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Some fundamental aspects of thermally activated processes involved in stress corrosion cracking in high temperature aqueous environments

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ABSTRACT

Complex mechano-chemical interactions at material/solution interfaces under elevated temperatures are involved in the stress corrosion cracking (SCC) of structural materials in light water reactor environments. Elucidating the thermally activated processes of SCC is of great importance for the mechanistic understanding and quantitative predictions. Steady state stress corrosion crack growth rates have been measured and used to calculate apparent activation energies under various test conditions. The observed apparent activation energies have been analyzed, based on the concept that multiple sub-processes with different thermally activated rate-controlling steps contribute simultaneously to the SCC crack growth.

1. Introduction

Temperature has been proved to be one of the key engineering parameters in the stress corrosion cracking (SCC) of structural materials in light water reactor (LWR) environments [1–4]. Testing at high temperature has been used in the accelerated crack growth rate (CGR) tests for nickel-base alloys and weld metals in simulated pressurized water reactor (PWR) primary water environments [3]. It is also expected that more details of the cracking processes could be obtained from the response of the cracking behavior to temperature. Several types of temperature-dependent crack growth behavior have been observed for sensitized 304 stainless steels (SS) in simulated boiling water reactor (BWR) environments [2,5]. It has been observed that CGRs of nickel based alloys and weld metals in simulated PWR primary water environments generally increase with increasing temperature [3]. Recently the effects of temperature on the CGR of low-carbon SS in hydrogenated [6] and oxygenated pure water have been studied [7,8]. The complexity of the analysis of the effect of temperature on physicochemical processes has been discussed by Santarini [9]. There were some analyses of the thermal activation of SCC [3,10] or chemical–mechanical systems [11]. The effects of temperature on SCC growth of several typical austenitic alloys in simulated LWR environments are presented. Using fracture mechanics specimens and in situ monitoring, steady state CGR at various temperatures are measured and used both to assist in interpreting the effects of temperature on SCC growth and aid the understanding some fundamental aspects of SCC processes.

2. Phenomenological description of the temperature-dependent crack growth behavior

Laboratory results show that there are mainly two types of temperature-dependent SCC growth behavior for structural materials in simulated light water reactor environments, namely, type T-A and type T-B [1–5,12–19], Fig. 1. CGR increases monotonically with temperature within type T-A, i.e., $(\partial \mathrm{CGR}/\partial T) > 0$, Fig. 1(a). There are two subtypes for type T-A, i.e., T-A$_\mathrm{I}$ with $(\partial^2 \mathrm{CGR}/\partial T^2) > 0$ and T-A$_\mathrm{D}$ with $(\partial^2 \mathrm{CGR}/\partial T^2) < 0$. There is a maximum CGR at a threshold temperature T_th in type T-B, where $(\partial \mathrm{CGR}/\partial T) > 0$ for $T < T_\mathrm{th}$, $(\partial \mathrm{CGR}/\partial T) = 0$ at $T = T_\mathrm{th}$, or where $(\partial \mathrm{CGR}/\partial T) < 0$ for $T > T_\mathrm{th}$. Both Type T-A and Type T-B have been reported for SCC growth of sensitized 304SS in simulated BWR environments [2,5]. Experimental data show that SCC growth of Ni-based alloy 600 and weld metals, such as alloys 182/82 in simulated PWR primary water is thermally activated and follows a Type T-A$_\mathrm{I}$ temperature-dependence [12–18]. Similarly, recent results show that CGR of cold worked (CW) low-carbon SS in high temperature pure water is thermally activated and follows a Type A$_\mathrm{I}$ temperature-dependence [6–8]. An apparent activation energy (E_aae) is often used to quantify the effect of temperature on CGR. There are several ways for measuring and calculating E_aae from the experimental CGR data, either based on maintaining the bulk water chemistry constant or based on maintaining the (bulk) electrochemical parameters such as electrochemical potential (ECP) constant at different temperatures, Fig. 2. The ECP-dependent crack growth behavior has been addressed [20,21],

$$E_{\mathrm{aae,I}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\mathrm{ECP}_1, \mathrm{DO}_a, T_1)}{\mathrm{CGR}(\mathrm{ECP}_2, \mathrm{DO}_a, T_2)}\right), \tag{1}$$

