



## Effect of long-term aging on the fracture toughness of primary coolant piping material Z3CN20.09M

Weiwei Yu<sup>a,b</sup>, Minyu Fan<sup>b</sup>, Hongbo Gao<sup>b</sup>, Dunji Yu<sup>a</sup>, Fei Xue<sup>b</sup>, Xu Chen<sup>a,\*</sup>

<sup>a</sup> School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, China

<sup>b</sup> Suzhou Nuclear Power Research Institute, Suzhou 215004, China



### ARTICLE INFO

#### Keywords:

Long-term aging  
Primary coolant piping  
Spinodal decomposition  
Fracture toughness  
SZW  
Critical crack size

### ABSTRACT

In this study, a series of accelerated thermal aging tests were performed on primary coolant piping materials (Z3CN20.09M) at 400 °C for 0, 2000, 5000 and 18,000 h to investigate the resulting microstructure change and fracture toughness. To further assess their fracture toughness, 0.2 mm offset line method and stretch zone width method were both employed to determine the fracture toughness. The results indicated that after long-term thermal aging, round shaped particles and characteristic morphology of spinodal decomposition were found in ferrite, thus resulting in the thermal aging embrittlement of Z3CN20.09M. Subsequently, the *J*-*R* curves and *J*-*T* curves all decreased significantly with the increasing thermal aging time. Furthermore, the fracture toughness parameters  $J_{50}$ ,  $J_{SZW(2D)}$ ,  $J_{SZW(3D)}$  and  $dJ/d\Delta a$  were first decreased rapidly and then slow saturated. In addition, the  $J_{SZW(2D)}$  and  $J_{SZW(3D)}$  values were significantly lower than  $J_Q$  while the reduction of the critical crack size based on  $J_{SZW(2D)}$  and  $J_{SZW(3D)}$  parameters was about 30% compared with  $J_Q$ , and thus  $J_{SZW}$  was relatively close to the onset of crack initiation.

### 1. Introduction

The cast austenitic stainless steels (Z3CN20.09M) are widely fabricated into primary coolant pipes utilized in nuclear power plants (NPPs) with pressurized water reactors (PWR) due to their adequate strength and superior corrosion resistance (Xue et al., 2009; Byun et al., 2016; Faure and Leggatt, 1996). Z3CN20.09M has a duplex microstructure which consists of  $\alpha$ -ferrite and  $\gamma$ -austenite phases. In general, spinodal decomposition might occur in the  $\alpha$ -ferrite phase, in which the  $\alpha$ -ferrite phase was separated into a Fe-rich  $\alpha$ -phase and a Cr-rich  $\alpha'$ -phase, thus leading to a decrease in mechanical properties of Z3CN20.09M (Xue et al., 2011; Li et al., 2014; Chen et al., 2015; Troshchenko et al., 2007; Chandra et al., 2012; Erauzkin and Irisarri, 2010). Hence, with an increase in aging time, Z3CN20.09M suffers a loss of fracture toughness gradually and then the fracture characteristics change from ductile dimples into brittle cleavages in ferrite and tearing in austenite (Li et al., 2013; Silva et al., 2016; Iturgoyen and Anglada, 2010). Furthermore, the degree of fracture toughness reduction has a positive relationship with ferrite content while the decline increases with rising temperature (Jeon et al., 2016).

Considering that most nuclear power plants are designed to operate more than 40 years in general, fracture toughness value should be known to assess the safety of piping during the long term operation

period (Viehrig et al., 2015). Long-term thermal aging results in the declining plastic deformation ability of the ferrite phases and the decrease in impact toughness of Z3CN20.09M (O'Donnell et al., 1996). Moreover, the loss of impact energy is mainly due to the reduction of stable crack propagation energy in the aging process. In the impact fracture process, cracks initiate firstly and fast propagate in the ferrite phases, and then the ferrite phase fracture along the cleavage planes. Subsequently, the cracks extend to the austenite phases and connected through materials (Li et al., 2011). However, it is not enough just to investigate the impact fracture property of Z3CN20.09M despite the difficulty of static fracture toughness test, especially when the material shows a high plasticity.

Primary coolant piping materials (Z3CN20.09M) are of fundamental importance in the nuclear power industry, but only few reports about localization primary coolant piping materials, especially the influence of long-term aging on the fracture toughness have been published so far. In this study, Z3CN20.09M was performed on an accelerated thermal aging test at 400 °C for nearly 0 h, 2000 h, 5000 h and 18,000 h respectively to investigate the resulting microstructure change and fracture toughness. To further assess their fracture toughness, 0.2 mm offset line method and stretch zone width method were both employed to determine the critical initial fracture toughness  $J_I$  while *J*-*T* curves were transformed from *J*-*R* curves to obtain the crack instability criterion  $J_{50}$ .

\* Corresponding author.

E-mail address: [xchen@tju.edu.cn](mailto:xchen@tju.edu.cn) (X. Chen).

