Table of Content

	Page No.
List of Figures	v
List of Tables	xi
List of Symbols	xiii
Preface	XV
Chapter-1 Introduction and Literature Survey	1
1.1 Introduction	1
1.2 Why nitrogen instead of nickel in austenitic stainless steel?	2
1.3 Development of Nickel Free Austenitic Stainless Steel1.3.1 Role of Alloying Elements1.3.2 Phases in High Nitrogen Austenitic Stainless Steel1.3.3 Production Route	4 5 7 8
1.4 Applications of Fe-Cr-Mn-N Alloys1.4.1 Biomedical Applications1.4.2 Other Applications	9 10 12
 1.5 High Temperature Oxidation 1.5.1 Thermodynamics of Oxidation 1.5.2 Oxidation Kinetics 1.5.3 Oxidation of Fe-Cr-Mn-N Austenitic Stainless Steel 1.5.4 Oxidation Under Moist Air Environment 	13 14 15 16 17
1.6 Metal Dusting	19
1.6.1 Thermodynamics of Metal dusting1.6.2 Mechanism of Metal dusting1.6.3 Metal Dusting of Various High Temperature Alloys1.6.4 Role of Oxide Layer	19 20 22 23
1.7 Solid Particle Erosion1.7.1 Effect of Erosion Parameters1.7.2 Erosion of Austenitic Stainless Steel	24 25 28
1.8 Aqueous Corrosion	30
1.9 Motivation	32
1.10 Scope of Work	32
1.11 Objective of Work.	33
Chapter-2 Material and Methods	35
2.1 Introduction	37
2.2 Material	37

 2.3 Experimental Methods 2.3.1 Oxidation Test 2.3.2 Metal Dusting 2.3.3 Solid Particle Erosion Test 2.3.4 Corrosion Test 	38 38 39 42 44
2.4 Characterization Techniques	45
Chapter-3 Oxidation Behavior of Fe-18Cr-21Mn-0.65N Austenitic Stainless Steel	47
3.1. Introduction	49
 3.2 Results 3.2.1 Visual Observation 3.2.2 Oxidation Kinetics 3.2.3 XRD Analysis 3.2.4 Morphology of Oxidized Surface and Cross section 3.2.5 Precipitation behavior 	50 50 51 54 55 61
3.3 Discussion3.3.1 Oxidation Behavior3.3.2 Precipitation of Cr₂N	64 64 67
3.4 Conclusions	67
Chapter-4 Metal Dusting Behavior of Fe-18Cr-21Mn-0.65N Austenitic Stainless	
Steel	49
4.1. Introduction	71
 4.2. Results 4.2.1 Visual Observation and Weight Gain Analysis 4.2.2 XRD Analysis 4.2.3 Surface Morphology 4.2.4 Cross Sectional Analysis 4.2.4.2 EPMA Analysis 	72 72 72 74 79 83
 4.3. Discussion 4.3.1 Regime I: Metal Dusting Features at 400-500°C 4.3.2 Regime II: Metal dusting cum Oxidation at 600-700°C 	86 86 90
4.4. Conclusions	93
Chapter-5 Erosion Behavior of Fe-18Cr-21Mn-0.65N Austenitic Stainless Steel	95
5.1 Introduction	97
 5.2. Results 5.2.1 Weight Loss Analysis 5.2.2 Erosion rate 5.2.3 Hardness Profile 5.2.4 Tensile Behavior 5.2.5 Surface Morphology 5.2.6 Cross Sectional Analysis 	98 98 100 100 102 104 106

5.3. Discussion5.3.1 Effect of Oxidation	108 109
5.3.2 Effect of Temperature	111
5.3.3 Effect of Impact Angle	113
5.4 Comparison with Literature	115
5.5 Conclusions	117
Chapter-6 Potentiodynamic Corrosion Behavior of Fe-18Cr-21Mn-0.65 Stainless Steel	N Austenitic 119
6.1 Introduction	121
 6.2. Results 6.2.1 EIS analysis 6.2.2 Polarization Test 6.2.3 Surface layer analysis 	122 122 126 127
 6.3. Discussion 6.3.1 EIS Study 6.3.2 Potentiodyanmic Polarization Test 6.3.3 Surface layer analysis 6.3.4 SEM-EDS analysis 6.3.5 Mechanism of Corrosion 	137 138 138 139 142 144
6.4. Conclusions	146
Chapter-7 Summary and Suggestion for Future Work	149
7.1 Introduction	151
 7.2 Summary 7.2.1 Oxidation Behavior 7.2.2 Metal Dusting 7.2.3 Solid Particle Erosion Behavior 7.2.4 Potentiodynamic Corrosion Behavior 	151 151 151 152 152
7.3 Suggestions for Future Work:	153
References	155
Glossary of Words	169
Appendices	173
List of Publications	183

