

TABLE OF CONTENT

Contents

LIST OF ABBREVIATIONS AND SYMBOLS	xvi
PREFACE	xx
LIST OF FIGURES	xxiii
LIST OF TABLES	xxviii
CHAPTER 1	1
INTRODUCTION	1
Objective of the current work	9
Novelty statement for present work.....	11
CHAPTER 2	13
LITERATURE REVIEW.....	13
2.1 Composites	13
2.1.1 Classification of composites	13
2.1.2 Classification based on reinforcement.....	15
2.2 Types of reinforcement used in metal matrix composites	16
2.3 Metal matrix composites and their importance	17
2.4 A review of aluminium metal matrix composites	22
2.4.1 Silicon carbide reinforced AMC	22
2.4.2 Fly ash reinforced AMC	23
2.4.3 Al ₂ O ₃ reinforced AMC.....	25
2.4.4 SiC and Al ₂ O ₃ reinforced hybrid AMC	26
2.4.5 Albite reinforced AMC	27
2.4.6 Aluminum nitride reinforced AMC	27
2.4.7 Carbon nanotube reinforced AMC.....	28
2.4.8 Whisker and short fibre-reinforced AMC	29
2.5 Synthesis and characterization methods of ABO whisker by various routes:	29
2.6 Aluminum matrix composites reinforced with Al ₁₈ B ₄ O ₃₃ whisker-framework.....	32
2.7 Aluminium metal matrix composites reinforced with coated alumina borate whiskers	34
2.8 Dry sliding and wear behaviors of aluminum metal matrix composites	36
CHAPTER 3	43

EXPERIMENTAL PROCEDURE.....	43
3.1 Material selection for the synthesis of ABO _w as reinforcement	43
3.1.1 Synthesis of ABO _w as reinforcement	43
3.1.2 Sample preparation for various testing	44
3.2 Material selection for coating of ABO powder.....	44
3.2.1 Coating of ABO powder	45
3.2.2 Sample preparation for various testing	46
3.3. Development of ABO _w reinforced in aluminium metal matrix composite by powder metallurgy process	47
3.3.1 Material selection and fabrication process for composite.....	47
3.3.2 Sample preparation for various testing	48
3.4. Development of SiO ₂ coated ABO _w reinforced in aluminium metal matrix composite by powder metallurgy process.....	48
3.4.1 Material selection and fabrication process for composite.....	48
3.4.2 Sample preparation for various testing	49
3.5 Development of SiO ₂ coated ABO _w reinforced in aluminium (319) alloy composite synthesized by compo-casting.....	49
3.5.1 Material selection for the matrix of the composite	49
3.5.2 Fabrication process of composites by compo-casting	49
3.6 Characterization	51
3.6.1 X-ray diffraction analysis of composites	51
3.6.2 Microstructural characterization of reinforcement and composites.....	52
3.7 Density measurement of reinforcement and composites	53
3.8 Thermal Analysis of the ceramic whiskers/reinforcement.....	54
3.8. Differential thermal analysis (DTA/DSC) and Thermo-gravimetric analysis (TGA).....	54
3.9 Hardness measurement of composites	54
3.10.1 Measurement of tensile strength of base alloy and composites.....	55
3.10.2 Measurement of flexural strength	56
3.10.3 Measurement of compressive strength of base aluminium and composites	56
3.10.4 Measurement of mechanical strength by non-destructive testing.....	57
3.11 Dry sliding friction and wear testing of materials.....	58
CHAPTER 4	61
Synthesis and Characterization of ‘Whiskers’	61