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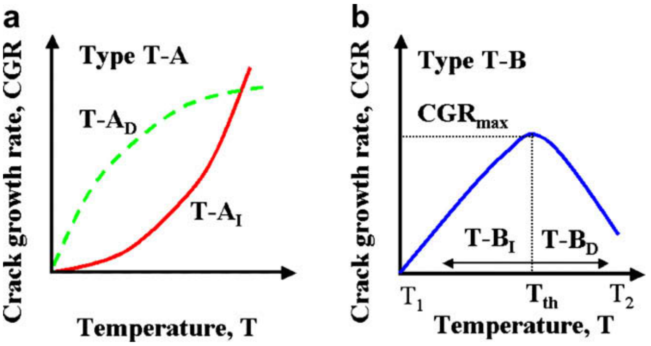

Fig. 1. Schematic representation of several types of temperature-dependent crack growth behavior. (a) Monotonic increase and (b) increase with a maximum.

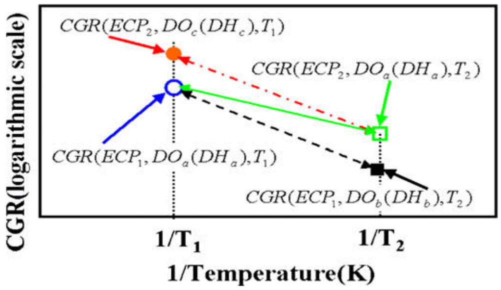

Fig. 2. Schematic of methods for measuring and calculating the apparent activation energy for CGR.

$$E_{\mathrm{aae,II}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\mathrm{ECP}_1, \mathrm{DO}_a, T_1)}{\mathrm{CGR}(\mathrm{ECP}_1, \mathrm{DO}_b, T_2)}\right), \tag{2}$$

$$E_{\mathrm{aae,III}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\mathrm{ECP}_2, \mathrm{DO}_c, T_1)}{\mathrm{CGR}(\mathrm{ECP}_2, \mathrm{DO}_a, T_2)}\right), \tag{3}$$

where $\mathrm{ECP}_1 = \mathrm{ECP}(\mathrm{DO}_a, T_1) = \mathrm{ECP}(\mathrm{DO}_b, \mathrm{T}_2)$, and $\mathrm{ECP}_2 = \mathrm{ECP}(\mathrm{DO}_a, T_2) = \mathrm{ECP}(\mathrm{DO}_c, T_1)$.

The calculated E_{aae} does not have a unique value based on the constant ECP criteria. Simply selecting different standard conditions or ECPs can result in different values of E_{aae}, i.e., $E_{\mathrm{aae,II}}$ at ECP_1 and $E_{\mathrm{aae,III}}$ at ECP_2, Fig. 2. Similarly, in hydrogenated water without DO or with a negligible amount of DO, the following holds true:

$$E_{\mathrm{aae,IV}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\mathrm{ECP}_1, \mathrm{DH}_a, T_1)}{\mathrm{CGR}(\mathrm{ECP}_2, \mathrm{DH}_a, T_2)}\right), \tag{4}$$

$$E_{\mathrm{aae,V}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\mathrm{ECP}_1, \mathrm{DH}_a, T_1)}{\mathrm{CGR}(\mathrm{ECP}_1, \mathrm{DH}_b, T_2)}\right), \tag{5}$$

$$E_{\mathrm{aae,VI}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\mathrm{ECP}_2, \mathrm{DH}_c, T_1)}{\mathrm{CGR}(\mathrm{ECP}_2, \mathrm{DH}_a, T_2)}\right). \tag{6}$$

