VIGNANA BHARATHI
Institute of Technology

(Sponsored by Swami Vivekananda Educational Trust, Hyd.)
(Approved by AICTE, Accredited by NBA, Permanently Affiliated to JNTUH)
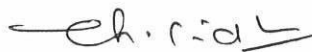
CERTIFICATE

FILE AND LINK No: MRP-6945/16 (SERO/UGC)

NAME OF THE PRINCIPAL INVESTIGATOR: Dr. Ch.S.L.N.Sridhar
Vignana Bharathi Institute of Technology,

Aushapur, Hyderabad, Pin: 501301

1. **TITLE OF THE PROJECT:** Synthesis-Structural-Dielectric and Magnetic studies on

Titanium doped nanocrystalline Manganese-Zinc Ferrites

Certified that the project has been successfully completed and Executive summary of the

report, Research documents, monograph, academic papers published under Minor research

project has been posted on the website of the college

Signature of the Principal Investigator

Signature of the Principal
With seal and stamp
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

VBIT
Aushapur (V),
Ghatkesar (M)

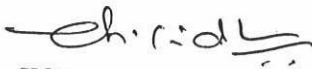## Settlement proforma

UTILISATION CERTIFICATE

FILE AND LINK No: MRP-6945/16 (SERO/UGC)

NAME OF THE PRINCIPAL INVESTIGATOR: Dr. Ch.S.L.N.Sridhar
Vignana Bharathi Institute of Technology,

Aushapur, Hyderabad, Pin: 501301

1. **TITLE OF THE PROJECT:** Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites",

1. Certified that the grant of Rs. 1,75,000/ (Rupees one lakh seventy five thousand only) approved by UGC and the grant received Rs1,62,500/(Rupees one lakh sixty two thousand five hundred only) from the University Grants Commission under the scheme of support for Minor Research Project entitled "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites", vide UGC letter No. F.MRP-6945/16 (SERO/UGC) dated 28/7/2017has been fully utilized for the purpose for which it was sanctioned and that the balance of Rs.12,5000 has been spent by institute which has to be released from UGC in accordance with the terms and conditions laid down by the University Grants Commission. If as a result of check or audit objection, some irregularity is noticed at a later stage, action will be taken to refund or regularize the objected amount.
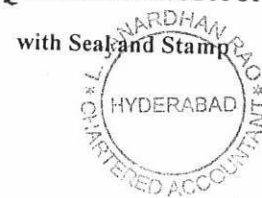
SIGNATURE OF THE

PRINCIPAL INVESTIGATOR

PRINCIPALwith Seal and Stamp

PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

STATUTORY AUDITOR
with Seal and Stamp

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

UDIN: 2018474AAAAF7145

VBIT
Aushapur (V),
Ghatkesar (M)
MEDCHAL DIST-501 301
VIGNANA BHARATHI INSTITUTE OF TECHNOLOGY

JANARDHAN RAO
HYDERABAD
CHARTERED ACCOUNTANT

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

STATEMENT OF EXPENDITURE IN RESPECT OF MINOR RESEARCH PROJECT (II YEAR)

1. Name of Principal Investigator: Dr. Ch.S.L.N.Sridhar

2. Dept. of PI: Physics

   Name of College: Vignana Bharathi Institute of Engineering & Technology

3. UGC approval Letter No. and Date: MRP-6945/16 (SERO/UGC), 28/7/2017.

4. Title of the Research Project:" Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites".

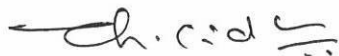5. Effective date of starting the project: 15/08/2017

6. a. Period of Expenditure: From 29-01- 2019to 07/08/2019

   b. Details of Expenditure

S.No.	Item	Amount Approved (Rs.)	Amount Received (Rs.)	Expenditure Incurred (Rs.)	Amount to be released by UGC
i.	Books & Journals	00	00	00	
ii.	Equipment	00	00	00	
iii.	Contingency including special needs	5000	4000	5000	1000
iv.	Field Work/Travel (Give details in the proform) a	7500	6000	7500	1500
v.	Hiring Services	25000	20000	25000	5000
vi.	Chemicals & Glassware	25000	20000	25000	5000
	GRAND TOTAL	62,500	50,000	62,500	12,500

7. if as a result of check or audit objection some irregularly is noticed at later date, action will be taken to refund, adjust or regularize the objected amounts.

8. It is certified that the grant of Rs. **62,500/** (Rupees sixty two thouysand five hundred only) approved by UGC and the grant received Rs **50,000** (Rupees fifty thousand only) from the University Grants Commission under the scheme of support for Minor Research Project entitled "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites", vide UGC letter No. F. MRP-6945/16 (SERO/UGC)dated 28/7/2017has been fully utilized for the purpose for which it was sanctioned and that the balance of Rs.**12,500** has been spent by institute which has to be released from UGC in accordance with the terms and conditions laid down by the University Grants Commission.

SIGNATURE OF PRINCIPAL INVESTIGATOR

PRINCIPAL
PRINCIPAL
Vignana Bharathi Institute of Technolc
Aushapur(V), Ghatkesar(M), Medchal Dist-501 3

(Seal)

Annexure - III

STATEMENT OF EXPENDITURE IN RESPECT OF MINOR RESEARCH PROJECT (CONSOLIDATED, I & II Year)

1. Name of Principal Investigator: Dr. Ch.S.L.N.Sridhar

2. Dept. of PI: Physics

Name of College: Vignana Bharathi Institute of Engineering & Technology

3. UGC approval Letter No. and Date: MRP-6945/16 (SERO/UGC), 28/7/2017.

4. Title of the Research Project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites".

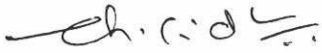5. Effective date of starting the project: 15/08/2017

7. a. Period of Expenditure: From 10/02/2018 to 07/08/2019

   b. Details of Expenditure

S.No.	Item	Amount Approved (Rs.)	Amount Received (Rs)	Expenditure Incurred(Rs.)	Amount to be released by UGC
i.	Books & Journals	10,000	10,000	10,000	00
ii.	Equipment	40,000	40,000	40,000	00
iii.	Contingency including special needs	10,000	9,000	10,000	1,000
iv.	Field Work/Travel (Give details in the proforma).	15,000	13,500	15,000	1,500
v.	Hiring Services	50,000	45,000	50,000	5000
vi.	Chemicals & Glassware	50,000	45,000	50,000	5000
	GRAND TOTAL	**1,75,000**	**1,62,500**	**1,75,000**	**12,500**

7.if as a result of check or audit objection some irregularly is noticed at later date, action will be taken to refund, adjust or regularize the objected amounts.

8.It is certified. that the grant of Rs. 1,75,000/ (Rupees one lakh seventy five thousand only) approved by UGC and the grant received Rs.1,62,500/- from the University Grants Commission under the scheme of support for Minor Research Project entitled "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites", vide UGC letter No. F. MRP-6945/16 (SERO/UGC)dated 28/7/2017has been fully utilized for the purpose for which it was sanctioned and that the balance of Rs.12,500 has been spent by institute which has to be released from UGC in accordance with the terms and conditions laid down by the University Grants Commission.

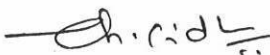SIGNATURE OF PRINCIPAL INVESTIGATOR

PRINCIPAL
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

(Seal)

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

Utilization certificate (II YEAR)

Certified that the grant of Rs. 62,500/ (Rupees sixty two thousand and five hundred only) approved by UGC and the grant received RS 50,000(Rupees fifty thousand only) from the University Grants Commission under the scheme of support for Minor Research Project entitled "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites",vide UGC letter No. F.MRP-6945/16 (SERO/UGC)dated 28/7/2017has been fully utilized for the purpose for which it was sanctionedand that the balance of Rs.12,5000 has been spent by institute which has to be released from UGC in accordance with the terms and conditions laid down by the University Grants Commission.

SIGNATURE OF THE
PRINCIPAL INVESTIGATOR (Seal)

PRINCIPAL

9-1-2020

STATUTORY AUDITOR
(Seal)

PRINCIPAL
Vignana Bharathi Institute of Technology
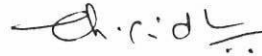Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

UDIN:20018474AAAAAF7145

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

Utilization certificate (Consolidated, I & II Year)

Certified that the grant of Rs. 1,75,000/ (Rupees one lakh seventy five thousand only) approved by UGC and the grant received Rs1,62,500/(Rupees one lakh sixty two thousand five hundred only) from the University Grants Commission under the scheme of support for Minor Research Project entitled "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites",vide UGC letter No. F.MRP-6945/16 (SERO/UGC)dated 28/7/2017has been fully utilized for the purpose for which it was sanctioned andthat the balance of Rs.12,5000 has been spent by institute which has to be released from UGC in accordance with the terms and conditions laid down by the University Grants Commission.