4.1 Morphological study of reinforcing whiskers.....	62
4.2. Physical properties (density/Porosity) of sintered samples	66
4.3. Thermal analysis	68
4.4 Flexural strength analysis	69
4.5 Chapter summary.....	71
CHAPTER 5	73
Synthesis and Characterization of Coated Alumina Borate Whiskers.....	73
5.1. Morphological study of coated whiskers	74
5.1.1 HRSEM analysis of coated composites.....	74
5.2 Phase evaluation of the coated samples.....	77
5.3 Water Absorption behaviour of the sintered sample.....	79
5.4. Thermo-Mechanical analysis of alumina boron refractories	80
5.5 Fractograph of MOR sintered samples	81
5.6 Fractograph of HMOR sintered samples	83
5.7 Chapter summary.....	85
CHAPTER 6	87
Microstructure, Mechanical and Wear Properties of ABO _w -Al Matrix Composites	87
6.1.Morphology and phase identification of ABO whiskers	87
6.2 Microstructural characterization and phase identification of composites	89
6.3. Measurement of density and porosity.....	91
6.4. Evaluation of mechanical properties	92
6.4.1. Vickers hardness and bending strength.....	92
6.4.2 Compression behaviour	93
6.5 Fracture characteristics	95
6.5.1 Fractography of bending failures.....	95
6.5.2. Fractography of compressive failures.....	96
6.6 Strengthening mechanisms	98
6.7 Dry sliding wear behaviour	100
6.8. Chapter summary.....	104
CHAPTER 7	107
Microstructure and Mechanical Behaviour of SiO ₂ Coated ABO _w -Al Composites	107
.....	107
7.1 Phase identification and microstructural examination.....	108

7.1.1 Phase identification of fabricated composites.....	108
7.2.2. Electron microscopy	108
7.2. Porosity analysis	111
7.3 Mechanical characterization	112
7.3.1 Vicker’s hardness and flexural strength	112
7.3.2 Diametral compression behaviour and Young’s modulus.....	113
7.4. Fracture surface analysis.....	116
7.4.1 Fractograph of flexural test.....	116
7.4.2 Fractograph of diametral compression test.....	117
7.5 Chapter summary	119
CHAPTER 8	121
Mechanical and Wear Properties of Compo-Cast SiO ₂ Coated ABO _w / Al-319 Composites.....	121
.....	121
8.2. Microstructure of the fabricated composites	124
8.2.1 Scanning electron microscopy	124
8.2.2 Transmission electron microscopy.....	126
8.3 Mechanical properties of fabricated ccomposites.....	128
8.3.1 Tensile properties	128
8.3.2 Fracture morphology of composites	130
8.4. Dry sliding wear behaviour.....	134
8.4.1 (a) Effect of sliding distance on weight loss-.....	134
8.4.2 (b) Effect of sliding distance.....	135
8.4.3 (c) Effect of applied load	136
8.4.4 Analysis of wear and friction	137
8.4.5 Characterization of the worn surfaces.....	138
8.5 Scratching, wear, and fabrication studies.....	140
8.6 Chapter summary	142
CHAPTER 9	145
SUMMARY AND FUTURE WORKS.....	145
9.3 Suggestions for future work.....	150
REFERENCES.....	153
List of Publication.....	170
National/International Conferences/Workshop Contributions.....	171

LIST OF FIGURES

Figure 1.1: Electric car body structure	2
Figure 1.2: Typical car-body of high-speed train made of aluminum alloy	3
Figure 1.3: Model of an aluminium alloy axle box	4
Figure 1.4: Model of aluminium alloy petrol boat	5
Figure 1.5: Model of 2024 aluminium alloy aircraft	6
Figure 1.6: Different parts of a flat-plate collector	7
Figure 2.1: Classification of composite materials based on the matrix used	14
Figure 2.2: Classification of composites basis of reinforcement	15
Figure 2.3: Type of reinforcements used in MMCs	17
Figure 3.1: Process flow chart of development of ABO reinforcement	44
Figure 3.2: (a) Mixing (b) Oven (c) Furnace (d) Sonication (e) Heating (f) coated ABO _w	46
Figure 3.3: : Process flow chart of development of oxides coated ABO _w powder	47
Figure 3.4: (a) Schematic of the experimental set-up used (b) pictorial view of compo- casting techniques	50
Figure 3.5: Schematic and pictorial measurement of a tensile test specimen	55
Figure 3.6: (a) DCS test set-up (b) schematic measurement of DCS test specimen	57
Figure 3.7: (a) Pin-on-disc type tribometer (b) schematic and diagram of the wear test specimen	58
Figure 4.1: SEM micrographs of samples sintered at (a) 950 °C, (b) 1100 °C, (c)1200 °C, (d)1300 °C and (e)1400 °C. Encircled area in 1400 °C sample shows agglomerate formation and melting of whiskers	62
Figure 4.2: EDAX of sample sintered at 1300 °C	63
Figure 4.3: Average particle length at various sintering temperatures	63
Figure 4.4: XRD patterns of samples sintered at (a) 950 °C, (b)1100 °C,	65