A characteristic parameter, $\Delta\mathrm{ECP}_{\mathrm{NiO/Ni}}$, as defined in Eq. (14), has been used as a criterion by Morton et al. [17] in the determination of E_{aae} for primary water stress corrosion cracking (PWSCC) growth rates of nickel based alloys and weld metals, Eqs. (8) and (9).

$$\Delta\mathrm{ECP}_{\mathrm{Ni/NiO}}(\mathrm{DH}, T) = \mathrm{ECP}(\mathrm{DH}, T) - \mathrm{ECP}_{e,\mathrm{Ni/NiO}}(\mathrm{DH}, T), \tag{7}$$

$$E_{\mathrm{aae,VII}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\Delta\mathrm{ECP}_{\mathrm{Ni/NiO},1}, \mathrm{DH}_a, T_1)}{\mathrm{CGR}(\Delta\mathrm{ECP}_{\mathrm{Ni/NiO},1}, \mathrm{DH}_d, T_2)}\right), \tag{8}$$

$$E_{\mathrm{aae,VIII}} = R\left(\frac{T_1 \cdot T_2}{T_1 - T_2}\right) \ln\left(\frac{\mathrm{CGR}(\Delta\mathrm{ECP}_{\mathrm{Ni/NiO},2}, \mathrm{DH}_e, T_1)}{\mathrm{CGR}(\Delta\mathrm{ECP}_{\mathrm{Ni/NiO},2}, \mathrm{DH}_b, T_2)}\right), \tag{9}$$

where $\mathrm{ECP}_{e,\mathrm{NiO/Ni}}$ is the equilibrium potential for the Ni/NiO reaction. The DH is adjusted to achieve $\Delta\mathrm{ECP}_{\mathrm{Ni/NiO}}(\mathrm{DH}_a, T_1) = \Delta\mathrm{ECP}_{\mathrm{Ni/NiO}}(\mathrm{DH}_d, T_2)$ in Eq. (8), and $\Delta\mathrm{ECP}_{\mathrm{Ni/NiO}}(\mathrm{DH}_b, T_2) = \Delta\mathrm{ECP}_{\mathrm{Ni/NiO}}(\mathrm{DH}_e, T_1)$ in Eq. (9).

2.1. Austenitic alloys in hydrogenated high temperature water

2.1.1. PWSCC of Ni-base alloy 600 and weld metals

It is generally accepted that crack growth of nickel based alloy 600 and weld metals in simulated PWR primary water are thermally activated [3]. Experimental data generally show a type T-A$_\mathrm{I}$ temperature-dependent relationship, with relatively consistent activation energies within each set of data [1,3,12–17]. The temperature effect on CGR has been included in the disposition curves for PWSCC CGR of thick plate alloy 600 (MRP-55) [22] and for weld metal alloys 182, 132 and 82 (MRP-115) [23,24]. A thermal activation energy of 130 kJ/mol is used in both MRP-55 and MRP-115.

However, the activation energy could deviate from this value in specific sets of PWSCC CGR data. For example, Cassagne et al. [12] reported three values of activation energy, 108, 139, and 174 kJ/mol for PWSCC CGR in three heats of alloy 600 materials. G.A. Young et al [15] reported $E_{\mathrm{aae,DH}} = 176 \pm 17.8$ kJ/mol under constant DH and $E_{\mathrm{aae},\Delta\mathrm{ECP(Ni/NiO)}} = 125 \pm 47$ kJ/mol under constant ΔECP(Ni/NiO) for an alloy 600 base metal. Despite the different combinations of heats of materials, water chemistry, loading level and testing methods, together with the scattering in the CGR data, the experimental activation energies for PWSCC of nickel alloy 600 and weld metals 182/132/82 generally fall in a consistent range above 100 kJ/mol, with few exceptions under the constant ΔECP(-Ni/NiO) criteria [15], showing the strong thermal activation of the physicochemical processes of SCC.

