Declaration

I hereby declare that the work presented in this Thesis titled *Experimental Investigation on Development and Structural Integrity Assessment of Glass to Metal Joint* submitted to the Indian Institute of Technology Jodhpur in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy, is a bonafide record of the research work carried out under the supervision of Dr. Rahul Chhibber.The contents of this thesis in full or in parts, have not been submitted to, and will not be submitted by me to, any other Institute or University in India or abroad for the award of any degree or diploma.

Rakesh Joshi
P14ME003

Certificate

This is to certify that the thesis titled *Experimental Investigation on Development and Structural Integrity Assessment of Glass to Metal Joint* submitted by *Rakesh Joshi* (P14ME003) to the Indian Institute of Technology Jodhpur for the award of the degree of *Doctor of Philosophy*, is a bonafide record of the research work done by him under my supervision. To the best of my knowledge, the contents of this report, in full or in parts, have not been submitted to any other Institute or University for the award of any degree or diploma.

Rahul Chhibber
Ph.D. Thesis Supervisor

Acknowledgements

First and foremost, my thanks go to my Ph.D. Thesis Supervisor, Dr Rahul Chhibber, for introducing me to the area of dissimilar material joining and for his help, patience, and motivation.

I also thank faculty and the technical staff members of Mechanical Engineering Department of IIT, Jodhpur for their support. I thank the Ministry of New and Renewable Energy (MNRE), Government of India for providing the financial assistance to carry out the experimental work during my Ph.D.

My stay at the Institute was a wonderful experience because of my labmates, my friends, Sanjoy chatterjee, Vishwa Deepak Kumar and Shivanjali Saxena.

I also thank my friend, Dr Yogesh Kumar Singla for sharing his invaluable experience at every step of my Ph.D.

I owe a lot to my parents, my elder brother, Sister-in-law and my nephew (Aadvik), who encouraged and helped me at every stage of my personal and academic life, and longed to see this achievement come true.

Rakesh Joshi

Ph.D. Student

List of Figures

Figures	*Title*	*page*
1.1	Concentrated solar power plant: (a) parabolic trough technology, (b) tower based technology, and (c) dish technology	*1*
2.1	Structure of metal and glass: **(a)** crystalline structure of metal and **(b)** amorphous nature of glass	*3*
2.2	Stress-Stain behavior of glass and metal	*3*
2.3	Glass-metal joining mechanism **(a)** with metal oxide layer **(b)** with monoxide layer **(c)** without oxide layer	*4*
2.4	Classifications of Glass-metal joints [Jacobs,2007 and Chhibber , 2014]	*5*
2.5	Application of glass-metal joining	*7*
2.6	Wettability phenomenon in term of contact angle of liquid drop	*9*
2.7	Wetting behavior with surface roughness	*9*
2.8	Structure of oxide formed after pre-oxidation (a) FeO, (b) Fe_3O_4, and (c) Fe_2O_3	*11*
2.9	**(a)** Matched and **(b)** unmatched glass-metal joint geometry	*11*
2.10	(a-d) The direction of different stresses in compression glass metal joint, (e) Stresses generated in compression glass metal joint during heating and cooling	*12*
2.11	Graphical scheme of induced corrosion in glass-metal joint	*16*
2.12	(a) Parabolic trough receiver tube, (b) Glass broken receiver tube, (c) Enlarged view of glass-metal joint Interface	*17*
2.13	The geometry of parabolic trough receiver tube	*18*
3.1	Existing Glass-metal Joint Geometry (Matched Joint geometry)	*19*
3.2	Proposed glass-metal joint geometry (Unmatched joint geometry)	*20*
3.3	Methodology adopted	*21*
4.1	Heating curve of Specimen for pre-oxidation study	*23*
4.2	Different design condition of glass–metal joint	*25*
4.3	Induction coil design modification for experimentation	*26*
4.4	Cracking of glass tube because of higher metal ring thickness	*27*
4.5	Geometry of compression glass-metal joint	*27*
4.6	Experimental procedure of glass-metal joint fabrication	*28*
4.7	(a) Experimental Set-up of stress measurement in the glass metal joint, (b) Color fringe scale to calculate (n) , (c) Scaled up view of polarimeter, (d) Rotational scale to show index marl 0-1 and (f) scale,(e, f) showing the intensity of point of interest and maximum stress intensity at the interface).	*29*
4.8	Melting of glass tube along with glass sealant using compression joint geometry	*31*
4.9	Glass pallet specimens over pre-oxidized austenitic stainless steel 304 metal specimen	*32*
4.10	Specimen mounting holder for adhesion test	*32*
4.11	Thermal conductivity experimental set-up	*33*
4.12	Glass sealant pallet after melting in furnace	*34*
4.13	Experimental phase diagram of SiO_2-B_2O_3-Na_2O glass system	*35*
4.14	Polyhedral design on the basis of mixture design methodology	*36*
4.15	Pre-oxidized metal specimen mounted on resin	*37*
4.16	Experimental set-up for vacuum leak rate measurement	*39*
4.17	A schematic of experimental setup for in-situ wettability measurement	*39*
4.18	Glass-metal joint fabrication process (a) Machined metal ring.(b) Metal ring after pre-oxidation, (c) Borosilicate glass tube, (d) Graphite jig, (e) Glass-metal joint after fabrication, (f) Metal plate, (g) pre-oxidized metal plate, (h) glass pallet, (i) glass sealant	*40*

