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Cyclic oxidation behaviour of low alloy steel And aisi 304 steel inair at 900°c

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ABSTRACT : *The oxidation behaviour of Low alloy steel its German designation (20mnmoni55) and AISI 304 steel has been studied in air under cyclic conditions at 900°C for 50 hr cyclic. Oxidation kinetics was established by thermogravimetric technique. Each cycle consisted of 1 hour heating at 900°C followed by 20 min of cooling in air. Low alloy steel sample followed somewhat linear rate of oxidation with more weight gain while AISI 304 sample followed the non-linear rate law of oxidation with less weight gain. The microstructural feature and phase transformation of oxide scales were characterise by X-ray diffraction (XRD) and field emission scanning electron microscopy/energy dispersive X-ray (FESEM/EDAX) techniques. Keywords: Low alloy steel, AISI304 steel, Air Oxidation*

1. INTRODUCTION

Chromium and Molybdenum containing Ferritic steel are well known for their good mechanical properties combining high temperature strength and creep resistance with high thermal fatigue life, as well as with good thermal conductivity, weldability, and resistance to corrosion and graphitisation. Because of these characteristics

this type of steels have attracted special interest for application in industrial processes related to carbochemistry, oil refining, carbon gasification and energy generation in thermal power plants, where components like, heat exchangers, boilers and pipes operate at high temperatures and

pressures for long periods of time [1,2]. On the other hand iron based austenitic alloy is one of the most utilized high temperature alloy components in industrial engineering plants [3]. At high temperature exposure the interaction between a metal or an alloy and the surrounding gases and combustion products leads to corrosion, thus leading to failure for materials and structures [4-6]. It is commonly

reported that as a result of the oxidation process under Cyclic conditions a protective Cr-containing oxide and Fe-containing oxide is developed on the surface of the steel causing a decrease of the

oxidation rate with time. On the other hand, depending on the oxidation temperature and the chemical composition of the steel, both, the mechanisms of formation and the microstructural characteristics of the oxide scale, along with the degree of protection it provides, are different [7]. Since very limited papers are available for cyclic high temperature oxidation established of Low alloy steel and AISI 304 steel, therefore the present work has been focused to study the oxidation behaviour of Low alloy steel and AISI 304 steel in air at 900°C for 50 hr cycle. The reason of 50 hr cycle (short duration) is to know whether these materials are able to form a protective scale or not.

2. EXPERIMENTAL PROCEDURE

The experimental work was performed by using samples of Low alloy steel & AISI 304 steel. The Low alloy steel samples were obtained from, BARC, Mumbai ,India and AISI 304 steel was received from Jindal steel, Hissar, India and its chemical composition is shown in Table 1. Each specimen measuring dimensions of approximately 20 x 15 x 3.5 mm were cut from plate sections and grinded by using 220, 320, 1/0, 2/0, 3/0, 4/0 grid SiC emery papers. Subsequently, it is polished on cloth polishing disc by using alumina powder followed by diamond paste.

3. HIGH TEMPERATURE OXIDATION

The weight change measurements were used to study the kinetics of high temperature oxidation behaviour of both sample specimens in air at 900°C. Both mirror polished samples were kept in the alumina boats and then inserted inside SiC tube furnace. Cyclic high temperature oxidation studies were performed in air with each cycle consisting of 1 h of heating at 900°C followed by 20 min of cooling at room temperature for up to 50 cycles. The cyclic loading was imposed to create the aggressive conditions, similar to the actual conditions, for corrosion testing. The weight changes were taken by electronic balance (make Contech, India) having sensitivity of 1 mg. Spalled scales was also taken into consideration. Corroded samples from air oxidation were analysed by XRD (BRUKER-binary V3) and

FESEM/EDAX techniques to elucidate the corrosion mechanisms.

4. RESULTS

4.1 HIGH TEMPERATURE OXIDATION STUDIES

The oxidation of sample which occurred in air at a temperature of 900°C is shown in .Fig1 (a-b). The oxidation behaviour of low alloy steel in air was

linear because the oxide layer formed used to peel of very easily and on the other hand AISI 304 steels behaviour was parabolic and also the weight gain of AISI 304 steel was less because the oxide layer which were falling in boat were very thin in size. The graph reveals that AISI 304 steel is better than low alloy steel in an environment of air oxidation (for 50 cycles).

Table 1: Chemical Composition of Samples.

Type of steel	C	Mn	Si	S	P	Cr	Mo	Cu	Ni	Al	Fe
20MnMoNi5	0.200	1.48	0.266	-	0.02	0.170	0.500	0.072	0.598	0.044	Bal
5	0	7	9	-	8	0	0	5	3	3	.
AISI 304	0.115	1.66	0.675	0.000	-	18.00	0.217	0.719	6.801	-	Bal
	8	0	0	6		0	2	4			.

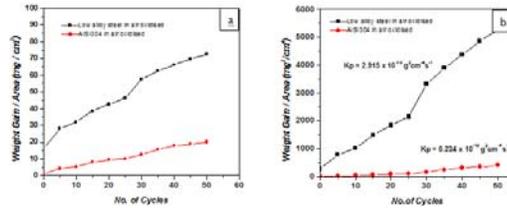


Fig.1.(a-b) Weight gain versus No. of cycles plot for low alloy steel & AISI 304 bare steel exposed to

cyclic study in air environment at 900°C for 50 cycles.