2.1.2. Austenitic stainless steels in hydrogenated water

Arioka et al. [19] found that CGR of 316 SS in simulated PWR primary water increased monotonically with increasing temperature, with an E_{aae} of 107 kJ/mol. Guerre et al. [25] reported that CGR of CW 316L increased with temperature from 289 °C to 320 °C, with an E_{aae} of about 65 kJ/mol. Tice et al. [26] compiled the CGR data from CW SS CT specimens and reported a statistically based $\mathrm{E}_{\mathrm{aae}}$ of about 65 kJ/mol. Andresen et al. [6] also reported the monotonic increase of CGR with increasing temperature from 288 °C to 340 °C for CW 316L in hydrogenated pure water, with an E_{aae} of 64.8 kJ/mol. Fig. 3 shows the strong thermally activated crack growth for a one-dimensional (1-D) CW 316 L SS in a simulated PWR environment, with an E_{aae} of about 93 kJ/mol. CGR of a heavily cold worked 316L SS with a yield strength (RT) of about 1000 MPa increases with increasing temperature from 290 °C to 315 °C [18]. However, a drop in CGR was observed at 340 °C. Such a type T-B temperature-dependent SCC growth behavior for cold worked SS needs further experimental verification and mechanistic analysis.

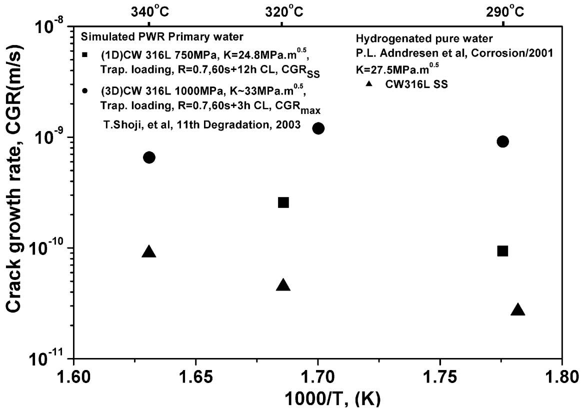

Fig. 3. The Arrhenius plots of CGRs of hardened 316L stainless steels in hydrogenated water environments.

2.2. Austenitic alloys in simulated BWR environments

2.2.1. Sensitized stainless steels in oxygenated pure water with or without chemical additions

Early results of the effects of temperature on SCC of sensitized 304SS were obtained mainly from slow strain rate tests (SSRT) in water with relatively high conductivity, created by intentionally adding ions to simulate early BWR environments or due to unsatisfactory control of water chemistry, compared to more recent emphases and improvements in water chemistry [2,27–29]. Both Type T-A and Type T-B have been observed. Ford and Povich [27] and Agrawal et al. [28] showed a monotonic increase in CGR with temperature from 200 °C to 288 °C for sensitized 304SS in 8 ppm water, but a decrease in CGR after increasing temperature from 250 °C to 288 °C in 0.2 ppm water. Ruther et al. [29] reported that maximum IGSCC for lightly sensitized 304SS occurred at temperatures between ~200 °C and 250 °C in 0.2 ppm DO pure water. Data of Ruther et al generally showed a weak dependence of CGR on temperature between 200 °C and 290 °C, with an E_{aae} of about 16 kJ/mol according to the later analyses [2]. Weeks et al. [30] found that CGRs of furnace-sensitized 304SS in pure water containing 22 ppm DO were quite dependent on SSRT strain rates and showed a maximum at about 200 °C.