SIGNATURE OF THE
PRINCIPAL INVESTIGATOR

PRINCIPAL

9-1-2020
STATUTORY AUDITOR

(Seal)
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

VBIT
Aushapur (V),
Ghatkesar (M)
VIGNANA BHARATHI INSTITUTE OF TECHNOLOGY
MEDCHAL DIST-501 301

(Seal)

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

UDIN: 20018474AAAAF7145

DETAILED STATEMENT OF EXPENDITURE FOR TRAVEL / FIELD WORK (incl. Special needs)

UGC Reference No. F: MRP-6945/16 (SERO/UGC)

Name of the Principal Investigator: Dr. Ch.S.L.N.Sridhar

Title of research project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"

..ɔ OF YEAR I

Name of the place visited	FROM	DATE	TO	DATE	Mode of Journey	Expenditure incurred(Rs)
VIJAYAWADA	HYDERABAD	26-2-18	VIJAYAWADA	27-2-2018	BUS	376+376=752
LB-NAGAR	HOME (BALAPUR)	26-2-18	LB-NAGAR		AUTO	150
KLU-..DDESWARAM	HOME (BANDER ROAD)	27-2-18	KLU		AUTO	200
..AYAWADA BUS STOP	KL UNIVERSITY	27-2-18	BUS STAND		AUTO	200
LBNGR	BALAPUR	28-2-18	LB NAGAR		AUTO	150
..ISAKHAPATNAM	HYDERABAD	30-6-2018	VIZAG	1/7/2018	BUS	1120+1120 = **2240**
LB-NAGAR	HOME (BALAPUR)	30-6-18	LB-NAGAR		AUTO	150
GVP-COLLEGE VIZAG	GVP	1/7/2018			AUTO	146
VIZAG BUS STOP		1/7/2018			AUTO	146
..BNGR-BALAPUR	LB NAGAR	2/7/2018	BALAPUR		AUTO	150
VIJAYAWADA	HYDERABAD	8/7/2018	VIJAYAWAADA	11/7/2018	BUS	376+376 = **752**
LB-NAGAR	HOME (BALAPUR)	8/7/2018	LB NAGAR		AUTO	150
KLU-V..DDESWARAM	HOME (BANDER ROAD)	9/7/2018	KLU		AUTO	200
..IJAYAWADA BUS STOP	KLU	11/7/2018	BUS STAND		AUTO	200
..BNGR-BALAPUR	LB NAGAR	12/7/2018	BALAPUR		AUTO	150
..VIJAYAWADA	HYDERABAD	5/8/2018	VIJAYAWADA	7/8/2018	BUS	568+496= **1064**
LB-NAGAR	LB NAGAR	5/8/2018	BALAPUR		AUTO	150
KLU-/..DDESWARAM	HOME(BANDER ROAD)	6/8/2018	KLU		AUTO	200
..IJAYAWADA BUS STOP	KLU	7/8/2018	BUS STAND		AUTO	200
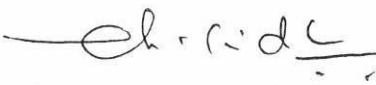
PNGR-BALAPUR	LB NAGAR	8/8/2018	BALAPUR		AUTO	150
				GRAND TOTAL		7500

END OF YEAR II

VISITED PLACE	MODE OF TRANSPORT	Ticket No.	Date	A
LB NAGAR	AUTO		5/5/2019	
VIJAYAWADA	BUS	61571143	5/5/2019	
KLU - VADDESWARAM	AUTO		6/5/2019	
VIJAYAWADA BUS STAND	AUTO		6/6/2019	
HYDERABAD	BUS	61873404	6/5/2019	
LB NAGAR - BALAPUR	AUTO		7/5/2019	
BALAPUR - LB NAGAR	AUTO		28/05/2019	
LB NAGAR - VIJAYAWADA	BUS	61913024	28/05/2019	
KLU - VADDESWARAM	AUTO		28/05/2019	
VIJAYAWADA BUS STAND	AUTO		28/05/2019	
VIJAYAWADA - HYDERABAD	BUS	61913033	28/05/2019	
LB NAGAR - BALAPUR	AUTO		29/05/2019	
BALAPUR - LB NAGAR	AUTO		9/6/2019	
LB NAGAR - VIJAYAWADA	BUS	62041669	9/6/2019	
KLU - VADDESWARAM	AUTO		10/6/2019	
VIJAYAWADA BUS STAND	AUTO		10/7/2019	
VIJAYAWADA - LB NAGAR	BUS	62101691	10/6/2019	
BALAPUR - LB NAGAR	AUTO		5/5/2019	

GRAND
TOTAL =   7,500

TOTAL(YEAR1+YEARII)=15000

ture of the Principal Investigator

Signature of the
Principal

PRINCIPAL
Vignana Bharathi Institute of Techno
Aushapur(V), Chatkesar(M), Medchal Dist-501
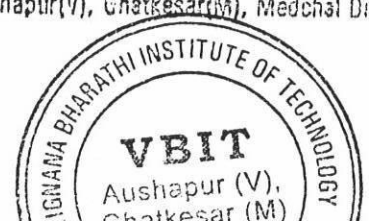
9-1-2020

Signature of the Statuory Auditor

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

HYDERABAD

VBIT
Aushapur (V),
Chatkesar (M)

DETAILED STATEMENT OF EXPENDITURE FOR TRAVEL / FIELD WORK (incl. Special needs)
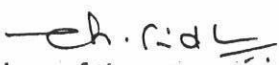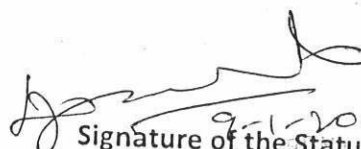
UGC Reference No. F: MRP-6945/16 (SERO/UGC)

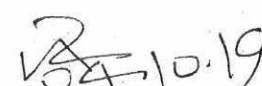Name of the Principal Investigator: Dr. Ch.S.L.N.Sridhar

Title of research project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"

| S.No	VISITED PLACE	MODE OF TRANSPORT	Ticket No.	Date	Amount
1	LB NAGAR	AUTO		5/5/2019	250
2	VIJAYAWADA	BUS	61571143	5/5/2019	674
3	KLU - VADDESWARAM	AUTO		6/5/2019	250
4	VIJAYAWADA BUS STAND	AUTO		6/6/2019	250
5	HYDERABAD	BUS	61873404	6/5/2019	605
6	LB NAGAR - BALAPUR	AUTO		7/5/2019	250
7	BALAPUR - LB NAGAR	AUTO		28/05/2019	250
8	LB NAGAR - VIJAYAWADA	BUS	61913024	28/05/2019	853
9	KLU - VADDESWARAM	AUTO		28/05/2019	250
10	VIJAYAWADA BUS STAND	AUTO		28/05/2019	250
11	VIJAYAWADA - HYDERABAD	BUS	61913033	28/05/2019	853
12	LB NAGAR - BALAPUR	AUTO		29/05/2019	250
13	BALAPUR - LB NAGAR	AUTO		9/6/2019	250
14	LB NAGAR - VIJAYAWADA	BUS	62041669	9/6/2019	727
15	KLU - VADDESWARAM	AUTO		10/6/2019	250
16	VIJAYAWADA BUS STAND	AUTO		10/7/2019	250
17	VIJAYAWADA - LB NAGAR	BUS	62101691	10/6/2019	853
18	BALAPUR - LB NAGAR	AUTO		5/5/2019	185

GRAND TOTAL : 7,500

Signature of the Principal Investigator

Signature of the Principal
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

Signature of the Statuory Auditor

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474
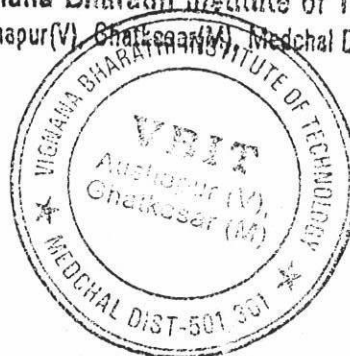
DETAILED STATEMENT OF EXPENDITURE FOR CHEMICALS (incl. Special needs)

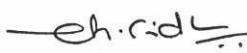**UGC Reference No. F:** MRP-6945/16 (SERO/UGC)
Name of the Principal Investigator: Dr. Ch.S.L.N.Sridhar
nanocrystalline Manganese-Zinc Ferrites"

