

CHAPTER 2
MATERIALS
AND
EXPERIMENTAL
METHODS

CHAPTER - 2

MATERIALS AND EXPERIMENTAL METHODS

2.1 INTRODUCTION

This chapter describes the details of materials and methods used for carrying out studies in the present investigation. It also gives details of various characterization techniques used in the study.

2.2 MATERIALS

The 904L Super Austenitic Stainless Steel was procured from M/s Bharat Aerospace Agency, Mumbai, as hot rolled rods of 16 mm diameter. Its chemical composition is presented in **Table 2.1**. As-received rods were solution treated at 1100 °C-for 1h and quenched in water. The hardness of the solution treated sample was measured to be 78 HB. The microstructure of the 904L steel in the solution treated condition was examined following mechanical polishing on emery papers and subsequently on velvet cloth using alumina powder and etching with aqua regia. Disc-shaped specimens of 14.5 mm diameter and 5 mm thickness were prepared for the oxidation and hot corrosion study with a surface finish of #600 grit. Prior to exposure, each sample was cleaned with acetone, dried and stored in desiccators.

Table 2.1: Chemical composition of the super austenitic stainless steel 904L in wt%.

C	Mn	Si	P	S	Ni	Cr	Cu	Mo	N	Fe
0.014	1.45	0.38	0.0024	0.02	24.1	19.95	1.59	4.02	0.062	Bal.

2.3. EXPERIMENTAL METHODS

2.3.1 OXIDATION TEST

In the present study, oxidation tests were performed at four different temperatures (500, 550, 600 and 650 °C) under cyclic and isothermal exposures up to 100 h. In cyclic oxidation tests individual sample was kept silica crucible and exposed to test temperature in still air for 15 cycles, in a muffle furnace, where the temperature of the furnace was controlled within ± 2 °C of the set temperature. The silica crucible with individual sample was weighed before and after each cycle. The initial 5-cycles consisted of 1 h soaking each at the test temperature, followed by 4-cycles of 5 h soaking, 3-cycles of 10 h soaking and rest 3-cycles of 15 h soaking, followed by cooling to room temperature. The samples were directly loaded and unloaded within a few seconds after the completion of each cycle. The weight change was measured using an Axis electronic balance of 0.1 mg accuracy.

In isothermal oxidation tests, individual sample was kept in silica crucible and the samples were continuously exposed to 500, 550, 600 and 650 °C separately for 25, 50, 75 and 100 h in four different muffle furnaces. After each isothermal exposure the sample was allowed to cool outside the furnace up to the room temperature and its weight change was measured using an Axis electronic balance of 0.1 mg accuracy. A schematic sketch of cyclic and isothermal test is shown in **Figure 2.1**.

