# Compatibility of Stainless Steel 316L in Flowing Non-isothermal Lead Bismuth Eutectic

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**Abstract.** Lead (44.8 wt%) and bismuth (55.2 wt%) eutectic (LBE) has been proposed to be used as spallation target in accelerator driven system (ADS) being developed in India. Selection of suitable material for the containment for LBE is a key issue in the success of ADS technology. Corrosion of stainless steel 316L (SS 316L) has been studied in LBE in a thermal convection loop at temperatures of 350 and 450°C for 4000 h. The dissolved oxygen concentration in LBE was 4 x 10<sup>-7</sup> wt % and the flow rate was 16 cm.sec<sup>-1</sup>. SS 316L showed a corrosion rate of 4.5 x 10<sup>-3</sup> µm.h<sup>-1</sup> after 4000 h at 450°C while no change was observed at 350°C. X-ray photoelectron spectroscopy (XPS) showed presence of thin film consisting of oxides of iron and chromium on the surface of SS 316L exposed at both temperatures. Tensile test and fractography did not show any degradation of mechanical properties or microstructure of SS 316L on exposure to LBE at either temperature. There was neither penetration of lead-bismuth nor any depletion of nickel from the surface of SS 316 L after 4000 h at 350°C.

Keywords: Lead-bismuth eutectic, SS 316L, corrosion PACS: 81.40.Np, 81.65.-b

## **INTRODUCTION**

Accelerator driven systems (ADS) being developed in India is proposed to have LBE as spallation target. Liquid LBE has favorable thermo-physical and nuclear properties, and low chemical reactivity towards water and air. However, austenitic steels are susceptible to corrosion by LBE at moderately high temperatures due to high solubility of nickel, iron and chromium [1]. Most successful method of reducing the attack on steel by LBE is by maintaining oxygen concentration in the eutectic to form and maintain a protective oxide film on the steel surface while preventing precipitation of lead oxide [2]. This is achieved by passing a suitable mixture of hydrogen and moisture through the molten eutectic [3, 4]. Stainless steel 316L (SS 316L) can be a candidate material for the containment of LBE. The operational temperature of LBE spallation target in the proposed ADS system is expected to be about 400°C and, therefore corrosion data needs to be generated around this temperature. Aiello et al observed 316L preferential dissolution of Ni and Cr with surface ferritisation when SS 316L was exposed to flowing LBE containing  $10^{-8}$  wt% at 400°C after 1500 h [5]. With similar dissolved oxygen, non-preferential penetration of LBE upto 5µm was observed for SS 316L after 1000 h at 450°C [6]. This paper reports data on the corrosion of stainless steel 316L after 4000 h exposure to flowing LBE in a thermal convection loop at temperatures with dissolved oxygen concentration of 4 x  $10^{-7}$  wt %. The compatibility tests were carried out non-isothermally at 350 and 450°C with the flow rate of LBE at 16 cm/sec. Corrosion rate of SS 316L has been determined gravimetrically, while tensile test and fractography have been used to evaluate the nature of fracture and changes in mechanical properties. X-ray photoelectron spectroscopy (XPS) has been used to analyze the composition of the surface film on the exposed steel. Scanning Electron Microscopy (SEM) and Energy dispersive X-ray (EDAX) analysis of the cross-section was used to study microstructural changes and the nature of attack at the interface of SS 316L and LBE.

#### EXPERIMENTAL

SS 316L of composition (wt%): Cr:17.3; Ni:12.1; Mo:2.3; Mn:1.8; Si:0.35; C:0.02; Fe: balance was used in this study. Tensile specimens of gauge length 12.5 mm and rectangular coupons of dimension 35 mm x 10 mm x 2 mm were solution annealed by heating at 1050 °C for 1 h followed by water quenching. Details of the sample preparation and the LBE thermal convection loop are similar to earlier studies [7]. Corrosion experiments were carried out for 4000 h at a flow rate of 16 cm/s and temperature of 350 (cold section) and 450°C (hot section) with 4 x  $10^{-7}$  wt% oxygen in the melt. Few coupons were cleaned in a 1:1:1 solution mixture of acetic acid, hydrogen peroxide and ethyl alcohol to remove residual eutectic from the specimen surfaces [5]. Changes in the weight of the specimens were recorded and the composition of the surface film on the specimens was studied by XPS. Tensile tests of exposed SS 316L specimens, without cleaning, were carried out in air at 25 °C at a strain rate of 8.0 x  $10^{-5}$  s<sup>-1</sup>along with annealed unexposed specimens of SS 316L. Nature of the fracture was studied by SEM fractography. Coupons of SS 316L exposed to LBE were sliced along the cross-section, polished and then electrolytically etched in oxalic acid as per ASTM A262 Practice A [8]. SEM and EDAX were used to observe the microstructure and the composition at the interface of SS316L with LBE.