S.No	Item	Qty.	Bill.No.	Date	Amount
1	Manganese(II)Nitrate 25g SA	1No's	28342990	1/25/2018	1,850
2	Zinc Nitrate 500g Finar	1No's	28342990	1/25/2018	324
3	Ferric Nitrate 500g Finar	1No's	28342990	1/25/2018	209
4	Titanium Dioxide 500 g Finar	1No's	28230010	1/25/2018	380
5	Potassium Hydroxide pellets 500g Finar	1No's	28152000	1/25/2018	348
6	Xylene 500 ml Finar	1No's	29024400	1/25/2018	306
7	Acetone 500 ml Finar	2 No's	29141100	1/25/2018	586
8	Ammonia solution 500 ml Finar	1No's	28142000	1/25/2018	161
9	Beaker 250ml Borosilicate	2 No's	70179090	1/25/2018	144
10	Beaker 500ml Borosilicate	4No's	70179090	1/25/2018	464
11	Beaker 1lit Borosil	4No's	70179090	1/25/2018	960
12	Specific gravity bottle 25ml Borosilicate glas	4No's	70179090	1/25/2018	920
13	Spatula rod Type 6"	2 No's	90189025	1/25/2018	44
14	Spatula rod Type 8" S.S	4No's	90189025	1/25/2018	104
15	Litmus Paper Red 10bkt Finar	1No's	38220000	1/25/2018	242
16	Litmus Paper Blue10bkt Finar	1No's	38220000	1/25/2018	242
17	Alumina crucible 25ml	2 No's	6909	1/25/2018	2200
18	Deionized water 5Lit	3 No's	2853	1/25/2018	360
19	Magnetic bead 6X25 mm	3No's	85051190	1/25/2018	450
20	Cotton Rolls	1No's	56012110	1/25/2018	120
21	Tissue Roll 200g	6No's	48030090	1/25/2018	180
22	White Nose Mask	4No's	70179090	1/25/2018	220
23	Magnetic bead 6X30 mm	1No's	85051190	1/25/2018	160
24	Sample Vials	2 Gros	70179090	1/25/2018	280
25	Nitrile Gloves	3 Pair	40159030	1/25/2018	45
26	Bottle Brush 12"	2 No's	9603	1/25/2018	40
27	Manganese(II)Nitrate Hydrate 98% 500g SA	1No's	28342990	1/25/2018	5314
	CGST				1465
	SGST				1465

28	Acetone AR 500 ml sd finer	3	GST-37784	3/5/2019	759.5
29	NICKEL NITRATE AR V500 GR	1	GST-37784	3/5/2019	1433.5
30	DISTILLED WATER 5LT PACK	2	GST-37784	3/5/2019	166.5
31	INDICATOR PAPERS	1	GST-37784	3/5/2019	186.5
32	DE IONISED WATER 5LT	5	536	26-07-2019	750
33	FERRIC NITRATE AR 500GR	1	536		900
34	TUSSUE ROLE 200GR	1	536		35
35	Acetone AR 500 ml sd finer	2	536		660
36	Al foils 9m	1	536		88
	CGST				219
	SGST				219

GRAND TOTAL = 25000.00

Signature of the Principal Investigator

Signature of the Principal

PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

Signature of the Statuory Auditor
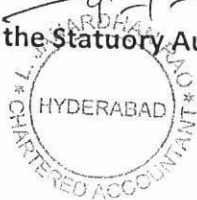
L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

DETAILED STATEMENT OF EXPENDITURE FOR CHEMICALS

UGC Reference No. F: MRP-6945/16 SERO/UGC)

BILL NO. 111

Name of the Principal Investigator: Dr.Ch.S.L.N.Sridhar

Date : 16- 201

Title of the Project : "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites "

END OF YEAR I

S.NO	ITEM	HSN/SAC	GST	Quantity	Rate	Disc %	Amount
1	NITRILE HW GLOVES	40159030	12	6 PAIRS	180	0	1,080.00
2	COLUMN BRUSH 24"	9603	18	2	29	0	58
3	BOTTLE BRUSH 12"	9603	18	2	20	0	40
4	BEAKER 1LT	70179090	18	2	240	0	480
5	BEAKER 500ML	70179090	18	2	116	0	232
6	BEAKER 250 ML	70179090	18	2	72	0	144
7	ALUMINA CRUCIBLE 50 ML	6909	18	4	1350	0	5400
8	MAGNETIC BEAD 8X40 MM	85051190	18	2	255	0	510
9	ACETONE 2.5LT PHYNOL	29141100	18	3	835	0	2505
10	NOSE MASK V-44	6307	5	50	13.6	0	680
11	PH INDICATOR PAPER 1-14 200 LS	38220000	12	1	188	0	188
12	MAGNESIUM NITRATE 500G FINER	28342920	18	1	180	0	180
13	CUPRIC NITRATE 500G FINER	28342920	18	1	654	0	654
14	COBALT NITRATE HEXA HIDRATE 100G FINER	28342920	18	1	545	0	545
15	HYDROCHLORIC ACID 500ML FINER	28061000	18	1	185	0	185
16	SULPHURIC ACID 500 ML FINER	28070010	18	1	232	0	232
17	DEIONISED WATER 5 LT	2853	18	2	120	0	240
18	ACETONE HPCL 1 LT FINER	29141100	18	1	487	0	487
19	ZINC NITRATE AR	28342990	18	1	346	0	346

	500G FINER						
20	FERRIC NITRATE 500G FINER	28342990	18	1	193	0	193
21	MANGENESE (II) NITRATE HYDRATE 98% 500G SA	28342990	18	1	5314	0	5314
22	NICKEL NITRATE 500G FINER	28342990	18	1	1436	0	1436
23	TISSUE ROLL 200G	48030090	18	2	35	0	70
24	SAMPLE VIALS	70179090	18	1 GROS	140	0	140
						Total	21339
						CGST	1838.27
	-					SGST	1838.27
						GRAND TOTAL	25015.5

END OF
YEAR II

S.No	Item	Qty.	Bill.No.	Date	Amount
1	Manganese(II)Nitrate 25g SA	1No's	28342990	1/25/2018	1,850
2	Zinc Nitrate 500g Finar	1No's	28342990	1/25/2018	324
3	Ferric Nitrate 500g Finar	1No's	28342990	1/25/2018	209
4	Titanium Dioxide 500 g Finar	1No's	28230010	1/25/2018	380
5	Potassium Hydroxide pellets 500g Finar	1No's	28152000	1/25/2018	348
6	Xylene 500 ml Finar	1No's	29024400	1/25/2018	306
7	Acetone 500 ml Finar	2 No's	29141100	1/25/2018	586
8	Ammonia solution 500 ml Finar	1No's	28142000	1/25/2018	161
9	Beaker 250ml Borosilicate	2 No's	70179090	1/25/2018	144
10	Beaker 500ml Borosilicate	4No's	70179090	1/25/2018	464
11	Beaker 1lit Borosil	4No's	70179090	1/25/2018	960
12	Specific gravity bottle 25ml Borosilicate glass	4No's	70179090	1/25/2018	920
13	Spatula rod Type 6"	2 No's	90189025	1/25/2018	44
14	Spatula rod Type 8" S.S	4No's	90189025	1/25/2018	104
15	Litmus Paper Red 10bkt Finar	1No's	38220000	1/25/2018	242
16	Litmus Paper Blue10bkt Finar	1No's	38220000	1/25/2018	242
17	Alumina crucible 25ml	2 No's	6909	1/25/2018	2200
18	Deionized water 5Lit	3 No's	2853	1/25/2018	360
19	Magnetic bead 6X25 mm	3No's	85051190	1/25/2018	450
20	Cotton Rolls	1No's	56012110	1/25/2018	120
21	Tissue Roll 200g	6No's	48030090	1/25/2018	180
22	White Nose Mask	4No's	70179090	1/25/2018	220
23	Magnetic bead 6X30 mm	1No's	85051190	1/25/2018	160
24	Sample Vials	2 Gros	70179090	1/25/2018	280
25	Nitrile Gloves	3 Pair	40159030	1/25/2018	45
26	Bottle Brush 12"	2 No's	9603	1/25/2018	40
27	Manganese(II)Nitrate Hydrate 98%	1No's	28342990	1/25/2018	5314

	500g SA				
	CGST				
	SGST				1465
					1465
28	Acetone AR 500 ml sd finer	3	GST-37784	3/5/2019	759.5
29	NICKEL NITRATE AR V500 GR	1	GST-37784	3/5/2019	1433.5
30	DISTILLED WATER 5LT PACK	2	GST-37784	3/5/2019	166.5
31	INDICATOR PAPERS	1	GST-37784	3/5/2019	186.5
32	DE IONISED WATER 5LT	5	536	26-07-2019	750
33	FERRIC NITRATE AR 500GR	1	536		900
34	TUSSUE ROLE 200GR	1	536		35
35	Acetone AR 500 ml sd finer	2	536		660
36	AI foils 9m	1	536		88
	CGST				219
	SGST				219

GRAND TOTAL = **25000**

TOTAL(YEAR 1+YEAR II)=50000

Signature of the Principal Investigator

Signature of Principal
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist.