List of Figures

	Page No.
Figure 1.1: Schaeffler diagram.	5
Figure 1.2: Schematic representation of metal dusting mechanisms.	21
Figure 2.1 (a) Optical microstructure and (b) X-ray diffraction of Fe-18Cr-	
21Mn-0.65N austenitic stainless steel in solution annealed	
condition.	38
Figure 2.2: (a) Photograph of the oxidation test set up (b) inside view of two	
zone split tube furnace, and (c) Axis digital balance.	39
Figure 2.3: Experimental setup for metal dusting test.	41
Figure 2.4: Photograph of Ducom air jet erosion tester.	43
Figure 2.5: (a) SEM micrograph of alumina particles and (b) Particle size	
distribution of erodent particles (Al ₂ O ₃).	44
Figure 2.6: Photograph of CorrTest electrochemical work station, with flat	
type corrosion cell.	44
Figure 3.1: Photographs of the samples oxidized at 400-700°C for 100 h in	
static and dynamic air.	50
Figure 3.2 : ΔW vs time plots for oxidation at 400-700°C up to 100 h in (a)	
static air, (b) dynamic air-2 lpm, and (c) dynamic air-6 lpm.	51
Figure 3.3 : ΔW^2 vs time plots for oxidation at 400° to 700°C up to 100 h in	
(a) static air, (b) dynamic air-2 lpm, and dynamic air-6 lpm.	52
Figure 3.4: Plots for determination of activation energy for oxidation at 400-	
700°C under Static and Dynamic air conditions.	53
Figure 3.5: XRD patterns of the oxidized Fe-18Cr-21Mn-0.65N austenitic	
stainless steel at 400-700°C, up to 100 h in (a) static air and (b)	
dynamic air (6 lpm).	54
Figure 3.6: SEM micrographs and elemental analysis of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel oxidized at (a)400, (b)500, (c)600,	
and (d)700 °C for 100 h in dynamic air (6 lpm).	56
Figure 3.7: SEM micrographs and elemental analysis of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel oxidized at (a) 400°C, (b)500°C,	
(c), 600°C and (d)700 °C up to 100 h in static air.	58

Figure 3.8: SEM-EDS point analysis of cross section of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel oxidized up to 100 h at different	
temperatures; (a, b) 400°C, (c, d) 500°C, (e, f) 600 °C and (g, h)	
700 °C in dynamic-61pm and static air.	60
Figure 3.9: EDS mapping of cross section of the Fe-18Cr-21Mn-0.65N	
austenitic stainless steel oxidized for 100h at: (a)400°C, (b)500°C,	
(c)600°C and (d)700°C in dynamic air-6 lpm.	60
Figure 3.10: EDS mapping of cross section of the, Fe-18Cr-21Mn-0.65N	
austenitic stainless steel oxidized for 100 h in static air at:	
(e)400°C, (f)500°C, (g)600°C and (h)700°C.	61
Figure 3.11: TEM bright field images (a, c, e, g) and corresponding diffraction	
patterns (b, d, f, h) of the samples exposed at 400, 500, 600, and	
700°C respectively, up to 100 h.	63
Figure 3.12:TTT diagram of the Fe-18 Cr-21 Mn-0.65 N austenitic stainless	
steel calculated using J-Mat Pro software.	63
Figure 3.13: Schematic of oxidation mechanism in (a) static and (b) dynamic	
air at 500-700°C.	66
Figure 4.1: Photographs of metal dusted coupons exposed for 300 h at (a)	
400°C, (b) 500°C, (c) 600°C and (d) 700°C.	72
Figure 4.2: Plots, resulting from exposure of 300 h (a) weight gain vs time	
and (b) carbon deposited with respect to temperature.	72
Figure 4.3: XRD patterns of (a) metal dusted coupons exposed at 400°C,	
500°C, 600°C, and 700°C (b) carbon deposited at the surface from	
300 h of exposure at 400°C and 500°C.	73
Figure 4.4: SEM micrographs and corresponding EDS of (a) carbon deposited	
region; (b) area of less deposition of carbon; (c) carbon filaments	
showing branched structure; (d) fragmented particle and (e) pits	
formation at the surface after carbon removal on metal dusted	
coupon exposed at 400°C for 300 h.	75
Figure 4.5: SEM micrographs and corresponding EDS of (a) carbon deposited	
region; (b) area of less deposition of carbon; (c) globular structure	
containing carbon filaments and (d) surface showing formation of	
pits on metal dusted coupon exposed at 500°C for 300 h.	77

