge of fatigue crack growth rate data in air and in corrosive environments is essential to ensure the safety and reliability of relevant components of nuclear power plants manufactured from these types of steels. In terms of safety against the stable corrosion fatigue crack growth rate, the development of resistance to intergranular corrosion is a major technological step.

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Conflicts of Interest: The authors declare no conflict of interest.

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