Fracture mechanics specimen tests have been used for quantifying the effect of temperature on SCC CGRs. Hale [31] reported that CGR sensitized SS in 0.2 ppm DO pure water showed a drastic increase when changing temperature from 100 °C to 150 °C. However, it seemed that CGR was weakly dependent on temperature at 151–292 °C. CGR results from Magdowski and Speidel [32] on sensitized 304SS in pure water with a wide range of DO showed statistically a thermally activated temperature-dependence with an apparent energy of about 46 kJ/mol between about 150 °C and 288 °C, while there seemed to be a change in crack growth mechanism below about 150 °C. Such dependence was weakened due to quite a large range of DO and related parameters such as corrosion potential and pH, and the CGR evaluation procedure [2]. Andresen [2] reported a peak in the CGR of sensitized 304SS in pure water containing 0.27 μs/cm H_2SO_4 with 0.2 ppm DO at about 200 °C, by using CT specimens under trapezoidal loading with 1000 s constant load at K_{max} = 33 MPa $m^{0.5}$ plus R = 0.5/0.01 Hz unloading/reloading. Jenssen and Janson [5] recently reported a monotonic increase in CGR with increasing temperature for sensitized 304SS in 0.5 ppm DO high purity water over the range of about 150–290 °C, using CT specimens under quasi-constant K_{max} of 30 MPa $m^{0.5}$. However, a CGR peak occurred at about 150 °C in sulfate solutions. These results are different from the comments by Andresen [2], who earlier suggested a trend of a monotonic increase in CGR with temperature in high conductivity (or impurity containing) water, while observing a CGR peak in relatively clean water. Also, the relatively weak effect of sulfate on crack growth at 288 °C reported by Jenssen and Janson [5] differs from previous reports of the strongly enhancing effect of sulfate [33,34]. It seems that more evidence is necessary to clarify this issue. The above results show that water chemistry can significantly affect the types of temperature-dependence. The values of E_{aae} based on constant DO or constant ECP criteria could well be different. A quite low $E_{aae,\ ECP}$ for CGR of sens. 304 has been reported [2].

2.2.2. Strain-hardened non-sensitized stainless steels in oxygenated pure water

CGR values of CW 316 L SS measured in oxygenated high purity water at 200, 250 and 288 °C at two DO levels and two K levels are shown in Fig. 4 [7,8]. CGR monotonically increases with temperature. The present results show that the activation energies are different under different conditions. The activation energy tends to be higher in the higher temperature range, especially at low-K levels. The activation energies for the CGR data at K = 30 MPa $m^{0.5}$ are significantly lower than those at K = 15 MPa $m^{0.5}$. These results shows that crack growth of strain-hardened non-sensitized 316L SS in oxygenated water could be controlled by multiple processes.

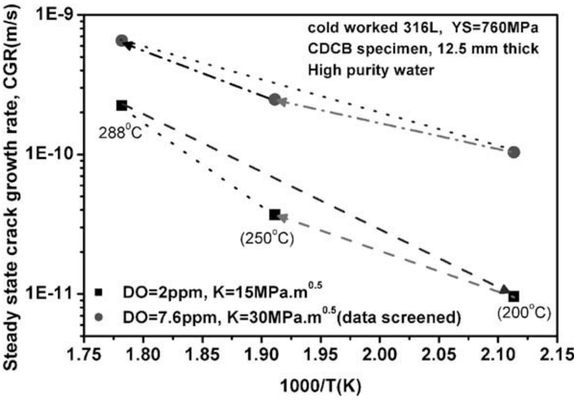

Fig. 4. Arrhenius plots for CGR of a CW 316L SS in pure water at different temperatures.

3. Some fundamental aspects of thermally activated SCC processes

Complex mechano-chemical interactions or multiple physicochemical processes at material/solution interfaces are involved in SCC. The apparent activation energy reflects the combined effects of temperature on the complex physicochemical processes that take place during crack growth. Several mechanisms have been proposed for SCC crack growth of austenitic alloys in high temperature water [3,4,20]. Deformation–oxidation processes at the crack tip are assumed to be crucial in crack growth [4]. Faraday's law has been used to relate the oxidation rate and CGR,