(c)1200 °C, (d)1300 °C, and (e)1400 °C.

Figure 4.5: (a) Bright field Image (b) corresponding SAED of ABO _w powder sintered at 1300 °C (c) bright-field image and corresponding SAED (inset) of network of ABO _w powder sintered at 1400 °C (d) magnified bright field image of particle from fig.4.5c and corresponding SAED (inset)	66
Figure 4.6: DSC and TG curves of alumina borate mixture	68
Figure 4.7: Room temperature and high temperature (700°C) flexural strength with corresponding bonding temperature	70
Figure 5.1: HRSEM image of ABO _w powder coated with (a) Cr ₂ O ₃ (b) SiO ₂ (c) ZnO (d) TiO ₂	74
Figure 5.2:(a) The EDAX spectrum of Cr ₂ O ₃ coated surface of ABO Whiskers	75
Figure 5.2:(b) The EDAX spectrum of SiO ₂ coated surface of ABO _w whiskers	76
Figure 5.2: (c) The EDAX spectrum of SiO ₂ coated surface of ABO _w whiskers	76
Figure 5.2 (d) The EDAX spectrum of TiO ₂ coated surface of ABO _w whiskers	77
Figure 5.3: (a) XRD patterns of samples calcined at 500 °C for 4 h	78
Figure 5.3: (b) XRD patterns of samples calcined at 1100 °C for 1 h	78
Figure 5.4: Physical properties (density/porosity) of coated samples	80
Figure 5.5: Fractograph of modulus of rupture at room temperature of (a) Cr ₂ O ₃ (b) SiO ₂ (c) ZnO (d) TiO ₂ coated samples	82
Figure 5.6: Fracture surface after the HMOR test at 1000 °C of (a) Cr ₂ O ₃ (b) SiO ₂ (c) ZnO (d) TiO ₂ coated samples	83
Figure 5.7: Fracture surface after the HMOR test at 1000 °C room temperature of (a) Cr ₂ O ₃ (b) SiO ₂ (c) ZnO(d) TiO ₂ coated samples	84
Figure 6.1: SEM image of ABO whiskers calcined at 1300°C used as reinforcement	88
Figure 6.2: XRD pattern of ABO whisker calcined at 1300°C	88
Figure 6.3: (a) TEM bright field image and (b) corresponding SAED of ABO whiskers	89
Figure 6.4: SEM micrographs of (a) aluminum base metal and Al-ABO _w composite with (b) 5 wt.% ABO _w whiskers; (c) 10 wt.% ABO _w whiskers and (d) 15 wt.% ABO _w whiskers	90
Figure 6.5: XRD patterns of sintered Al-ABO _w composites with different compositions	91

