



Studies of the tensile and corrosion fatigue behaviour of austenitic stainless steels

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ABSTRACT

Corrosion fatigue behaviour of four types of austenitic stainless steels were investigated in boiling 45% magnesium chloride solution at a stress ratio of 0.25 and a frequency of 0.1 Hz. Type 316LN stainless steel possessed the best resistance and type 304 stainless steel had the lowest resistance to corrosion fatigue. XPS studies on the fracture surface indicated that the presence of nitrogen as NH_4^+ ion in the surface film of type 316LN stainless steel gave it the highest resistance to corrosion fatigue. Fractographic examination showed wholly transgranular cracking in all cases.

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1. Introduction

The base for the various stainless steels is the binary Fe–Cr system (Fig. 1) [1], the properties of which are modified by the addition of several major alloying elements such as Ni, Mo, and Mn as well as minor ones such as C and N. Fe–Cr–Ni alloys are the most predominantly used austenitic stainless steels. These steels are susceptible to a number of manifestations of localised corrosion attacks, of which the major problem is caused by sensitisation, which can cause premature failure of welded components in an environment under the action of tensile or cyclic stresses. Molybdenum is added to type 316 stainless steel to enhance the pitting and crevice corrosion resistance, besides high temperature mechanical properties. With increasing Mo contents, M_{23}C_6 precipitation and IGC become increasingly influenced by the precipitation of intermetallic phases [2]. The problem of sensitisation can be overcome by using low carbon austenitic stainless steel. Loss of strength due to reduction in carbon content can be offset by addition of nitrogen to the stainless steel. Together with chromium and molybdenum, nitrogen additions to austenitic stainless steel also improve resistance to pitting and crevice corrosion, sensitisation, and stress corrosion cracking [3–12]. The studies on the corrosion fatigue behaviour of austenitic stainless steels have been few and far between [13–20]. However, these studies do not deal with the comparison of the corrosion fatigue behaviour of various types of austenitic stainless steels. Also, no reports are available in literature on the effect of alloying elements on the corrosion fatigue behaviour of austenitic stainless steels except a study by Onoro [21] in which high resis-

tance to corrosion fatigue was reported in type 317 stainless steel with high molybdenum content.

The present work compares the corrosion fatigue behaviour of a few austenitic stainless steels commonly used in nuclear reactor applications. For this purpose, AISI types 304, 316, 316L and 316LN stainless steels were investigated for their corrosion fatigue behaviour in boiling 45% MgCl_2 solution. The relative resistances of these steels to corrosion fatigue give an insight into the effect of various alloying elements on the corrosion fatigue behavior of austenitic stainless steel. The corrosion-fatigue tested fracture surfaces of these steels were studied using X-ray photoelectron spectroscopy (XPS) to give an insight into the corrosion fatigue behaviour. The tensile behaviour of these steels was also studied.

2. Experimental procedures

The materials used in this study were AISI types 304, 316, 316L and 316LN stainless steels. The chemical compositions of the steels are listed in Table 1. It is observed that type 304 stainless steel has a higher carbon content, and type 316LN stainless steel has a higher nitrogen content than other steels. All the steels were studied in the mill-annealed condition.

Microstructural examination was carried out on the steels after polishing them to 1 μm finish and then etching them electrolytically in 10% (by weight) ammonium persulphate solution at a current density of 1 A/cm^2 for about 3 min. Grain size measurements were also carried out.

Round tension specimens (Fig. 1) were made from the different steels as per the guidelines given in ASTM E466 [22]. The specimens were then ground up to 600 grit finish using SiC papers. Tensile tests were carried out at an initial strain rate of $1 \times 10^{-4} \text{ s}^{-1}$.

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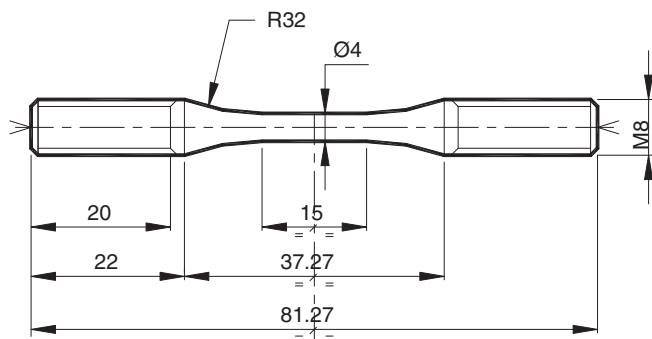


Fig. 1. A schematic of the round tension specimen used in this study.