**Nomenclature**

$a$	crack length	$J_{SZW(2D)}$	critical initial fracture toughness (stretch zone width method by 2D scanning electron microscopy)
$\Delta a$	crack extension	$J_{SZW(3D)}$	critical initial fracture toughness (stretch zone width method by 3D imaging)
$B$	net thickness	$J_{50}$	crack instability criterion
$B_N$	net thickness due to side grooving	$dJ/d\Delta a$	rate of increase of J-crack growth resistance
$c$	half length of the surface crack	$K_I$	stress intensity factor
$C$	fitting parameter in J-R curves	$n$	strain hardening exponent in J-R curves
$C_{C(i)}$	specimen crack opening compliance on an unloading/re-loading sequence, corrected for rotation	$N$	fitting parameter in the Ramberg-Osgood equation of stress-strain curves
CMOD	crack mouth opening displacement	$P_i$	instantaneous load
$E$	elastic modulus	SZW	stretch zone width
$J_i$	critical initial fracture toughness	$T$	tearing modulus
J-R curves	J-crack resistance curve	$\alpha$	materials coefficient in Ramberg-Osgood relationship
J-T curves	crack instability assessment diagram	$\gamma$	crack length dependent factors
$J_Q$	critical initial fracture toughness (0.2 mm offset line method)	$\eta$	geometry factor
$J_{SZW}$	critical initial fracture toughness (stretch zone width method)	$\nu$	Poisson's ratio
		$\sigma_0$	flow stress

In addition, the effects of long-term aging on the critical crack size for  $J_{SZW}$  and  $J_Q$  were studied according to the RES-M code (RSE-M, 2010; NUREG/CR 6142, 1994).

## 2. Material and experimental details

### 2.1. Material

The material used in this investigation was Z3CN20.09M cast austenitic stainless steel, cut from the centrifugal casting primary coolant piping of 31" diameter utilized in nuclear power plants (NPP) with pressurized water reactors (PWR). The chemical compositions of the virgin material were listed in Table 1. The typical microstructure of the material was shown in Fig. 1. The ferrite phases accounted for about 16.7% of the virgin material and were embedded in the austenite phases, subsequently forming an interlaced network.

### 2.2. Long-term thermal aging experiments and microstructure change

The embrittling influence mechanism of long-term thermal aging was known to be closely related to the local enrichment of Cr atoms largely caused by the spinodal decomposition or  $\alpha$ -phase precipitation in ferrite phases (Chandra et al., 2012; Li et al., 2013). Z3CN20.09M were heat treated for 0 h, 2000 h, 5000 h and 18,000 h respectively at 400 °C, which was the highest temperature in the temperature range of spinodal decomposition, ensuring the invalidity of other degradation mechanisms which were even a little different with the spinodal decomposition (Xue et al., 2009; Li et al., 2014). After different long-term thermal aging time, the microstructure change of Z3CN20.09M was observed using a JEM-2010 transmission electron microscope (TEM).

### 2.3. Fracture test

After long-term thermal aging, Z3CN20.09M was subjected to fracture toughness tests on a servo hydraulic universal testing machine (MTS Model 810). Compact tension (CT) test specimens with the thickness of 25 mm ( $B$ ) and the width of 50 mm ( $W$ ), made from thermal aged Z3CN20.09M, were employed for carrying out monotonic single specimen J-R tests at room temperature. According to ASTM E1820 (2013) specifications (ASTM, 2015), fracture toughness specimens were fatigue pre-cracked under decreasing  $\Delta K$ , and then each specimen was side-grooved to 10% of its gross thickness on each side.

### 2.4. Scanning electron microscopy

After fracture toughness tests, microstructural observation using a Tescan VEGA TS 5136XM scanning electronic microscopy (SEM) was employed to analyze fracture surfaces and investigate effect of long-term aging on the fracture modes of Z3CN20.09M. Subsequently, fracture surfaces were also applied to study the SZW spanning the culmination of pre-fatigue cracks and the initiation of ductile fracture. As a result, the  $J_{SZW}$  can be identified at  $\Delta a = \Delta a_{SZW}$  according to J-R curves.

### 2.5. 3D surface topography

A large scatter in the values of  $J_{SZW}$  is inherent in the 2D scanning electron microscopy method due to the subjective nature of interpretation and measurement of the stretch zone width. To minimize scatter, the precise determination of the stretch zone width needs a fast, reproducible and reliable method. Nevertheless, the 3D imaging together with the possibility of construction of the 3D surface profiles gives a clear view of the fracture topography, which can hardly be achieved in 2D by tilting and looking from side positions (Weidner et al., 2013). Thus the VK-X100K laser scanning confocal microscope system was employed to determine the stretch zone width.

## 3. Results and discussion

### 3.1. Microstructure change

Transmission electron microscope (TEM) was used to observe the microstructure change of Z3CN20.09M after different thermal aging time. Fig. 2(a) shows the microstructure of ferrite and austenite near the phase boundary in Z3CN20.09M after thermal aging at 400 °C for nearly 5000 h. It is observed that a mottled structure is formed in ferrite, which is typical for spinodal decomposed Fe-rich domains and Cr-rich domains, which had been reported earlier in literature (Li et al., 2014; Li et al., 2013; Silva et al., 2016). With the increasing thermal aging time, it leads to a further development of the phase boundary

**Table 1**  
Chemical composition of the testing material (wt%).

C	Si	Mn	P	S	Cr	Mo	Ni	Co	N
0.019	0.8	0.88	0.011	0.0044	20.18	0.19	8.2	0.05	339 ppm

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