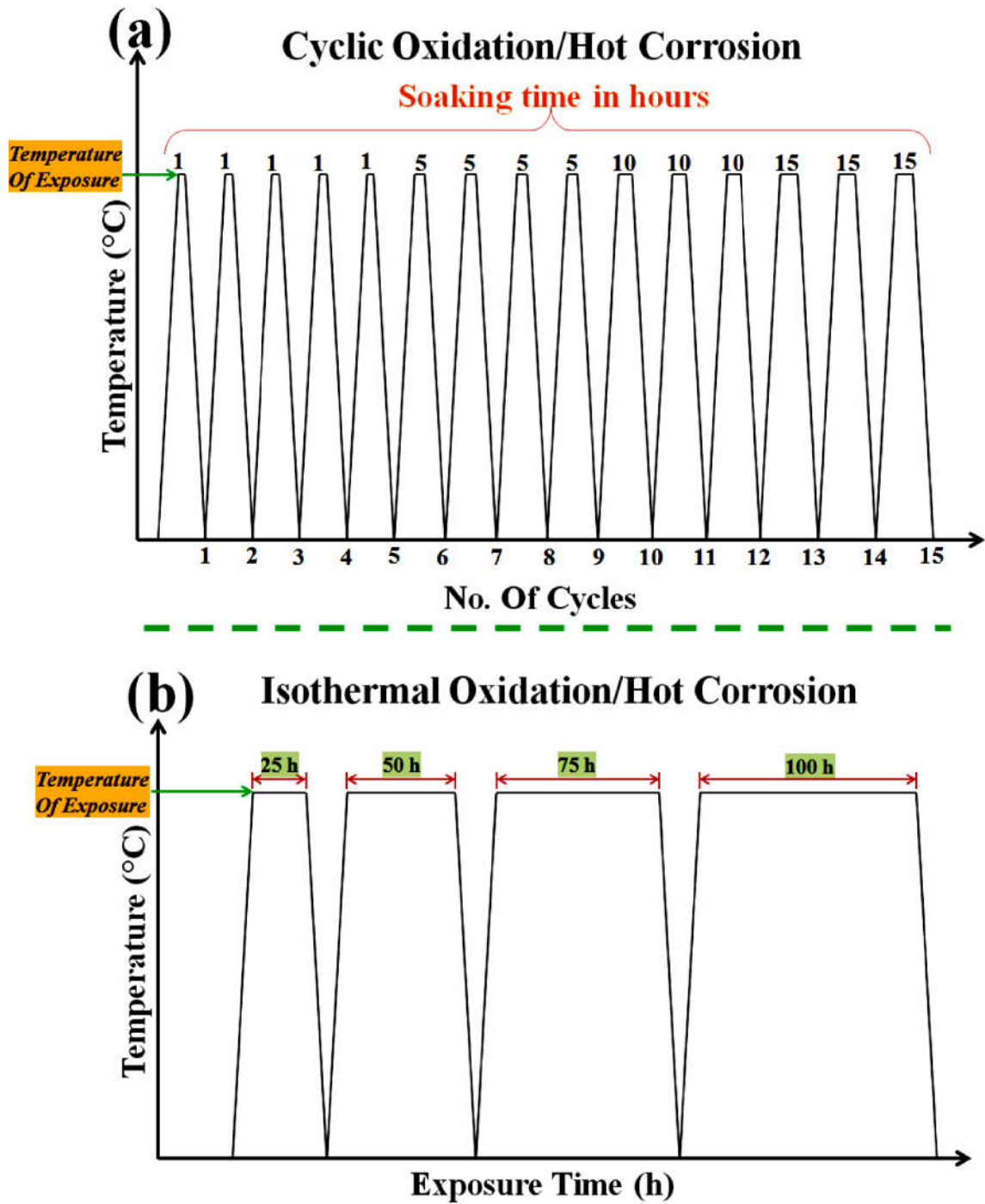


Figure 2.1: Schematic sketch of (a) Cyclic and (b) Isothermal Tests.

2.3.2 HOT CORROSION TEST

For hot corrosion tests, each sample of 14.4 mm diameter and 5 mm thickness was spray coated with salt solution using spray gun. Two different mixtures of the salts NaCl, KCl, Na₂SO₄ and K₂SO₄, were prepared and designated as SM1 and SM2, having Cl:S ratios of 40:60 and 60:40 respectively, as per the compositions given in **Table 2.2**. The solutions were prepared by dissolving salts in freshly prepared double distilled water. For salt coating the samples of known weight were first kept on a hot plate maintained at 120 °C and salt solution was sprayed over the surface. The water of the solution evaporated and salt was deposited on the surface uniformly. The process of salt spray continued till the deposition up to 3-4 mg/cm² (**Figure 2.2**). After the salt coating the samples were weighed and kept in an oven at 110°C to avoid absorption of moisture.

Table 2.2: Composition of the salt mixtures.

Designation	Composition of the salt mixtures	Cl:S ratio
SM1	20wt% NaCl + 30wt% Na ₂ SO ₄ + 20wt% KCl + 30wt% K ₂ SO ₄	40:60
SM2	30wt% NaCl + 20wt% Na ₂ SO ₄ + 30wt% KCl + 20wt% K ₂ SO ₄	60:40

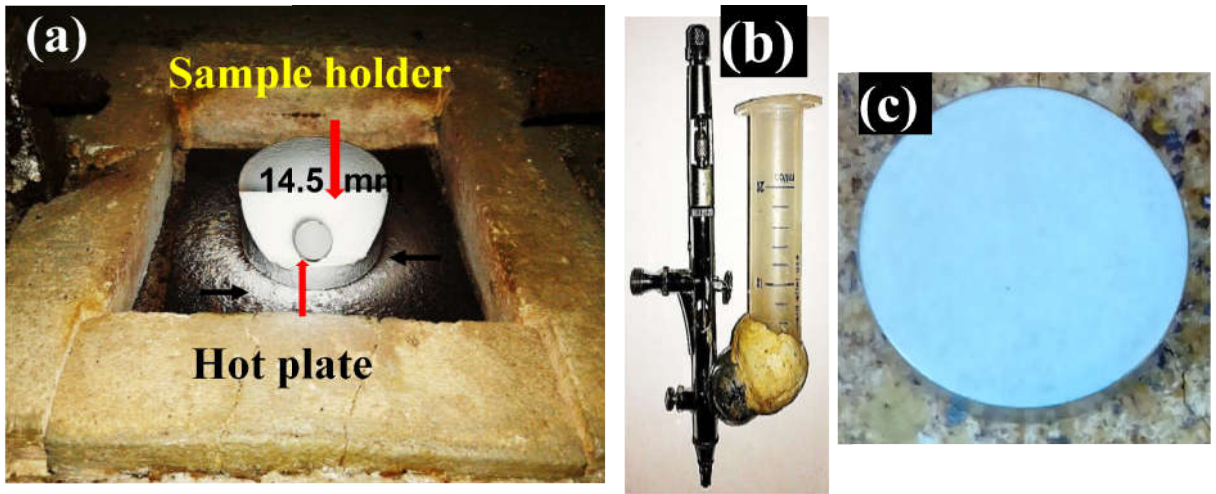


Figure 2.2: Salt coating on 904L sample: (a) hot plate with a sample, (b) salt spray gun and (c) salt coated sample.