The tensile test results were analyzed for yield strength (YS), ultimate tensile strength (UTS) and ductility (% total elongation). Corrosion fatigue tests were carried out at various values of mean stress under load control mode in boiling 45% (by weight) MgCl_2 solution at a stress ratio of 0.25 and a frequency of 0.1 Hz. Mean stress was varied by varying both the maximum and minimum stresses. Except the region of interest in the gauge length, the rest of the specimen and the specimen train were masked with silicone elastomer. Care was taken to avoid crevice between the specimen and elastomer. Boiling 45% (by weight) MgCl_2 solution was used in this study since it is a recommended ASTM Standard Practice for evaluating SCC resistance of metals and alloys [23]. Also, it causes quick failures of specimens and the results are highly reproducible. The stress range was varied by varying both the maximum and minimum stress. Number of cycles to total failure was used as the assessment criterion for determining the susceptibility to corrosion fatigue. The fracture surfaces of the corrosion fatigue-failed specimens were examined in a scanning electron microscope (SEM) to study the cracking modes.

X-ray photoelectron spectroscopy (XPS) studies were carried out on the fracture surface of the corrosion-fatigue tested specimens in a SPECS make XPS system with a monochromatised Al K α ($E = 1486.74$ eV) source and a PHOIBOS 150 analyzer with a chosen energy resolution for recording survey spectra. The pressure in the analysis chamber was maintained at 2×10^{-9} mbar throughout the measurements. The spectra were collected using SPECS Lab 2 data analysis software supplied by the manufacturer. The full width at half-maximum (FWHM) obtained for Ag 3d $_{5/2}$ (368.3 eV, BE) line is 0.75 eV. The analyzed regions were Fe2p $_{3/2}$, Cr2p $_{3/2}$, Ni2p $_{3/2}$, Mo3d, O1s, N1s and C2s. High resolution spectra were obtained using 12 eV pass energy. A Shirley background subtraction [24] was used to obtain the XPS signal intensity. After a background subtraction, and processing with Casa XPS software developed by Fairley [25], the XPS signals were separated into the contributions of the different species. The peaks were fitted using an asymmetric Gaussian/Lorentzian mixed function. The evaluation of the spectra was performed using the parameters of standard peaks. Ar $^{+}$ ion sputtering was used to study the film composition across its thickness. Ar $^{+}$ ion sputtering was carried out with primary beam energy of 5 kV and a current of 50 μ A. The

Table 1
Chemical composition of the various stainless steels in weight%.

Stainless steel	C	Cr	Ni	Mo	Si	Mn	S	P	N
Type 304	0.079	18.97	10.78	0.56	0.61	1.47	0.005	0.043	0.04
Type 316	0.044	16.43	12.48	2.12	0.72	1.81	0.002	0.025	0.03
Type 316L	0.015	16.31	12.09	2.2	0.37	1.6	0.013	0.028	0.04
Type 316LN	0.025	18.16	11.9	2.4	0.28	1.6	0.044	0.01	0.09

Ar⁺ ion sputtering was done for 1, 2 and 4 min. For every 1 min of sputtering, about 10 Å of the passive film got removed.

3. Results and discussions

Fig. 2 shows a typical microstructure of the various austenitic stainless steel of the present study in the mill-annealed condition. Equiaxed grains free of precipitates and containing annealing twins were observed. Table 2 shows the average grain sizes measured in the various steels. The average grain size of the steels was nearly the same and varied in the range of 29–49 μm .

The tensile behaviour of the various stainless steel was studied by assessing their yield strength (YS) (0.2% proof stress), the ultimate tensile strength (UTS) and ductility (% total elongation). Table 3 shows that type 304 stainless steel possessed the highest YS and UTS, and lowest ductility. This indicated the influence of carbon content, which was more than two times than that present in any of the other steels. The rest of the steels ranked in order of decreasing YS and UTS, and increasing ductility was type 316LN

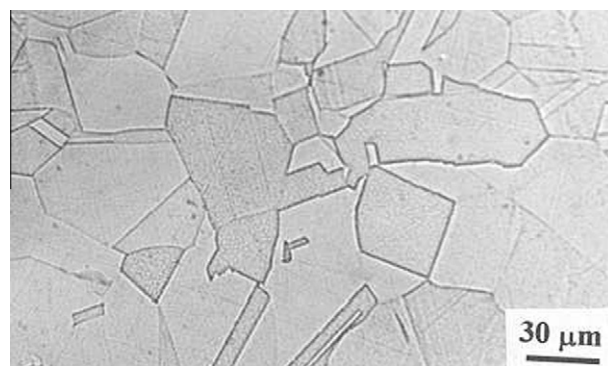


Fig. 2. A typical microstructure of the mill annealed austenitic SS of the present study.

Table 2
Grain size of the various austenitic stainless steels used in the present study.

Stainless steel	Type 304 SS	Type 316 SS	Type 316L SS	Type 316LN SS
Grain size (μm)	49	34	32	29

Table 3
Tensile properties of the various austenitic stainless steel.

Stainless steel	YS (MPa)	UTS (MPa)	Total elongation (%)
Type 304	416	688	78.2
Type 316	292	614	93.1
Type 316L	242	583	95.2
Type 316LN	386	669	80

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