$$\mathrm{CGR} = \frac{\mathrm{d}a}{\mathrm{d}t} = \left(\frac{M}{\rho z F}\right) \cdot \left(\frac{Q_\mathrm{d}}{t_\mathrm{d}}\right), \tag{10}$$

$$t_\mathrm{d} = \varepsilon_\mathrm{d}/\dot{\varepsilon}_\mathrm{ct}, \tag{11}$$

$$Q_\mathrm{d} = \int_0^{t_\mathrm{d}} i(t)\mathrm{d}t, \tag{12}$$

$$Q_\mathrm{d} = Q_\mathrm{I} + Q_\mathrm{II} + Q_\mathrm{III}, \tag{13}$$

$$Q_\mathrm{I} = i_0 t_0, \tag{14}$$

$$Q_\mathrm{II} = \int_{t_0}^{t_\mathrm{p}} i(t)\mathrm{d}t, \tag{15}$$

$$Q_\mathrm{III} = i_\mathrm{p}(t_\mathrm{d} - t_\mathrm{p}), \tag{16}$$

where M is the molecular weight, ρ is the density, F is Faraday's constant, z is the oxidation valence, ε_d is the physical degradation strain of film, ε_ct is the crack tip strain, $\dot{\varepsilon}_\mathrm{ct}$ is the crack tip strain rate (CTSR), $i(t)$ is the transient current at a time t during a film degradation process under stress/strain, i_0 is the active oxidation rate, i_p is the passive current density in the steady passive state, t_0 is the time for the onset of current decay, t_p is the time for the onset of the steady passivation, t_d is the time period for the film degradation. Q_I is the oxidation charge consumed during the fast oxidation period, Q_II is the oxidation charge consumed during the recovery period of the protective oxide film, and Q_III is the oxidation charge during the steady passive state.

Various combinations of rate constants and time constants could lead to many kinds of specific SCC growth kinetics, depending on various combinations of material/environment/loading in SCC systems. For example, in the slip oxidation mechanism, which has been thought to be a working mechanism for SCC of stainless steels in oxygenated pure water, there are $i_0 \gg i_\mathrm{p}$ and $t_\mathrm{p} \sim t_\mathrm{d}$ [20]. With the purely stress-enhanced oxidation mechanism, there is $i_0 \sim i_p$. The rate-controlling steps for different sub-processes could be quite different. For the active oxidation step, the aqueous mass transport of cathodic depolarizers may play an important role in determining the oxidation rate, i_0. In the steady passivation state, the solid-state mass transport of reactive species through the oxide film or the transference of the film into the solution could be rate determining. In the transient period, surface condition could be quite important for the coverage or the growth of a very thin oxide film, and the solid mass transport could limit the further growth of thicker oxide films. To deal with the effect of temperature on complex physicochemical processes, Santarini [9] has proposed an equation for the rate of a global corrosion phenomenon,

$$V = V(K_i, m_j, \varphi_k), \tag{17}$$

where V is the reaction rate, K_i are the characteristics constants of the physicochemical steps (i varying from 1 to n_k), m_j are the morphological parameters (j varying from 1 to n_m), and φ_k are the global physicochemical parameters (k varying from 1 to n_m). According to the definition by Santarini [9], physicochemical constants include kinetic constants, thermodynamic equilibrium constants, mechanical constants, etc. Local physicochemical constants include concentrations, concentration gradients, electric potentials, mechanical stresses, strain rate, etc. Parameters relative to the material and to the environment (far from the interfaces) are considered global. These concepts are quite helpful for identifying the role of thermal activation in SCC. Although there are many limitations to relating the apparent activation energy directly to specific reaction mechanisms for complex physical systems without intimate knowledge, as pointed out by Santarini [9], the temperature-dependence of CGR, the E_{aae} and its response to other key factors could be used to search for relevant parameters.