## **RESULTS AND DISCUSSION**

SS 316L specimens exposed to LBE containing 4 x  $10^{-7}$  wt% oxygen for 4000 h did not show any material loss at 350°C but a corrosion rate of 4.5 x  $10^{-3}\mu$ m.h<sup>-1</sup> was observed at 450°C. Aiello et al observed a corrosion rate 4.4 x  $10^{-3}\mu$ m.h<sup>-1</sup> for SS 316L after 4500 h exposure to flowing LBE containing dissolved oxygen of  $10^{-10}$ - $10^{-8}$  wt% at 400 °C [5]. Under similar oxygen environment, raising the temperature to 450°C resulted in the corrosion of SS 316L with maximum penetration of LBE up to 5 $\mu$ m in localized regions after 1000 h [6]. Higher concentration of oxygen, in the present study, provided better protection to SS 316L resulting in corrosion rate comparable to that obtained after 4500 h at 400°C or after 1000 h at 450°C. XPS spectra, shown in Fig.1, indicated the presence of thin layer consisting of Fe<sub>2</sub>O<sub>3</sub> and FeCr<sub>2</sub>O<sub>4</sub> on the surface of SS 316L exposed at 350 and 450°C. However, the thickness of the oxide film could not be determined. The presence of the oxide film prevented the corrosion of SS 316L at 350°C but was not protective enough at 450°C.



**FIGURE 1.** XPS spectra of (a) iron and (b) chromium on the surface of SS 316L exposed to LBE for 4000 h.

Tensile tests carried out in air at 25°C did not show any significant change in ductility of SS 316L after exposure at 350°C while the value increased by about 20% after exposure at 450°C compared to unexposed SS 316L as shown in Table 1. The ultimate tensile strength (UTS) of SS 316L was about 600 MPa after exposure to LBE for 4000 h at 350 and 450°C at compared to 550 MPa for unexposed steel. However, the yield strength (YS) remained almost unchanged. The simultaneous increase in ductility and UTS of SS 316L after exposure to LBE at 450°C requires further investigation.

Treatment condition	UTS (MPa)	YS (MPa)	Ductility (%)
Annealed	550±12	276±10	58±3
350°C, 4000h	600±8	273±12	63±2
450°C, 4000h	605±5	270±9	79±6

**TABLE 1.** Mechanical properties, in air at 25°C and strain rate of 8.0 x 10<sup>-5</sup> s<sup>-1</sup>, of SS 316L underdifferent treatment

Fractography of the specimens after tensile test, shown in Fig.2, revealed that the mode of fracture in SS316L was ductile in nature with dimples of uniform size at both 350 and 450°C and are not different from that of unexposed annealed steel.



FIGURE 2. Fractograph of SS 316L, (a) annealed, (b) 350°C, 4000 h and (c) 450°C, 4000 h

Fig.3 shows the cross-section of specimens of SS 316L exposed to LBE at 350 and 450°C and subsequently etched in oxalic acid. Typical "step" structure was observed at both temperatures due to differential etching of grains. Exposure to LBE did not result in carbide precipitation or sensitization of SS 316L at either temperature.



FIGURE 3. Cross-section of SS 316L exposed to LBE for 4000 h at (a) 350°C and (b) 450°C

EDAX analysis revealed presence of a layer of LBE (whitish region) on SS 316L as shown in Fig.3a-b. Analysis at 5  $\mu$ m (point: X5) and 20  $\mu$ m (point: X20) from the interface between LBE and steel surface showed iron, chromium and nickel content similar to the bulk SS 316L confirming that there was no penetration of LBE into the grain boundaries of SS 316L or depletion of nickel at either temperature after 4000 h exposure.

# CONCLUSIONS

SS 316L resists corrosion in LBE at 350°C even after 4000 h exposure due to the presence of oxide film of iron and chromium. However, at 450°C, SS 316L exhibited corrosion of 18  $\mu$ m despite the presence of oxide film. SS 316L did show any degradation of mechanical properties or microstructure on exposure to LBE at either temperature. There was neither penetration of lead-bismuth nor any depletion of nickel from the surface of SS 316L.

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