Figure 4.6: SEM micrographs and corresponding EDS of (a) oxide layer	
formed at the surface, (b) magnified image of the selected area (red	
rectangle) showing crystal formation of metal dusted coupon	
exposed at 600°C for 300 h.	78
Figure 4.7: SEM micrographs and corresponding EDS of metal dusted coupon	
exposed at 700°C for 300 h: (a) morphology of oxide layer formed	
at the surface, (b) magnified image of the selected area (red	
rectangle) showing needle-like structure formation.	79
Figure 4.8: SEM micrographs and corresponding EDS of (a) cross-section of	
metal dusted coupon exposed at 400°C for 300 h showing carbon	
deposition in filament form, (b) BSE micrograph showing pit	
depth.	80
Figure 4.9: SEM micrographs and corresponding EDS of (a) cross-section of	
metal dusted coupon exposed at 500°C for 300 h showing carbon	
deposition in filament form, (b) BSE micrograph showing pit	
depth.	81
Figure 4.10: SEM-BSE micrograph and EDS analysis showing two-layered	
structure of oxide scale and oxide/carbide region of metal dusted	
coupon exposed at 600°C for 300 h.	82
Figure 4.11: SEM-BSE micrograph and EDS analysis showing two-layered	
structure of oxide scale and oxide/ carbide region in the metal	
dusted coupon exposed at 700°C for 300 h.	83
Figure 4.12: EPMA area mapping of metal dusted coupon exposed for 300 h	
at (a) 400, (b) 500, (c) 600, and (d) 700°C respectively.	85
Figure 4.13: Schematic diagram showing mechanism of metal dusting from	
exposure at 400 and 500°C.	89
Figure 4.14: Schematic diagram showing the mechanism of metal dusting from	
exposure at 600 and 700°C.	93
Figure 5.1: Weight loss vs time plots of Fe-18Cr-21Mn-0.65N austenitic	
stainless steel eroded at (a) RT, (b) 400°C, (c) 500°C, (d) 600°C	
and (e) 700°C at three impact angles of 60, 75° and 90°, solution	
treated, pre-exposed at respective temperatures of erosion, from	
RT to 700°C.	99

Figure 5.2: Plots showing erosion behavior of the Fe-18Cr-21Mn-0.65N	
austenitic stainless steel: (a) erosion rate vs temperature, (b)	
erosion rate vs angle of impact.	100
Figure 5.3: Microhardness vs depth plot of Fe-18Cr-21Mn-0.65N austenitic	
stainless steel eroded at (a) room temperature, (b) 400°C, (c)	
500°C, (d) 600°C and (e) 700°C.	101
Figure 5.4: Tensile behavior of the Fe-18Cr-21Mn-0.65N austenitic stainless	
steel, solution treated and pre exposed from 400°C to 700°C for	
100 h, and tested at the respective temperature of pre-exposure: (a)	
engineering stress strain curves and (b) true stress strain plots.	103
Figure 5.5: SEM micrographs of the areas of the Fe-18Cr-21Mn-0.65N	
austenitic stainless steel, eroded at: room temperature, 400°C,	
500°C, 600°C and 700°C.	104
Figure 5.6: SEM micrographs of cross section of eroded scar showing the	
eroded crater profile of 18Cr-21Mn-0.65N-Fe austenitic stainless	
steel at RT, 400°C, 500°C, 600°C and 700°C.	106
Figure 5.7: SEM micrographs of cross section of areas of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel, eroded at: room temperature,	
400°C, 500°C, 600°C and 700°C.	108
Figure 5.8: XRD pattern of the Fe-18Cr-21Mn-0.65N austenitic stainless steel	
exposed at different temperature during erosion test.	109
Figure 5.9: BSE images of cross section of the Fe-18Cr-21Mn-0.65N	
austenitic stainless steel pre oxidized for 100 h (a) 600°C and (b)	
700°C.	110
Figure 5.10: Schematic diagrams showing mechanism of erosion of the Fe-	
18Cr-21Mn-0.65N austenitic stainless steel, at impact angles of (a)	
60, 75, and (b) 90 at 600 and 700°C.	110
Figure 6.1: (a) Nyquist and (b, c) Bode plots of Fe-18Cr-21Mn-0.65N	
austenitic stainless steel samples, unexposed and exposed for 100	
h at 400-700°C.	123
Figure 6.2: Equivalent circuit diagram used for fitting EIS data of Fe-18Cr-	
21Mn-0.65N austenitic stainless steel samples unexposed and	
exposed at 400-700°C for 100 h.	124