Table 2: Parabolic rate constant (Kp)

Substrate	Kp (10 ⁻¹⁰ g ² cm ⁻⁴ s ⁻¹)
Low alloy steel	2.915
AISI steel 304	0.234

The oxidation rate of both steel samples is shown in Table 2. The K_P value of low alloy steel is higher than AISI304 steel. Hence this table indicate that AISI304 steel has higher oxidation resistance at 900°C for 50 hr. as compared to low alloy steel. After 50 hrs cyclic oxidation at 900°C, both oxidized sample has been observed by visually and shown in Fig.2 (a-b). Oxidised AISI304 steel has crack free scale which is shown in Fig.2 (b), whereas low alloy steel has defects (crack) in scale which is shown in Fig.2 (a)

4.2 XRD RESULT FOR BOTH STEEL

From Fig 2(c), X-Ray Diffraction analysis it is found that iron oxide (Fe₂O₃), chromium oxide (Cr₂O₃) are major phases which are formed in low

alloy steel & in spallation of scale, where as NiO, NiCr₂O₄ oxide scales are present besides the Fe₂O₃ and Cr₂O₃ in AISI304 & in spalled scale sample of AISI304. Cr₂O₃ and NiCr₂O₄ are more protective oxide layer on the surface due to which further oxidation is prevented as it acts as barrier for corroding media to substrate but in case of low alloy steel, amount of Cr₂O₃ is very less which is mainly responsible for the oxidation resistance. Also it is found that no spinel are formed due to which it is having less protective oxide layer, this is also major factor which led to accelerated rate of weight gain.

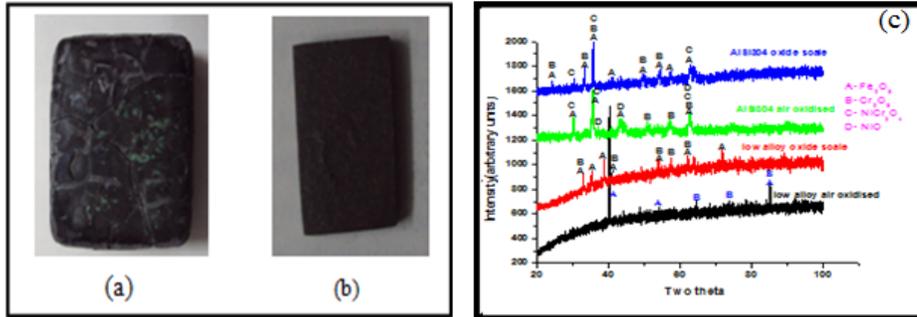


Fig 2 (a-b) Macrograph of low alloy steel air oxidised sample, AISI304 air oxidised sample (c) XRD graph

4.3 SURFACE SCALE ANALYSIS

The SEM/EDAX analysis for low alloy sample & AISI304 sample after oxidation in air for 50 cycles at 900°C is shown in Fig 3(a) and Fig 3(b). The oxide scale reveals the dominance phase in scale is Fe₂O₃ and along with this compounds of Fe₃O₄ and Cr₂O₃ are also formed. At higher magnification the morphology of scale indicates that the oxide formed are layer wise i.e. one by one layer but the weight wise composition is approximately same. The FESEM micrograph for oxidized low alloy steel shown in Fig 3(a), indicates that growth of scale has done from inside (having Fe₂O₃ phase) as compared to the other spot and crack in scale has formed in middle. Due to crack and porous scale oxygen penetrates more to the substrate and which in turns oxidation rate is high, while FE-SEM micrograph of oxidized AISI304 steel shown in Fig 3(b), indicate that Scale is mainly consist of Fe₂O₃ with little amount of Fe₃O₄, and very small amount of NiO is formed and also compact nature of scale is

for low alloy, AISI304 sample and its oxide scales in air oxidised environment at 900°C.

In other words we can say that diffusion of oxygen through scale is less, which reveals that this substrate has high oxidation resistance as compared to low alloy steel.

4.4 CROSS SECTIONAL ANALYSIS

Fig.4 (a-b) shows the cross-sectional FE-SEM micrograph of low alloy and AISI304 steel with variation of elemental in scale. At point 4 (Fig.4b) where the oxide layer separates from substrate shows a decrease in elemental composition but at outer side i.e. at point 7. (Fig.4b) there is more ferrous oxide and at point 1 there is less oxygen but high amount of ferrous as it is substrate material. (Fig.4a) shows a very thick oxide layer is formed on surface whereas (Fig.4b) shows a very thin oxide layer on the surface. (Fig.4a), shows that up to point 3, Fe is dominated i.e. Fe is more than O, as it is substrate. But at point 3 to 5, O is increasing & Fe is decreasing and point 5 to 6, O is less Fe is more, due to crack and porous.