	pallet placed over the metal specimen, (j) Glass sealant pallet melting over metal specimen.	
4.19	Heating curve of compression glass-metal joint specimen for thermal cycling testing	41
5.1	Surface morphology of metal specimens in as received condition (a), low level (b) and high level (c) of surface roughness	44
5.2	Surface Roughness variation with different grade emery paper	44
5.3	Oxide mass gain of different metal specimen at 700°C for 3 minutes	45
5.4	The oxide layer thicknesses on SS304 metal specimen with increasing surface roughness R_a (1.96-4.07µm)	45
5.5	The oxide layer thickness of copper metal specimen with increasing surface roughness R_a(1.1-3.5µm)	46
5.6	Oxide layer thickness in LCS 1018 metal specimen after pre-oxidation at 700°C at different surface roughness R_a(1.35-3.43µm)	46
5.7	XRD pattern of SS 304 in as-received ,in pre-oxidation and in joining condition	47
5.8	XRD pattern of copper in as-received ,in pre-oxidation and at joining condition	47
5.9	XRD pattern of LCS1018 in as-received ,in pre-oxidation and in joining condition	48
5.10	Contact angle and wetting area variation with surface roughness of SS304 and copper	49
5.11	(a) The top view of glass-metal joint interface,(b) Front view of failed compression glass-metal joint	49
5.12	**(a)** Polariscope "PS-100-BS" set-up **(b)** Stress calculation without tint plate.**(c)** Stress calculation with tint plate corresponding to 2mm metal thickness**(d)** Stress calculation with tint plate corresponding to 0.4 mm metal thickness	50
5.13	Residual axial stress variations with respect to metal thickness	51
5.14	Cracking of glass tube because of the higher metal ring thickness (2.5mm)	52
5.15	(a-f): Failure Condition of glass-metal joint assembly after fabrication with decrease in metal thickness	52
5.16	Variation of Thermal expansion mismatch with different metal thicknesses	53
5.17	Melting and wetting of different glass sealants	53
5.18	Adhesion strength and spreading area variation with different SiO_2/B_2O_3 ratio	54
5.19	Adhesion strength variations with respect to contact angle with different SiO_2/B_2O_3 ratio	54
5.20	XRD pattern of different glass sealant with different SiO_2/B_2O_3 ratio	55
5.21	FTIR spectra of different glass sealant with different SiO_2/B_2O_3 ratio	56
5.22	DTA curve of different glass sealant with respect to temperature and Tg, Tc variation with different SiO_2/B_2O_3 ratio	57
5.23	Percentage weight loss curve for different specimen and weight loss with different SiO_2/B_2O_3 ratio	58
5.24	Weight loss per minute with respect to temperature with different SiO_2/B_2O_3 ratio	58
5.25	Thermal conductivity of glass sealant with different SiO_2/B_2O_3 ratio	59
5.26	Scanning electron micrograph of glass sealants with respect to decreasing SiO_2/B_2O_3 ratio	59
5.27	EDAX of glass sealants with respect to decreasing SiO_2/B_2O_3 ratio	60
5.28	Elemental composition of different glass sealant with different SiO_2/B_2O_3 ratio	60
5.29	X-Ray Diffraction pattern of glass sealant specimen	61
5.30	Predicted versus actual values of various glass sealant response (a)Tg ,(b) Tc,(c) Spreading area,(d) Contact angle, (e) Density , (f) Thermal Conductivity, (g) Percentage weight loss	64
5.31	Contour surface plots for various glass sealant response (a)Tg ,(b) Tc,(c) Spreading area,(d) Contact angle, (e) Density , (f) Thermal Conductivity, (g) Percentage weight loss	64
5.32	(1-18) Scanning electron micrograph and Energy dispersive X-rays Image of glass sealant specimens	69
5.33	Adhesion strength variation with glass transition temperature (Tg)	69
5.34	Oxide layer mass gain at different pre-oxidation temperature and at different pre-oxidation time	72
5.35	Oxide layer thicknesses (µm) at pre-oxidation temperature 700°C at different pre-oxidation time interval	73