Figure 6.6: Engineering stress-strain curves of base metal and composites with different percentages of reinforcements	94
Figure 6.7: Macrographs showing longitudinal surface cracks during compressive failure of aluminium base metal and sintered Al-ABO _w composites	94
Figure 6.8: Typical fractography in bending test of (a) Al-10 wt.% ABO _w ; (b) Al- 15 wt.% ABO _w ; (c) EDS analysis point of Al-10% ABO _w ; (d) EDS spectrum from dislodged ABO _w	96
Figure 6.9: Fractography at low magnification of (a) aluminium base metal and composites with (b) 5 wt.% ABO _w (c)10 wt.% ABO _w and (d)15 wt.% ABO _w	97
Figure 6.10: Fractography at higher magnification of (a) base aluminium and composites with (b) 5 wt.% ABO _w (c)10wt.% ABO _w and (d)15 wt.% ABO _w .	97
Figure 6.11: Variation in wear rate with sliding distance (time) for different wt.% ABO _w in Al matrix composites	101
Figure 6.12: SEM images of worn surfaces of (a) base aluminum and composites with (b) 5 wt.% ABO _w (c) 10 wt.% ABO _w and (d)15wt.% ABO _w respectively	101
Figure 6.13: Line analysis perpendicular to wear track and its 2D-profilometric images of (a) base aluminium and composites with (b) 5wt.% ABO _w (c) 10 wt.% ABO _w and (d) 15wt.% ABO _w respectively	102
Figure 6.14: 3D-profilometric images of (a) base aluminium and composites with (b) 5, (c) 10 and (d) 15 wt.% ABO _w	103
Figure 6.15: Effect of sliding distance (time) on co-efficient of friction for different percentages of ABO _w in the composites	103
Figure 7.1: XRD patterns of (a) SiO ₂ coated composite sintered at 600° C for 2 h with different composition	108
Figure 7.2: SEM micrographs of (a) Pure Al (b) composite with SiO ₂ coated 5 wt% ABO _w whiskers (C1) (c) composite with SiO ₂ coated 10 wt% ABO _w whiskers (C2) (d) composite with SiO ₂ coated 15 wt% ABO _w whiskers (C3) sintered at 600°C	109
Figure 7.3: (a) Bright field Image of coated ABO _w and higher magnification ABO _w (inset) (b) bright field image of ABO _w /Al and corresponding SAED (inset) of composite sintered at 600°C	110
Figure 7.4: HRSEM image of reinforcement (a) calcined at 1300°C without coated ABO _w corresponding single whisker (inset) (b) coated ABO _w calcined at 1100 °C(c) corresponding EDS of uncoated whisker (d)	111

corresponding EDS of coated whisker

Figure 7.5: Measurement of flexural strength and hardness of base Al and composites sample sintered at 600°C 113

Figure 7.6: Diametral compressive strength of sintered sample at 600°C base Al and composites 114

Figure.7.7: The SEM micrograph of fractured surface (a) Pure aluminium (b) composite with SiO₂ coated 5 wt% ABO_w (C1) (c) 10 wt% ABO_w (C2) (d) 15 wt %ABO_w (C3) sintered at 600°C 116

Figure 7.8: Photographs of the sintered pallet at 600°C of (a) different composition (b) before diametral compression test (c) compressed pallet (d) Fractured pallet after the diametral compression test 118

Figure 7.9: The SEM micrograph of factured surface after DCS test (a) composite with SiO₂ coated 5wt% ABO_w (C1) (b) 10 wt % ABO_w (C2) (c) 15 wt% ABO_w (C3) (d) High magnification SEM image of SiO₂ coated whisker 118

Figure 8.1: (a) XRD spectra of uncoated and SiO₂ coated ABO_w at 1100°C for 4h 122

Figure 8.1: (b) Composite samples with different volume fraction of SiO₂ coated ABO whiskers 123

Figure 8.2: SEM micrographs of (a) uncoated and (b) SiO₂-coated ABO whiskers respectively (c) High-resolution SEM of SiO₂ coated whisker (d) EDS of SiO₂ Coated ABO_w 124

Figure 8.2 (e) Mapping of SiO₂ coated ABO_w reinforced Al-319 composite 125

Figure 8.3: TEM micrographs of interface of (a) composite sample S2 (b) ABO_w in Al-319) matrix (c) Corresponding SADP of composite (d) SADP of ABO_w in aluminium (Al-319) Composite 125

Figure 8.4: TEM micrographs of interface of (a) composite sample S2 (b) SiO₂ coated ABO_w in Al-319 matrix (c) corresponding SADP of composite (d) SADP of SiO₂ coated ABO_w in aluminium Composite 126