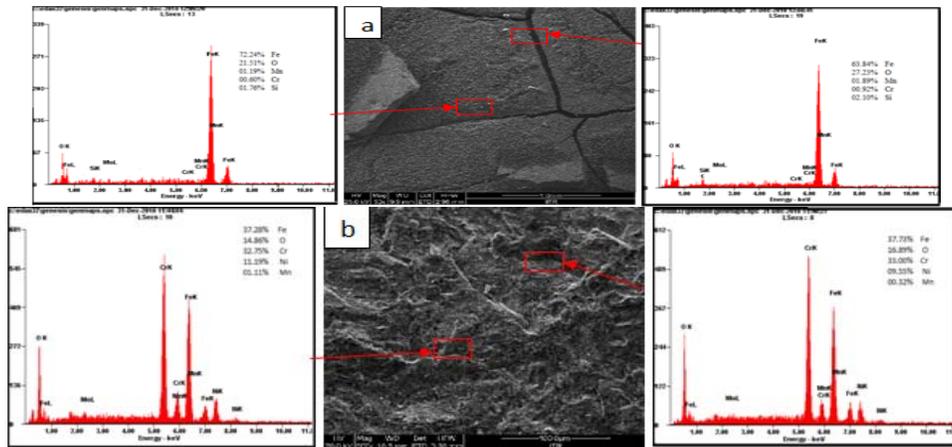


Fig: 3 (a-b) Surface scale morphology and EDAX analysis (wt. %) for low alloy steel and AISI304 steel subjected to cyclic air oxidation at 900°C.

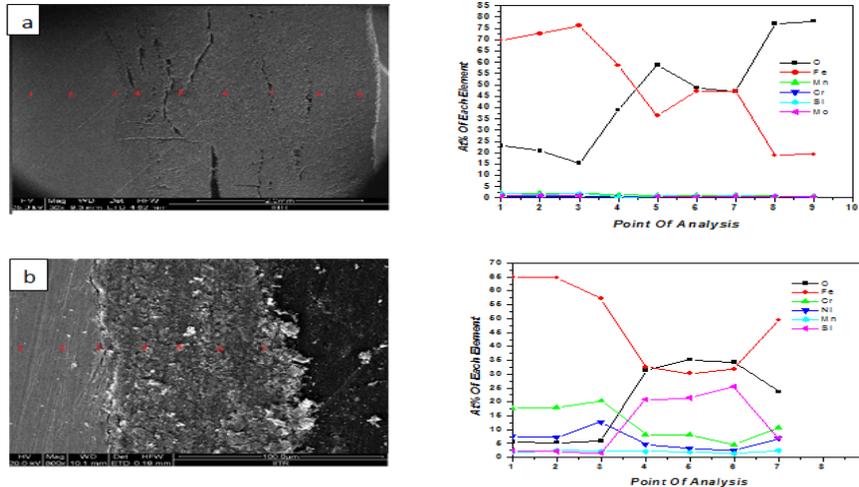


Fig: 4 (a-b) Distribution of element across FE-SEM/EDS Cross- Section of low alloy steel and AISI 304 steel subjected to air oxidation at 900°C. Point 7 to 9, O is more Fe is less, while (Fig.4 b) shows that up to point 5, O is more Fe is less and from point 5 to 7 Fe is more O is less, it is due to fragmentation of scale into the resin mounting.

5. DISCUSSION

Fig.1 (a-b) shows that oxidation resistance property of AISI 304 steel is better than low alloy steel. Internal oxidation further led to the cracking of the scale due to the mismatch in coefficient of thermal expansion in oxides scale from that of coating as suggested by P. Niranatlumpong [8]. As there are various elements and each have different thermal coefficient of expansion hence there will be more stress generated which will lead to more cracking. Through these cracks, corrosive gases can penetrate to the base material and will thus allow significant grain boundary corrosion attack [9-11]. In spite of cracking, in some cases, little weight gain has been recorded. Extrusion of materials from beneath and oxide protrusions are believed to be due to the greater specific volume of oxides similar to the findings of N.S. Bornstein [12]. The better oxidation resistance of AISI 304 steels may also be attributed to the less amount of formation of a NiO layer in the scale. During EDS analysis it was observed that, very less amount of NiO layer was seen in the oxide scale of AISI 304 steel as compared to spinel phase. This layer has been suggested to be loose- structured by X. Wu [13], which may not be able to provide effective protection.

6. CONCLUSION

The cyclic oxidation behaviour of low alloy steel follow linear where as AISI 304 steel shows parabolic nature. In low alloy steel hematite (Fe_2O_3) formed at top surface with less amount of Cr_2O_3 , where as in AISI304 steel Spinal is formed which is

more protective layer. This shows that corrosion resistance of low alloy steel is less than AISI304 steel. The K_p value of AISI304 steel is $0.234 \times 10^{-10} \text{ g}^2 \text{ cm}^{-4} \text{ s}^{-1}$ much low as compared low alloy steel $2.915 \times 10^{-10} \text{ g}^2 \text{ cm}^{-4} \text{ s}^{-1}$. AISI304 steel is found to be superior than low alloy steel and as per the cross section morphology the scale thickness of AISI304 steel is very thin as compared to low alloy steel.

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