Signature of the Statuory Auditor

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

DETAILED STATEMENT OF EXPENDITURE FOR CONTINGENCY (incl. Special needs)

UGC Reference No. F: MRP-6945/16 (SERO/UGC)

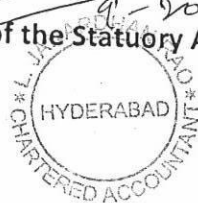Name of the Principal Investigator: Dr. Ch.S.L.N.Sridhar

Title of research project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"

S.No	Item	Qty.	Bill.No.	Date	Amount
1	SERVICE - CALIBRATION "INFRA" HP ANALYTICAL WEIGH SCALE - WEIGHMAN INDUSTRIES		1920/005	13-04-2019	2242.00
2	ALUMINIA CRUCIBLES W/O LiD 50 ML	2	535	26-07-2019	2758.00

GRAND TOTAL = 5000.00

Signature of the Principal Investigator

Signature of the Statuory Auditor

Signature of the Principal

PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

DETAILED STATEMENT OF EXPENDITURE FOR CONTINGENCY (incl. Special needs)

UGC Reference No. F: MRP-6945/16 (SERO/UGC)

Name of the Principal Investigator: Dr. Ch.S.L.N.Sridhar
Title of research project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"
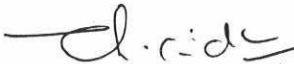
END OF YEAR I

S.No	Item	Qty.	Bill.No.	Date	Amount
1	BOROCIL GLASS BOTTLES	50	774	22-2-18	1062
2	Clamp Meter	1	23	2/7/2018	4056.84
	GRAND TOTAL				5000

END OF YEAR II

S.No	Item	Qty.	Bill.No.	Date	Amo
1	SERVICE - CALIBRATION "INFRA" HP ANALYTICAL WEIGH SCALE - WEIGHMAN INDUSTRIES		1920/005	13-04-2019	2242
2	ALUMINIA CRUCIBLES W/O LiD 50 ML	2	535	26-07-2019	2758

GRAND TOTAL =   5000.

TOTAL(YEAR 1+YEAR II)=10000

Signature of the Principal Investigator

Signature of the Principal
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

Signature of the Statutory Auditor

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

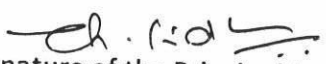# DETAILED STATEMENT OF EXPENDITURE FOR HIRING SERVICES (incl. Special needs)

UGC Reference No. F: MRP-6945/16 (SERO/UGC)

Name of the Principal Investigator: Dr. Ch.S.L.N.Sridhar

Title of research project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"
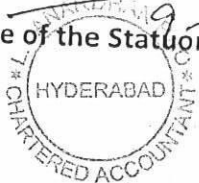
S.No	Item	Qty.	Bill.No.	Date	Amount
1	METAL POWDERS (XRD Screening) @ CMET	10	CMET/119/2019-20	17-07-2019	8,850
2	VSM ANALYSIS @ IICT	6	TSP/19-20/143	30-07-2019	15,000
3	DENSITY RECORDING @ OU	5	PD/2019	7/6/2019	1,150

GRAND TOTAL = 25,000.00

Signature of the Principal Investigator

Signature of the Principal

Vignana Bharathi Institute of Technology
Aushapur, Ghatkesar, Medchal Dist-501 301

Signature of the Statuory Auditor

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

DETAILED STATEMENT OF EXPENDITURE FOR HIRING SERVICES (incl. Special needs)

UGC Reference No. F: MRP-6945/16 (SERO/UGC) BILL NO: 1800053, 1800985

Name of the Principal Investigator:
Dr.Ch.S.L.N.Sridhar

Date : 23-05-18, 06-09-19

Title of the Project : "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites "
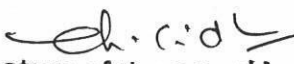
END OF YEAR I

S.No	Type of Test	Amount
1	PXRD Analysis	18,000.00
2	XED and FE-SEM	7,000.00
GRAND TOTAL		25000

END OF YEAR II

S.No	Item	Qty.	Bill.No.	Date	Amou
1	METAL POWDERS (XRD Screening) @ CMET	10	CMET/119/2019-20	17-07-2019	8,85(
2	VSM ANALYSIS @ IICT	6	TSP/19-20/143	30-07-2019	15,00
3	DENSITY RECORDING @ OU	5	PD/2019	7/6/2019	1,15(

GRAND TOTAL = 25,000.00

TOTAL (YEAR 1+YEAR II)= 50,000

Signature of the Principal Investigator

Signature of the Principal
PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

Signature of the Statuory Auditor

CA. L. JANARDHAN RAO
Chartered Accountant
M.No: 18474

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002.

Final Report of the work done on the Minor Research Project. (Report to be submitted within 6 weeks after completion of each year)

1. Project report No.Final: <u>Second (After twoyears)</u>dated: 25/09/2019

2. UGC Reference No. F:<u>MRP-6945/16 (SERO/UGC)</u>

3. Period of report: from: 15/08/2017 to07/08/2019

4. Title of research project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"

5. (a) Name of the Principal Investigator: <u>Dr. Ch.S.L.N.Sridhar</u>

   (b) Dept: <u>Physics</u>

   (c) College where work has progressed: <u>Vignana Bharathi Institute of Technology</u>

6. Effective date of starting of the project: 15/08/2017

7. Grant approved and expenditure incurred during the period of the report:

   a. Total amount approved Rs.<u>1,75,000/</u>

   b. Total expenditure Rs.<u>1,62,500/</u>
   c. Report of the work done: (Copy attached)

i) To synthesize Titanium doped Nanocrystalline Manganese Zinc ferrites using hydrothermal Method .

ii) To study the Structural Properties using XRD,SEM.

iii) To study the Dielectric Properties like Dielectric constant, Dielectric loss,Conductivity Using Impedance Analyzer.

iv) To find the Magnetic Properties like Saturation magnetization, Coercivity and Retentivity using VSM

ii. Work done so far and results achieved and publications, if any, resulting from the work (Give details of the papers and names of the journals in which it has been published or accepted for publication :-

- Hydrothermal Mehod is successfully used to synthesize Titanium doped nano crystalline Manganese Zinc Ferrites

- The crystallite Size is found to be in the range 42nm to 58nm
- The lattice parameter value is found to vary from $8.210 A^\circ$ to $8.263 A^\circ$
- The dielectric constant is found to be Maximum for $x=0.1$ and it is found to depend upon lattice parameter and cation inversion
- The loss factor is found to be between 0.18 to 0.23. The small dielectric loss makes these materials useful at higher frequencies.
- The impedance spectroscopy studies reveal the contribution of grain and grain boundary on the electrical properties of the materials.
- Single semicircle in the Nyquist plots corresponds to the predominance of the grain boundary
- The real dielectric modulus increases with frequency and at higher frequencies it reaches a maximum
- With increase in dopant concentration the position of the peak in imaginary electric modulus vs frequency shifts to lower frequencies providing indication of conductivity relaxation
- Reduced values of M_s with doping x due to spin canting mechanism and triangular spin arrangementin nanosized grains and lowered density of the samples.
- Attainment of zero value of H_c upto a critical size of ~49nm denotes the superparamagnetic nature of nanophased Mn-Zn-Ti ferrites.
- Lowered values of ε', tanδ and higher values of ρ render these materials usable for high frequency applications.

Paper titled Dielectric Properties of Superparamagnetic Titanium doped Nanophased Mn-Zn ferrites for High Frequency Applicationshas been Accepted for publication by " **Materials Research Express**" (**SCI JOURNAL**)

iii. Has the progress been according to original plan of work and towards achieving the objective. if not, state reasons

Yes: the progress is according to the original plan of work.

iv) Please enclose a summary of the findings of the study. One bound copy of the final report of the work done may also be sent to the concerned Regional Office of the UGC.

The copy of the summary of the findings of the study. The copy of the final Report will also be sent to the Regional office of the UGC.

v) Any other information: Nil.

SIGNATURE OF THE PRINCIPAL INVESTIGATOR

PRINCIPAL
PRINCIPAL
Vignana Bharathi Institute of Technol
Aushapur(V), Ghatkesar(M), Medchal Dist-501

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG NEW
DELHI – 110 002

**PROFORMA FOR SUBMISSION OF INFORMATION AT THE TIME OF SENDING THE FINAL
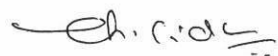REPORT OF THE WORK DONE ON THE PROJECT**

1. Title of the Project: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"

2. NAME AND ADDRESS OF THE PRINCIPAL INVESTIGATOR: Dr. Ch.S.L.N.Sridhar, VBIT, Hyderabad.

3. NAME AND ADDRESS OF THE INSTITUTION: Vignana Bharathi Institute of Technology, Aushapur, Hyderabad

4. UGC APPROVAL LETTER NO. AND DATE: MRP-6945/16 (SERO/UGC), 28/7/2017

5. DATE OF IMPLEMENTATION: 15/08/2017

6. TENURE OF THE PROJECT: Two years

7. TOTAL GRANT ALLOCATED:1,75,000/-

8. TOTAL GRANT RECEIVED: 1,62,500/-

9. FINAL EXPENDITURE: 1,75,000/-

10. TITLE OF THE PROJECT: "Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites"

11. OBJECTIVES OF THE PROJECT:

12. i) To synthesize Titanium doped Nanocrystalline Manganese Zinc ferrites using hydrothermal Method .