Figure 6.3: Potentiodynamic polarization plots of the Fe-18Cr-21Mn-0.65N	
austenitic stainless steel samples: (a) unexposed, and exposed at	
(b) 400°C, (c) 500°C, (d) 600°C, (e) 700°C for varying duration.	125
Figure 6.4: XRD patterns of the unexposed sample and the samples exposed	
at 400-700°C for 100 h.	128
Figure 6.5: XPS plots of Mn 2p _{3/2} after polarization test of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel samples (a)unexposed, and exposed	
for 100h at (b) 400°C, (c) 500°C (d) 600°C and (e) 700°C.	129
Figure 6.6: XPS plots of Fe 2p _{3/2} after polarization of Fe-18Cr-21Mn-0.65N	
austenitic stainless steel samples (a) un <i>expos</i> ed and exposed at (b)	
400°C, (c) 500°C (d) 600°C and (e) 700°C for 100 h.	130
Figure 6.7: XPS plots of Cr 2p _{3/2} after polarization test of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel samples (a)unexposed, and exposed	
at (b) 400°C, (c) 500°C (d) 600°C and (e) 700°C for 100 h.	131
Figure 6.8: XPS plots of O 1s after polarization test of Fe-18Cr-21Mn-0.65N	
austenitic stainless steel samples (a) unexposed and those exposed	
at (b) 400°C, (c) 500°C (d) 600°C and (e) 700°C for 100 h.	132
Figure 6.9: SEM-EDS analysis of corroded surface of Fe-18Cr-21Mn-0.65N	
austenitic stainless steel samples, (a) unexposed and those exposed	
at (b) 400°C, (c) 500°C (d) 600°C, (e&f) 700°C for 100 h.	134
Figure 6.10: SEM micrographs of longitudinal cross sections, normal to	
corroded surfaces of the Fe-18Cr-21Mn-0.65N austenitic stainless	
steel, (a) unexposed and exposed for 100 h: at (b) 400°C, (c) 500°C	
(d) 600°C and (e) 700°C.	137
Figure 6.11: SEM micrographs of surface of the Fe-18Cr-21Mn-0.65N	
austenitic stainless-steel samples exposed for 100 h at: (a) 600°C	
and (b) 700°C.	137
Figure 6.12: Schematic mechanism of corrosion for the Fe-18Cr-21Mn-0.65N	
austenitic stainless steel samples, (a) unexposed, and exposed at	
400-500°C, (b) exposed at 600-700°C for 100 h.	145

List of Tables

	Page No.
Table 1.1: Comparison of mechanical properties of different austenitic	
stainless steels.	3
Table 2.1: Carbon activity and partical pressure of oxygen at corresponding	
temperature.	40
Table 2.2 : Physical properties of aluminum oxide (Al ₂ O ₃) erodent.	42
Table 2.3: Operating conditions for solid particle erosion test.	43
Table 3.1: Values of exponent 'n'.	52
Table 3.2 : Weight gain per unit area (ΔW) and parabolic rate constant (k_p).	53
Table 3.3: Phases formed at different temperatures, characterized by XRD.	54
Table 3.4 : Diffusion coefficient of cations through chromia layer.	65
Table 4.1: Phases identified by XRD analysis after metal dusting process at	
different temperatures.	73
Table 4. 2: Gibbs free energy (kcal/mol K ⁻¹) of formation of various carbides	
and oxides.	87
Table 5.1 . Erosion rate (ER) at different impact angles and temperatures.	99
Table 5.2: Tensile properties of the Fe-18Cr-21Mn-0.65N austenitic stainless	
steel, solution treated and exposed from 400°C to 700°C for 100 h,	
and tested at the respective temperature of pre-exposure.	103
Table 5. 3: Bulk hardness of the Fe-18Cr-21Mn-0.65N austenitic stainless-	
steel specimens of cross section, from the center region at room	
temperature, solution treated and exposed from 400°C to 700°C for	
100 h, eroded at the respective temperature of pre-exposure.	103
Table 5.4: Strain hardening and strength coefficient of the Fe-18Cr-21Mn-	
0.65N austenitic stainless steel, solution treated and exposed from	
400 to 700°C for 100 h and tested at the respective temperature of	
pre-exposure.	103
Table 5.5: Effect of the angle of impact on the depth of erosion scar after	
erosion at RT, 400, 500, 600 and 700°C.	114
Table 5.6. Comparison of erosion rate of various nickel containing austenitic	
stainless steels.	116

Table 6.1: Electrochemical impedance spectroscopy (EIS) fitted parameters of	
Fe-18Cr-21Mn-0.65N austenitic stainless steel samples unexposed	
and exposed at 400-700°C.	123
Table 6.2: Polarization test parameters of Fe-18Cr-21Mn-0.65N austenitic	
stainless steel samples unexposed and exposed at 400-700°C for	
different time intervals.	124