5.36	XRD pattern of SS304 metal substrate during, as-received, pre-oxidation with different pre-oxidation time and at joining condition	*74*
5.37	Oxide layer deposited on the SS304 metal specimen after pre-oxidation	*75*
5.38	Load versus extension variation of glass-metal joint during adhesion test	*76*
5.39	Adhesion strength and spreading area of glass sealant with change in oxide layer mass	*76*
5.40	Estimation of spreading area of glass sealant over metal	*76*
5.41	XRD pattern of glass-metal interface	*77*
5.42	(a) SEM micrograph of glass sealant at 5 kx, (b) SEM micrograph of glass-metal joint interface at 1 Kx (c) EDAX image of glass-metal joint interface taken corresponding to 1 KX, (d) EDAX images of glass sealant taken corresponding to 5 KX.	*78*
5.43	X-ray dot mapping at the interface of glass sealant-metal joint	*79*
5.44	Level of vacuum leak rate achieved with leak testing	*80*
5.45	In-Situ wetting behavior of glass sealant over metal substrate at different temperature	*80*
5.46	Variation of contact angle with temperature	*81*
5.47	(a-f) Residual stress distribution glass-metal joint subjected to thermal cycling for different temperature 150 °C, 200 °C and 250 °C respectively	*82*
5.48	The residual stresses variation in compression glass-metal joint subjected to thermal cycling at different temperatures	*82*
5.49	Compression glass-metal joint specimen for thermal cycling testing	*83*
5.50	Change in weight of glass-metal joint specimen subjected to different cyclic loading	*83*
5.51	(a-h) Macrograph of glass-metal joint condition after subjecting to atmospheric cycling for different time duration	*84*
5.52	(a-h) Macrograph of glass-metal joint after exposing to water environment cycling for different time duration	*85*
5.53	(a-c) Macrograph of glass-metal joint subjected to different cyclic loading temperature	*86*
5.54	Change in weight of glass-metal joint specimen subjected to different cycling loading at different temperature	*86*

List of Tables

Table	*Title*	*page*
2.1	Historical development in the area of glass-metal joint	6
2.2	Application of various glass-metal joint combinations	8
2.3	Various glass sealants and their chemical composition used in glass-metal joining	13
2.4	Various glass compositions used in the past for various application	13
2.5	Effect of glass constituent on glass sealant properties [Jean., 2009; Donald et al., 2011]	14
4.1	Chemical compositions of metal specimens	23
4.2	Induction heating setup process parameter	26
4.3	Dimension of different glass-metal joint specimen used	27
4.4	Glass sealant design matrix	31
4.5	Design matrix of glass sealant formulation	36
4.6	Design matrix of glass-metal joint for different environmental condition	41
5.1	Oxide formed during pre-oxidation process in different metal specimen	48
5.2	Assignment of different band observed in the spectra of different glass reported by researchers [Rada.et.al 2006]	56
5.3	Output response of glass sealant obtained though thermal studies	62
5.4	Wetting, spreading and density response of glass sealant	62
5.5	Chemical composition of glass sealant pallet obtained after melting	70
5.6	Error % in glass sealant compositions	71
5.7	The optimized composition of glass sealant based on required objective	71
5.8	EDAX elemental composition on SS304 specimen after pre-oxidation	75
5.9	Oxide layer mass gain and oxide layer thickness at different pre-oxidation time intervals.	75

List of Symbols

Symbol	*Description*
$\left(\frac{\Delta l}{l}\right)_t$	Total thermal expansion mismatch from room temperature to sealing temperature
μ_g	Poison ratio of glass
f	shape factor
E_m	Young modulus of glass
E_g	Young modulus of glass
t_m	Thickness of metal ring
t_g	Thickness of glass tube
δnm	Retardation
N	Fringe order
λ	Wave length
f	Fringe sacale reading
n	Lower order full fringe order
t_{mm}	Thickness of glass tube
$C_{Brewester}$	Brewster constant
d_g	Outer diameter of glass tube
l_g	Length of glass tube
d_m	Inner diameter of metal ring
t_m	Thickness of metal ring
S	Residual stress
θ	contact angle
γ_{sv}	Solid/vapor interfacial energy
γ_{sl}	Solid/liquid interfacial energy
γ_{lv}	liquid/vapor interfacial energy

List of Abbreviations

Abbreviation	*Full form*
PTRT	Parabolic trough receiver tube
MEMS	Micro-electronic Mechanical system
SOFC	Solid oxide Fuel cell
CTE	Coefficient of thermal expansion
M	Metal
O	Oxygen
SiO_2	Silica
DOE	Design of Experiment
EDAX	Energy Dispersive X ray Spectroscopy
SEM	Scanning Electron microscopy
XRD	X-Rays diffractometer
CSP	Concentrated solar power plant
CCP	Cubic close-packed
LCS	Low carbon steel
SS	Stainless steel
cu	Copper
POI	Point of interest
ICDD	International Centre for Diffraction Data
SIA	Structure Integrity Assessment
GMJ	Glass-metal joint