Figure 8.5: (a) Engineering stress –strain curve Al-319 base alloy and ABO_w/Al-319 composites 128

Figure 8.5: (b) Engineering stress –strain curve Al-319 alloy and SiO₂ /ABO_w/Al-319 composites 129

Figure 8.6: Tensile fractograph of the as cast Al-319 base alloy with EDS spectrum in a specific region 130

Figure 8.7: Tensile fractograph of the ABO_w/Al-319 composites containing (a) 2.5% ABO_w (b) 5% ABO_w (c) 7.5% ABO_w (d) 10% ABO_w 131

Figure 8.8: Higher magnification (10000x) tensile fractrograph of the encircled region ABO _w /Al-319 composites (From Figure 8.6) containing (a) 2.5% ABO _w (b) 5% ABO _w (c) 7.5% ABO _w (d) 10% ABO _w	131
Figure 8.9: Tensile fractrograph of the SiO ₂ /ABO _w /A-319 composites containing (a) 2.5% ABO _w (b) 5% ABO _w (c) 7.5% ABO _w (d) 10 % ABO _w	132
Figure 8.10: Higher magnification (10000x) tensile fractrograph of the encircled specific region of SiO ₂ ABO _w /Al-319 composites containing (a) 5%ABO _w (b)7.5% ABO _w without dimples (c) 7.5% ABO _w crack impediment by whisker (d) 10% ABO _w	133
Figure 8.11: (a) Variation of weight loss with the sliding distance of alloy and composite	135
Figure 8.11: (b): Variation of COF with the sliding distance of alloy and composite	136
Figure 8.11: (c) Variation of wear rate and normal load of alloy and composite	136
Figure 8.12 : SEM micrographs of worn surfaces with load variations (a) As cast (Al-319 alloy) (b) 2.5 % coated ABO _w /Al-319 (c) 5% coated ABO _w /Al-319 (d) 7.5% coated ABO _w /A319 (e) 10% coated ABO _w /Al-319	137
Figure 8.13: (a) EDS spectrum (elemental analysis) of Al- 319 alloy	138
Figure 8.13: (b) EDS spectrum in respect of SiO ₂ coated 2.5% ABO _w / Al-319 alloy	139
Figure 8.13: (c) EDS spectrum in respect of SiO ₂ coated 5.0% ABO _w / Al-319 alloy	139
Figure 8.13: (d) EDS spectrum in respect of SiO ₂ coated 7.5.% ABO _w / Al-319 alloy	140
Figure 8.14: Line analysis results perpendicular to wear track and 2D-profilometric images of (a) as cast (Al-319) and coated ABO _w containing (b) 2.5% ABO _w / (c) 5% ABO _w (d) 7.5% ABO _w (e) 10% ABO _w	141
Figure 8.15: Line analysis results perpendicular to wear track and 2D-profilometric images of (a) as cast (Al-319) and coated ABO _w containing (b) 2.5% ABO _w (c) 5% ABO _w (d) 7.5% ABO _w (e) 10% ABO _w	142

LIST OF TABLES

Table 2.1 Primary processing routes of AMCs	22
Table 3.1: List of raw material	45
Table 3.2 Compositional details of commercial aluminum (319) alloy	50
Table 4.1 Physical properties of sintered samples	67
Table 4.2: CMOR and HMOR of Sintered Samples	69
Table 5.1 Physical properties of uncoated and coated samples	79
Table 5.2: HMOR properties of the coated and uncoated sintered samples	81
Table 6.1: Densities and porosities of composite samples	92
Table 6.2: Bending strength and vickers hardness of samples	93
Table 6.3: Theoretical estimation of different strengthening effects	100
Table 7.1: Average % porosity of Al matrix and Composites	112
Table 7.2: Young's modulus of elasticity of sintered sample	115
Table 8.1: Mechanical properties of fabricated alloys	129
Table 8.2 Roughness parameters of worn surfaces of alloy and composite	141