If the sub-processes that promote SCC growth are thermally activated, Type T-A temperature-dependence is expected, such as is observed with PWSCC of nickel based alloys and weld metals, and for strain-hardened non-sensitized SS in oxygenated or hydrogenated pure water. For Type T-B temperature-dependence, there might be at least one inhibiting process, either thermodynamic or kinetic, that is enhanced by thermal activation. In some cases Type-B dependence has been found in water with added ions such as sulfate, implying that the possible negative-catalytic kinetic effect of added ions might lead to lower oxidation rates at higher temperatures. According to Vankeerberghen and Macdonald [10], Type T-B temperature-dependence for sensitized 304SS in dilute sulfuric acid solutions has been attributed to the competing effects of temperature on the thermally activated processes that occur at the crack tip and the properties of the bulk environment. Recent data [5] show that sensitized 304SS in high purity water would follow type T-A temperature-dependence, which provides additional proof that the added ions would play an important role in producing Type-T-B temperature-dependence. Such a temperature-dependent CGR behavior related to environmental factors indicated that the oxidation sub-processes rather than the material properties were the main rate-controlling steps in the crack growth.

One important feature of the PWSCC of nickel based alloys and weld metals is that the apparent activation energy is generally high and could reside in a relatively consistent range above 100 kJ/mol, with few exceptions based on constant $\Delta ECP_{Ni/NiO}$. Scott [1,3] has ascribed this kind of high and relatively consistent activation energy to an internal oxidation mechanism where the solid-state diffusion would be the rate-controlling step and generally has a consistent and high activation energy. It is postulated that stage I and the related Q_I would not have a significant contribution to the PWSCC growth, while stages II/III, with Q_{II}/Q_{III}, could be dominant.

The E_{aae} for CGR of CW 316 L in oxygenated pure water was found to be related both to the temperature itself and the loading level. The activation energy is not a constant over the whole temperature range and tends to be lower at lower temperatures or at a high K, Fig. 4. Such a change could be due to the effect of temperature on multiple sub-processes that each exhibits different degree of thermal activation. According to Eqs. (10) and (13),

$$\mathrm{CGR} = \left(\frac{M}{\rho z F}\right) \cdot \left(\frac{Q_I + Q_{II} + Q_{III}}{t_d}\right) \tag{18}$$

The apparent activation energies for Q_I, Q_{II}, and Q_{III} could be different. Generally Q_I for active oxidation would exhibit relatively low activation energy. Q_{II} and Q_{III} would be partly or fully controlled by the solid-state mass transport though the interfacial oxide film, thus would possess relatively high activation energies. The ratio of $(Q_{II} + Q_{III})/Q_I$ increase with increasing temperature and thus results in a higher E_{aae} in the higher temperature range, if the reaction mechanisms do not change. Also, the ratio of $(Q_{II} + Q_{III})/Q_I$ could be lower at a higher K due to the increase in crack tip strain rate [4], thus resulting in a lower activation energy. If Q_I is negligible in Q_d, E_{aae} could be relatively constant and high. Despite the complexity of SCC systems and other possible contributing factors such as the oxide film properties, the thermal activation of crack growth behavior for CW non-sensitized 316L could be explained with reference to the deformation–oxidation model [4].

4. Conclusions

Several types of temperature-dependent crack growth behavior in austenitic alloys and weld metals in simulated LWR environments have been categorized. Different definitions and measurements of apparent activation energy for crack growth rate have been described. SCC of nickel-base alloys and weld metals in simulated PWR environments is thermally activated and shows relatively high and consistent apparent activation energies. The temperature-dependence of sensitized 304SS in oxygenated pure water can exhibit either a monotonic increase with temperature or a peak value, depending on the water chemistry, especially the impurity concentration, and testing methodology. Steady state CGRs of CW 316L SS in oxygenated water increase with temperature and the thermal activation energy could vary both with temperature and the loading conditions. The thermally activated processes of SCC in austenitic alloys exposed to simulated LWR environments have been analyzed, based on a deformation/oxidation mechanism.

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