For hot corrosion test each salt coated sample was first kept in a silica crucible carefully to avoid any spalling of salt and was weighed. Subsequently, samples were subjected to cyclic and isothermal hot corrosion tests as per the procedure described in the earlier section 2.3.1.

2.4. CHARACTERIZATION

The surface of the oxidized and hot corroded specimen of SASS 904L was characterized using, scanning electron microscopy (SEM), X-ray diffraction (XRD) and Raman spectroscopy. The cross sections of oxidized and hot corroded samples were examined with EPMA (Electron probe Micro-Probe Analyzer).

2.4.1 OPTICAL METALLOGRAPHY

Optical metallography of the samples was carried out using Leitz Metalux-3 optical microscope at different magnifications. Optical micrograph of the solution treated specimen of 904L steel is shown in **Figure 2.3**. It is observed that there is one phase microstructure with equiaxed austenite grains and distinct annealing twins. The mean intercept length of the grains was found to be $\approx 40 \pm 0.5 \mu\text{m}$.

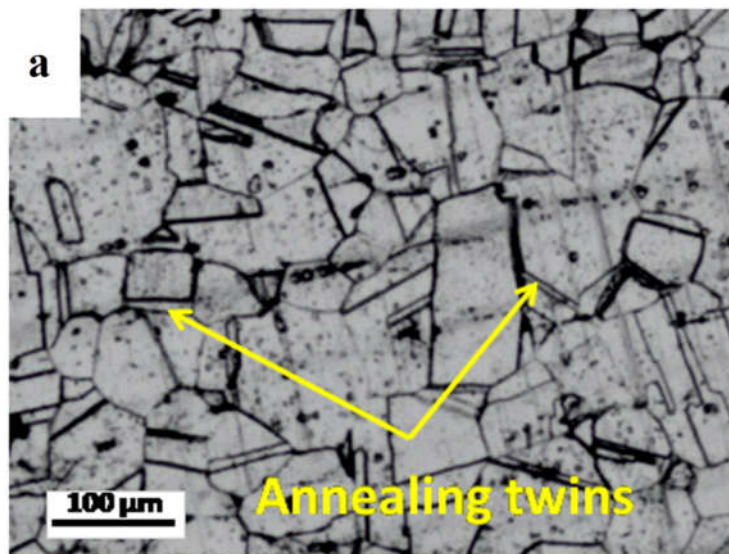


Figure 2.3: Optical micrograph showing microstructure of the SASS 904L solution treated at 1100 °C for 1h and water quenched.

2.4.2 SCANNING ELECTRON MICROSCOPY (SEM)

SEM (FESEM Quanta 200 FEG and Zeiss EVO/18) study with EDS spectrum of the specimens, subjected to various experimental conditions, was carried out to examine the surface, corrosion scale and cross-section. The hot corroded specimens were washed in hot distilled water and subjected to ultrasonic cleaning in acetone to remove loose salt particles and were finally dried. Further, the hot corroded samples of 20 mm diameter and 5 mm thickness were sectioned in two halves along the diameter, perpendicular to treated surface

and the resulting section was mechanically polished to examine the depth of the oxide scale formed on the surface. The products resulting from hot corrosion of the specimens were analysed using energy dispersive spectroscopy (EDS).

2.4.3 ELECTRON PROBE MICRO ANALYSIS (EPMA)

The cross sections of the hot corroded samples were examined for elemental distribution under Electron Probe Micro Analyzer (EPMA) CAMECA SX Five instrument at a voltage of 15 kV with wavelength dispersive spectroscopy (WDS) and current of 10 & 20 nA with a LaB6 source in the electron gun.

2.4.4 X-RAY DIFFRACTION (XRD)

X-ray diffraction study was carried out using RIGAKU X-ray diffractometer for characterization of phases formed after oxidation and hot corrosion. Cu-K α radiation of wavelength 1.5402Å with Ni filter was used.

2.4.5 RAMAN SPECTROSCOPY

Raman Spectroscopy study was carried out to detect oxide phases that are present on the outer surface of the oxide scale. For each sample, Raman spectra were randomly acquired with a Renishaw RM1000 spectrometer using a 514.532 nm using Renishaw for characterization of phases, wavelength 0.780 nm.