13. ii) To study the Structural Properties using XRD,SEM.

14. iii) To study the Dielectric Properties like Dielectric constant, Dielectric loss,Conductivity Using Impedance Analyzer.

15. iv) To find the Magnetic Properties like Saturation magnetization, Coercivity and Retentivity using VSM

16. WHETHER OBJECTIVES WERE ACHIEVED: YES

[55]   B.Issa, I.M.Obaida, B.A.Albiss and Y.Haik, Magnetic Nanoparticles, Surface Effects and Properties Related to Biomedicine Applications, **14**(11) (2013)21266 – 21305.<u>Int J Mol Sci</u>. 2013 Nov; 14(11): 21266–21305.

[56]   M.Rozman and M.Drofenik, Hydrothermal Synthesis of Manganese Zinc Ferrites, Journal of American Ceramic Society, **78** (1995) 2449 – 2455.

WHETHER ANY PH.D. ENROLLED/PRODUCED OUT OF THEPROJECT: NO

NO. OF PUBLICATIONS OUT OF THE PROJECT: One (SCI JOURNAL)

No. OF. PRESENTATIONS: 1(At Osmania University-National conference)
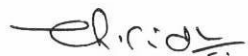
(PRINCIPALINVESTIGATOR)

(PRINCIPAL)

PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

ACCESSION CERTIFICATE

This is certified that Dr. Ch.S.L.N.Sridhar, Department of Physics, VBIT, Hyderabad has handed over the following books and journals purchased under the scheme of Minor Research Project to the Library of Vignana Bharathi Institute of Engineering & Technology, Hyderabad. The following are books and journals handed over by Dr. Ch.S.L.N.Sridhar (MRP-6945/16 (UGC/SERO).

S. No	Item	Qty.
1	Nano materials and Nano composites	1
2	ADV .Materials & Design for Electromagnetic interference	1
3	Engineering Applications of Nanotechnology	1
4	Nano Characterization	1
5	Nano technology for water treatment and purification	1
6	Defect structures and Properties	1
7	Hand Book of modern ferromagnetic materials-part-1	1
8	Hand Book of modern ferromagnetic materials-part-2	1
9	Modern Ferrite technology	1
10	Ferrite Nanoparticles	1

Signature of the
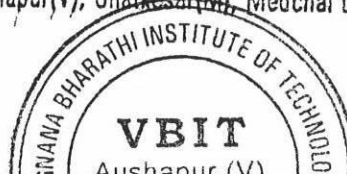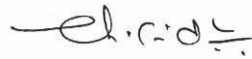Principal Investigator

Signature of the
Librarian

Signature of the
Principal

SRINIVASA RAO GANTA
Asst. Professor in LIS
& Librarian VBIT

PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301
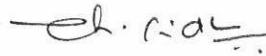
VBIT
Aushapur (V)

ASSETS CERTIFICATE

This is certified that Dr. Ch.S.L.N.Sridhar, Department of Physics, VBIT, Hyderabad has handed over the following equipment purchased under the scheme of Minor Research Project to the Department of Physics, Vignana Bharathi Institute of Engineering & Technology, Hyderabad. The following are equipments handed over by Dr. Ch.S.L.N.Sridhar (MRP-6945/16 (UGC/SERO).

S. No	Particulars	Company	Qty.
1	Hydraulic Press 15 Ton	MJL Laboratory	1
2	kbr die Set	MJL Laboratory	1

Signature of the
Principal Investigator

Signature of the
Head of the Dept.
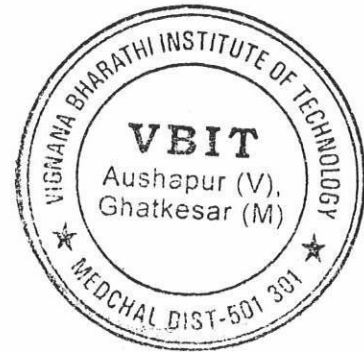
Signature of the
Principal

PRINCIPAL
Vignana Bharathi Institute of Technology
Aushapur(V), Ghatkesar(M), Medchal Dist-501 301

VBIT
Aushapur (V),
Ghatkesar (M)
VIGNANA BHARATHI INSTITUTE OF TECHNOLOGY
MEDCHAL DIST-501 301

THESIS

Title of Minor Research Project: Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites.

Principal Investigator: Dr. Ch.S.L.N.Sridhar, Professor. of Physics, Dept. Of Physics, Vignana Bharathi Institute of Technology, Aushapur, Hyderabad.

UGC Reference No. F: MRP-6945/16 (SERO/UGC)

Report of the work done
Synthesis-Structural-Dielectric and Magnetic studies on Titanium doped nanocrystalline Manganese-Zinc Ferrites

Introduction

Mn–Zn ferrites are an important class of soft ceramic magnetic materials, with relatively low cost, lower core losses, high electrical resistivity and high initial permeability and have a wide range of applications [1] in electronic or electrical peripherals. Rapid development in power electronic devices towards miniaturization tends to increase the operating frequency of the Mn-Zn ferrites to relatively higher values resulting in dramatic increase in power losses. One of the key strategies to improve the electromagnetic properties is to synthesize [2] the nanoferrites from their bulk counterparts. Occurrence of new physical phenomena like quantum confinement and larger surface to volume ratio in the nano regime along with improved power losses and enhanced electrical resistivity are reported [3, 4] in nanophased Mn-Zn ferrites. Owing to these phenomena, they have enormous technological and biomedical applications in ferrofluids, magnetocaloric refrigeration, Magnetic Resonance Imaging (MRI)and guided drug delivery. Although nanoparticles of pure metals like Fe, Co and Ni are found [5,6] to exhibit superparamagnetism, they have limited applications due to their chemical unstability and relatively lower sizes of few nanometers. On the other hand, magnetic metal oxides such as spinel ferrites have a great potential for applications as they are relatively inert and their properties can be improved [7] by addition of dopants. Single domain particles are formed for critical size varying from 10 – 40 nm resulting in an increase of coercivity with increase in crystallite size. In this single domain region, reduced coercivity to nearly zero is referred to as superparamagnetism (SPM). Beyond the critical size, coercivity decreases with increase in crystallite size reflecting upon the multi-domain nature of the ferrites. Occurrence of SPM is reported [8-12] in $MnFe_2O_4$, $MnZnFe_2O_4$, Lanthanum and Gadalonium doped Mn-Zn ferrites by other researchers.

Among a wide variety of techniques that exist for the synthesis of ferrites, Hydrothermal synthesis is proven [13] to be relatively user friendly and cost-effective technique which needs comparatively lower sintering temperatures, less energy and has an advantage of producing less agglomerated particles of controlled size. It is well recognized [14] in the field of electroceramics that the nature and the amount of dopant is one of the most important operational parameters towards tailoring or improving properties and performance of the product.

TiO$_2$ is an effective additive in improving [1] electromagnetic properties by replacing Fe^{3+} ions with Ti^{4+} ions at octahedral sites of the spinel lattice.Increased resistivity and decreased losses are reported [15,16] by substitution of 2Fe^{3+} ions by Fe^{2+} + Ti^{4+} pairs by localization of Ti^{4+} ion.It is reported [17] that the conductivity in Titanium doped Mn-Zn ferrites at lower Ti^{4+} concentrations is due to formation of Fe^{2+}–Ti^{4+} pairs, while at higher Ti concentrations, it is ascribed to increasing scattering of the electrons.Simultaneous codoping of TiO$_2$, CaCO$_3$ and SiO in the Mn-Zn ferrite lattice is found [18] to lead to the formation of Fe^{2+}–Ti^{4+} pairs replacing Fe^{3+} ions in the octahedral B-sites hindering the electron hopping, thus resulting in an increase of dc electrical resistivity and improved power losses of the material. An overall increase in ac resistivity and decrease in saturation magnetisation is observed [19] with increase in Ti^{4+} concentration in Mn$_{1-x}$Fe$_{2-2x}$Ti$_x$O$_4$ ferrites. Addition of Ti^{4+} ions in Ni-Zn ferrites is reported [20] to exhibit improved values of electrical resistivity while it is found to exhibit an adverse effect on saturation magnetization.Higher values of electrical resistivity are obtained [21] in Cobalt and Titanium doped Mn-Zn ferrites.

Incorporation of tetravalent ions like TiO$_2$ or SnO$_2$ into ferrites is reported [22] to be capable of development of high resistivity ferrites as they form stable bonds with Fe^{2+} ions and they impede the process of electron hopping. Moreover, it is also reported [1] that addition of pentavalent ions is useful in producing high conductivity ferrites whereas tetravalent ions have the ability to produce ferrites of comparatively higher values of electrical resistivity.In our previous work, for

both the cases of Sb^{5+} and Nb^{5+} doped nanocrystalline Ni-Zn ferrites synthesized [23,25] by hydrothermal method, we have obtained XRD patterns with pure spinel phase, exhibiting relatively larger values of saturation magnetisation (M_s), higher values of ac resistivity (ρ) and lower loss factor ($\tan\delta$) values which are viable for high frequency applications. When similar dopants, i.e., Sb^{5+} and Nb^{5+} are introduced [24, 26] into nanophased Mn-Zn ferrites, XRD patterns show certain hematite peaks along with spinel phase accompanied by higher values of ρ and lower values of $\tan\delta$ and M_s. Titanium ions dissolve [27] in the lattice and occupy regular tetrahedral and octahedral positions of the lattice and change the dielectric and magnetic properties. Electrical resistivity is found to be improved by formation of a liquid phase at the grain boundaries but addition of Ti^{4+} has a negative effect on the values of M_s. In view of the above reports on Ti^{4+} doped ferrites and other pentavalent Sb^{5+} and Nb^{5+} doped ferrites, a humble attempt is made to synthesize nanophased $Mn_{0.5}Zn_{0.5}Ti_xFe_{2-4x/3}O_4$ ferrites by hydrothermal method at lower dopant concentrations i.e., for x varying from 0 – 0.05 insteps of 0.01 and characterize them for possible applications.

2. Experimental

2.1 Preparation

Nanophased $Mn_{0.5}Zn_{0.5}Ti_xFe_{2-4x/3}$(x=0.0,0.01,0.02,0.03,0.04 and 0.05) are prepared using hydrothermal method. The starting precursors are A.R.Grade $Mn(NO_3)_2,6H_2O$, $Zn(NO_3)_2,6H_2O$, $Fe(NO_3)_3,9H_2O$ and TiO_2. Stoichometric quantities of the required materials are dissolved in deionised water and using Ammonia solution, P^H of 8 is obtained. The solution is then thoroughly mixed using magnetic stirrer for nearly one hour and is then heated in a autoclave at a temperature of 200^0C for four hours. After allowing to cool naturally, the solution is taken out and cleaned using deionised water. Hot air oven is used to dry the sample at 60°c for 24hrs. The material is then taken out and powdered using an agate mortar. The powder is then sintered at a temperature of 500^0C for 6hrs. Pellets of 12mm diameter are prepared at a pressure of 5Tons.

The pellets are then sintered at a temperature of 1000^0C for 6hrs. The sintered pellets are used for structural,electrical and Magnetic characterization.

2.2 Structural Studies

XRD studies were carried out with a Bruker (Germany, Model: D8) X-ray Diffractometer operated with CuK_α radiation with $\lambda = 1.5406$ Å. The lattice parameter (**a**) corresponding to the observed prominent reflections (identified with the help of standard JCPDS Card No.10-0467) is estimated using the equation

$$\mathbf{a} = d\sqrt{\left(h^2 + k^2 + l^2\right)} \qquad \underline{\hspace{1cm}} (1)$$

where, d is experimentally observed inter-planar spacing and
        $(h\ k\ l)$ are the Miller indices of the planes.

The theoretical density (ρ_x) for the samples was estimated by an equation

$$\rho_x = \frac{ZM}{N\mathbf{a}^3} \qquad \underline{\hspace{1cm}} (2)$$

where, Z refers to the number of molecules per unit cell in the spinel structure,
        M is the Molar mass of the sample,
a is the lattice parameter of the ferrite and
        N is the Avogadro's number.

Crystallite (**D**) size was determined by Scherrer formula givenby

$$\mathbf{D} = \frac{k\lambda}{\beta\cos\theta} \qquad \underline{\hspace{1cm}} (3)$$

where, k was taken as a constant (~1, i.e., unity in the wake of shape of the crystal),
        λ was the wavelength of X-rays and
        β was full width at half maximum.

2.3 Dielectric studies:

The dielectric response of all samples was measured using LCR meter Hioki 3532-50, LCR Hi TESTER, in the frequency range of 40Hz to 5 MHz over a temperature range of 200 ^0C.

The dielectric constant $(\varepsilon'(\square))$ at various frequencies was calculated using the measured capacitance value at the strong accumulation region from the following relation,

$$\varepsilon' = \frac{Cd}{\varepsilon_0 A}$$

_____ (4)

where, C is the capacitance (Farads),

d is thickness of the sample (2.87mm – 6.5 mm),

A is the cross-sectional area of the flat surface (11.30 mm^2–11.32 mm^2) and

ε_0 is the permittivity of free space (8.85x10^{-12} F/m).

$$\tan \delta = \frac{\varepsilon''(\boxed{})}{\varepsilon'(\boxed{})}$$

_____ (5)

where, $\varepsilon'(\boxed{})$ is the real part of permittivity and

$\varepsilon''(\boxed{})$ is the imaginary of permittivity

The a.c. conductivity of the samples at desired frequencies can be estimated using the equation

$\sigma_{a.c.} = \varepsilon' \varepsilon_0 \boxed{} \tan \delta$

_____ (6)

where, $\varepsilon'(\boxed{})$ is the real part of permittivity,

ε_0 is the permittivity of free space (8.85x10^{-12} F/m),

$\boxed{} = 2\pi f$ is the angular frequency and

$\tan \delta$ is the dielectric loss factor

2.4 Magnetic Studies

The magnetic studies were done using Microsense (EZ) Vibrating Sample Magnetometer.

In the spirit of Neel's two-sub lattice [28] model, local spins for the Ti doped Mn-Zn ferrites,

viz., local magnetic moments [29,30] were estimated in terms of Bohr's magneton n_B by

$$n_B = \left(\frac{M_w}{5585} \right)\left(\frac{M_s}{\rho} \right)$$

_____ (7)

where, M_w denotes the molar mass of the sample,

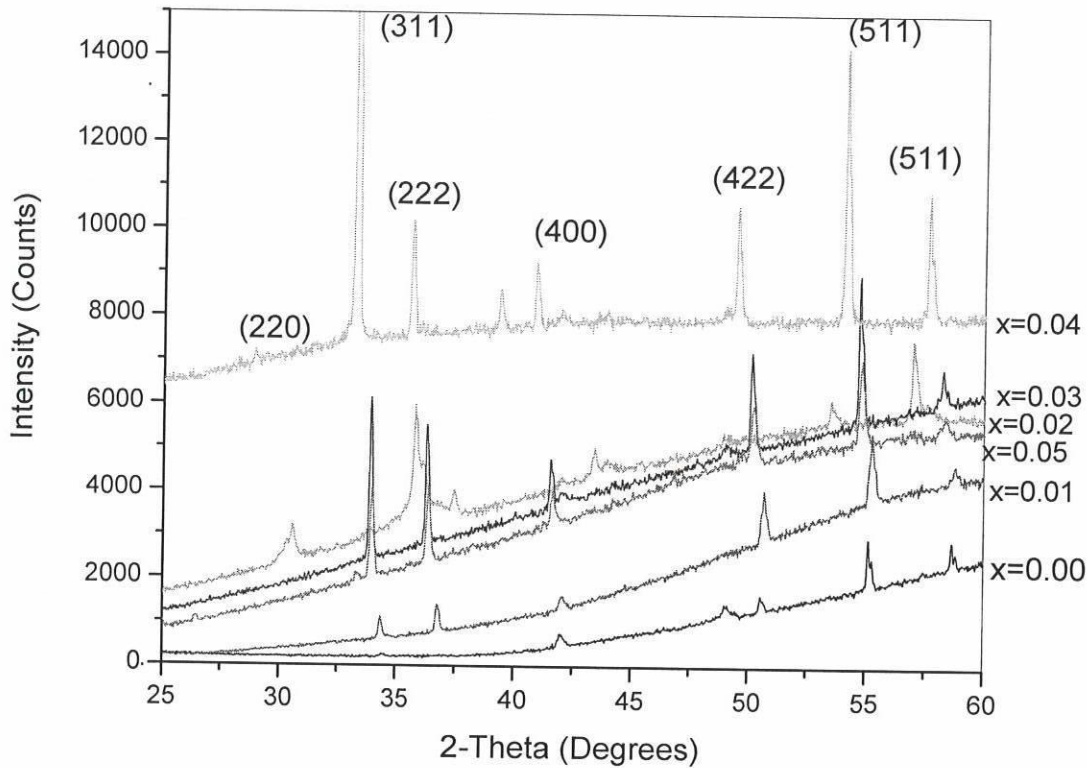M_s denotes the saturation magnetization and

ρ denotes the theoretical density

3.1 Structural Characterization

XRD Patterns of the $Mn_{0.5}Zn_{0.5}Ti_xFe_{2-4x/3}O_4$ ferrites for 'x' varying from 0 to 0.05 in steps of

0.01 sintered at 1000^{0}C are presented in Figure – 1. From Figure – 1, it is observed that the

present Ti^{4+} doped Mn-Zn ferrites are found to assume pure spinel structure without any

additional peaks by matching with standard JCPDS data. In our earlier reports [24,26] of Sb^{5+}

doped and Nb^{5+} doped Mn-Zn ferrites synthesized by hydrothermal method, the ferrites are sintered at 800^0C and an extra hematite phase is observed along with the spinel phase. But, in the present case of Ti^{4+} doped nanocrystalline Mn-Zn ferrites sintered at 1000^0C, XRD patterns exhibit pure spinel phase with no additional hematite peaks. The formation of pure spinel phase in the present case of Mn-Zn-Ti ferrites is attributed [31] to high temperature sintering conditions of 1000^0C. Relative broadening of XRD peaks evince the formation of nanocrystalline ferrites. The values of lattice parameter (**a**) determined from XRD data are provided in Table – 1. Variation of lattice parameter (a) with dopant concentration (x) is presented in Figure – 2. The lattice parameter (**a**) is found to vary from 8.303 A to 8.926 A. From Figure – 2, it is observed that at lower concentrations (i.e., upto x = 0.02), **a** is found to decrease with x. For intermediate concentrations, i.e., from x=0.02 to x=0.04, the values of **a** exhibit an increasing trend. Beyond x=0.04, again a decreasing trend of **a** with x is witnessed. Mn-Zn ferrite is a mixed spinel ferrite and Ti^{4+} ions are reported [32] to have a tendency to enter both A- and B-sites. The ionic radius of Ti^{4+} (0.56 A)ions is less than that of Fe^{3+} ions (0.63A) at A – sites. So, the initial decrease of **a** with x upto x = 0.02 is attributed to the replacement of Ti^{4+} ions with Fe^{3+} ions at A-sites. As the ionic radius of Ti^{4+} (0.74 A) ions at B-sites is larger than that of Fe^{3+}(0.69 A) ions, they tend to replace Fe^{3+} ions at B-sites resulting in increase of lattice parameter. Again, the decrease in **a** for x=0.05 is explained by the influence of repulsion parameter (b) and Madelung Constant (M). The lattice parameter **a** is proportional [33] to (b/M). At higher concentrations, more Ti^{4+} enter into B-sites, thus pushing more charge from B-sites to A-sites resulting in an increase of M. Thus, b/M decreases. Hence, lattice constant decreases for x = 0.05. It is also reported [34] that in case of ferrites that are neither completely normal spinel nor inverse spinel, the lattice parameter **a** is found to exhibit a non-linear behavior with dopant concentration (x). Thus, the non-linear behavior of lattice constant of nanocrystalline Mn-Zn ferrites with Titanium

concentration in the present case can also be attributed to the mixed spinel structure of Mn-Zn-Ti ferrites.

The crystallite size (**D**)is calculated using the (311) peak and the values are provided in Table – 1. The values of **D** are found to be in the range of 78 nm – 41 nm. An overall decreasing trend of **D** with x is presented in Figure – 3. In case of Sb^{5+} doped nanocrystalline Mn-Zn ferrites, the crystallite size is reported [24]to be in the range of 46nm – 14nm for Sb^{5+} doped ferrites, while incase of Nb^{5+} doped ferrites, **D** is found [26]to varyfrom 50nm – 14nm. Relatively larger values of **D** in the present case of Ti^{4+} doped Mn-Znferrites as compared to that of reported[24,26] values of other high valency dopedMn-Zn ferrites isattributed to higher sintering temperature of 1000^0C employed in the present case. The crystallite size is reported [35] to increase from 74nm – 137 nm in Ni-Ti dopedMn ferrites synthesized by standard ceramic method and sintered at 1300^0C.Decrease in **D** with Ti^{4+} doping in the present case of Mn-Zn

ferrites vouches for the successful substitution of dopant by the present hydrothermal method adopted in the synthesis of the ferrites.
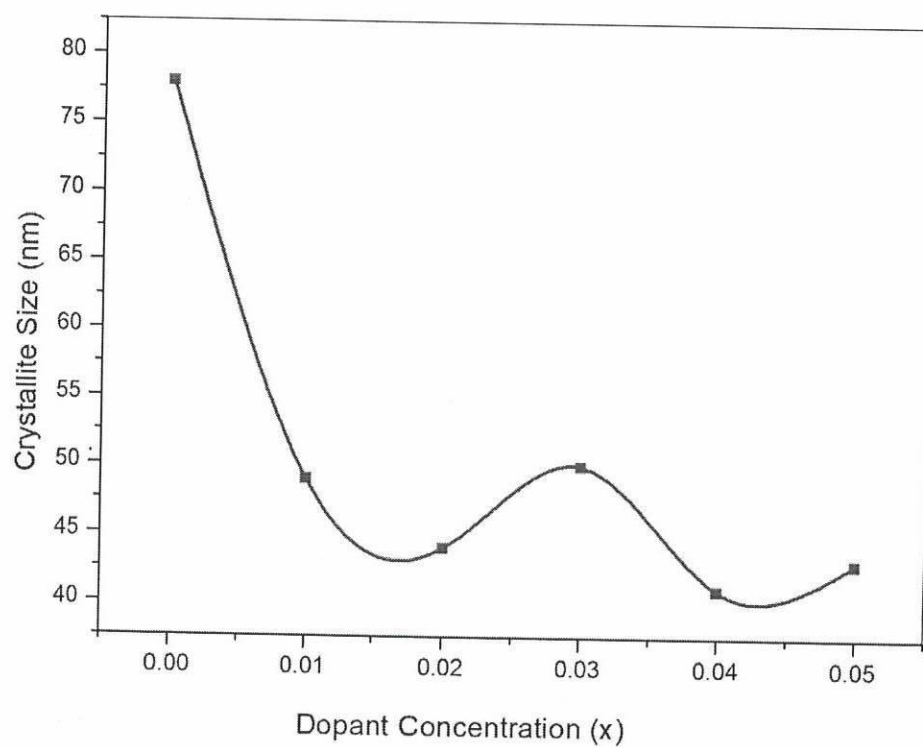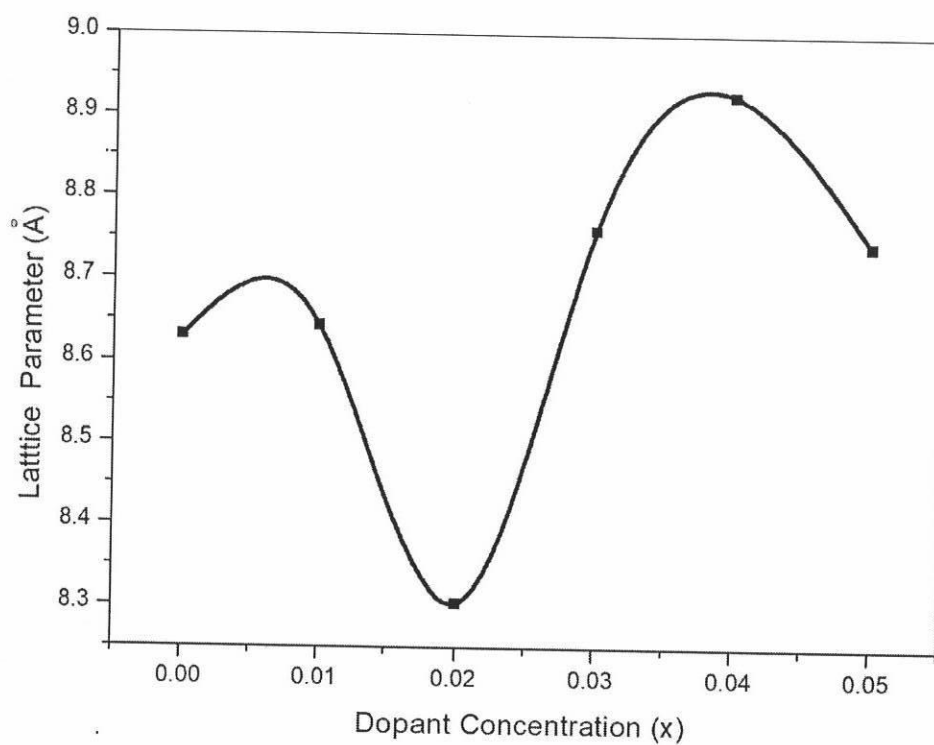
The representative FESEM Micrographs for x=0.00, x=0.01 and x=0.05are presented in Plates – 1(a-c). The images are clear and lesselectrostatically distorted. Well crystallized grains with almost uniform size can be observed. Decrease in particle size with increase in Ti^{4+} dopant concentration can be clearly observed from SEM micrographs. An overall view of structural properties indicate that the present hydrothermal method is successful in synthesizing single phase nanocrystalline Titanium doped Mn-Zn ferrites.
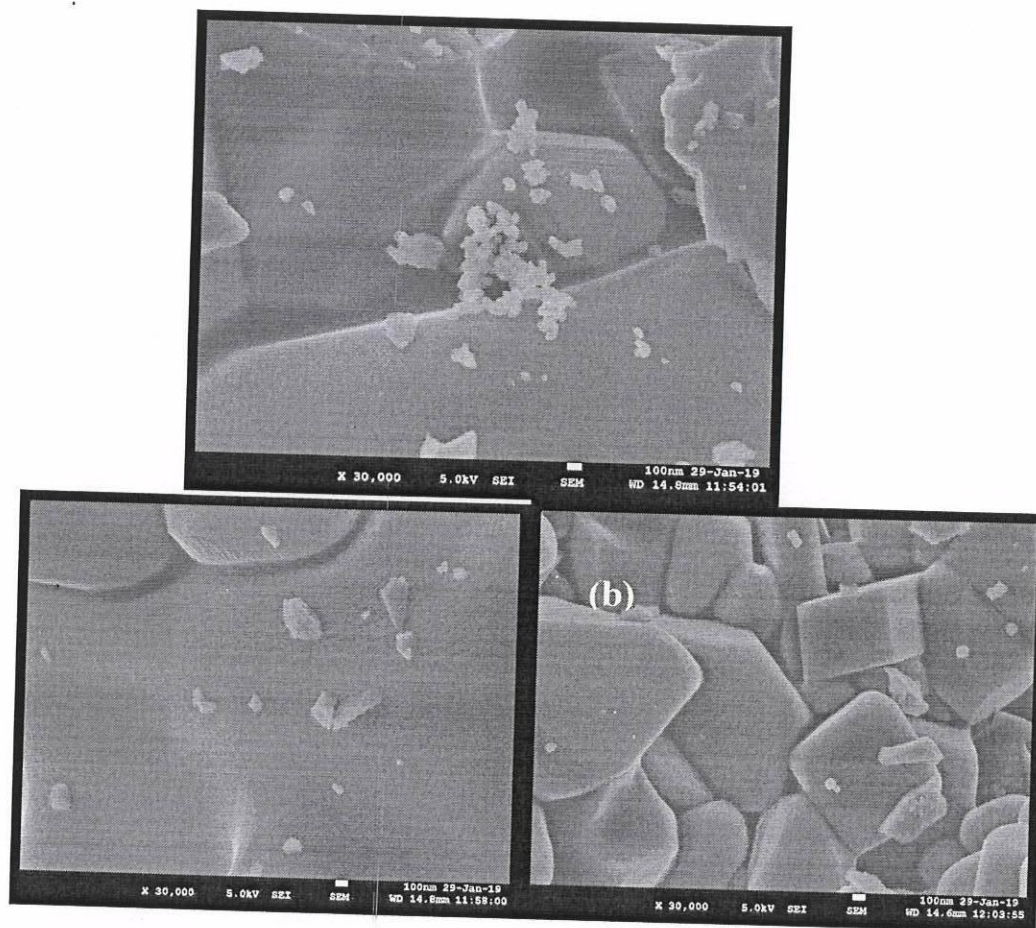
Composition	Lattice Parameter (Å)	Crystallite size (nm)	X-ray density (gm/cm^3)	Dielectric Studies at 1MHz			
				Permittivity ϵ'	Dielectric Loss ϵ''	Loss factor (tan δ)	ac Conductivity $(\sigma \times 10^{-7})\Omega^{-1}$ cm^{-1}
x = 0.00	8.6305	78	3.55	29	1.74	0.060	10.09
x = 0.01	8.6435	49	3.53	24	0.63	0.026	4.46
x = 0.02	8.303	44	3.98	27	0.98	0.036	6.32
x = 0.03	8.7593	50	3.39	25	0.17	0.006	1.15
x = 0.04	8.9255	41	3.20	21	0.34	0.016	2.17
x = 0.05	8.7422	43	3.40	18	0.15	0.008	1.36

3.2 Dielectric Studies

The variation of dielectric constant (ε') with varying ac frequency from 100mHz – 4 MHz is shown in Figure – 4. From Figure – 4, ε' is found to decrease with frequency. According to Maxwell-Wagner theory [36] both conductivity and dielectric constant depend on the hopping between Fe^{2+} and Fe^{3+} ions. The dielectric polarization in ferrites is a combined effect of grains and grain boundaries. At lower frequencies, the contribution due to grain boundaries will be more effective and at higher frequencies grains play a major role. In case of Manganese Zinc ferrites, the conduction process depends on electron hopping between Fe^{2+}/Fe^{3+} and hole hopping between Mn^{2+}/Mn^{3+} ions. At lower frequencies the hopping frequency follows the applied frequency. But, as the frequency increases, the electron exchange between $Fe^{2+} \leftrightarrow Fe^{3+}$ does not follow the AC field and hence the polarization decreases. Thus, the anticipateddecrease in dielectric constant with frequency is explained.

Variation of Dielectric constant (ε') with dopant concentration (x) is exhibited in the inset Figure – 4a. A non-linear variation of ε' with x is observed from Figure – 4a. However, an

overall decreasing trend of ε' with increase in x can be witnessed. As the concentration of Ti^{4+} ions increase in nanocrystalline Mn-Zn ferrites, they tend to replace Fe^{3+} ions at B-sites, consequently decreasing [34] the probability of hopping between Fe^{3+} and Fe^{2+} ions. So, the values of ε' of Ti^{4+} doped Mn-Zn ferrites are found to be lower than that of undoped sample. Relatively lower values of ε' ranging from 29 – 18 are obtained in the present case of Titanium doped nanocrystalline Mn-Zn ferrites.
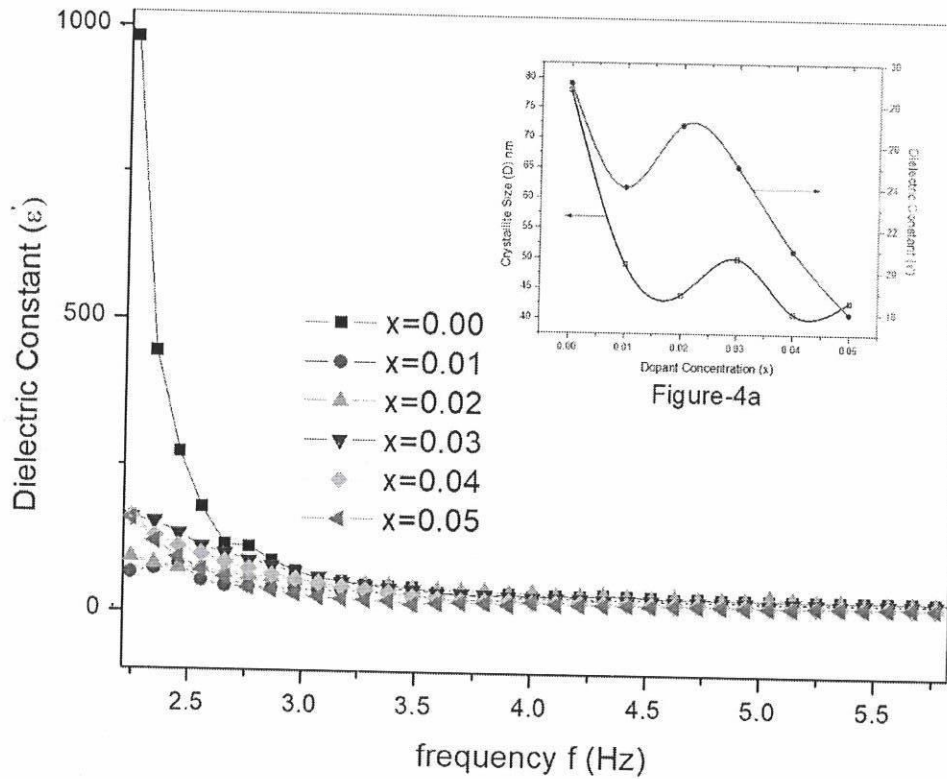
Figure-4a

Variation of ε' with **D** and x is shown in inset graph Figure – 4a. From Figure– 4a, a correlative trend between ε' and **D** with x is observed. The values of ε' are found to increase with increase in D. From the reports [37], it is observed that in the ferrite structure, the number of iron ions at octahedral sites increase [38] with increase in grain size. The more the number of iron ions formed at octahedral sites the more will be number of Fe^{2+} – Fe^{3+} ion pairs. So, polarization increases and results in an increase of ε'. Thus, the anomalies in the values of ε' with x are

attributed to the grain size. Similar grain size dependence of dielectric constant is also reported [24,26] in Sb[5+] and Nb[5+] doped Mn-Zn ferrites. Moreover, the variation of dielectric constant is found [22] to be inverse to that of lattice parameter, except for x = 0.02. The deviation observedat x=0.02is attributed to the relatively higher density of the sample.
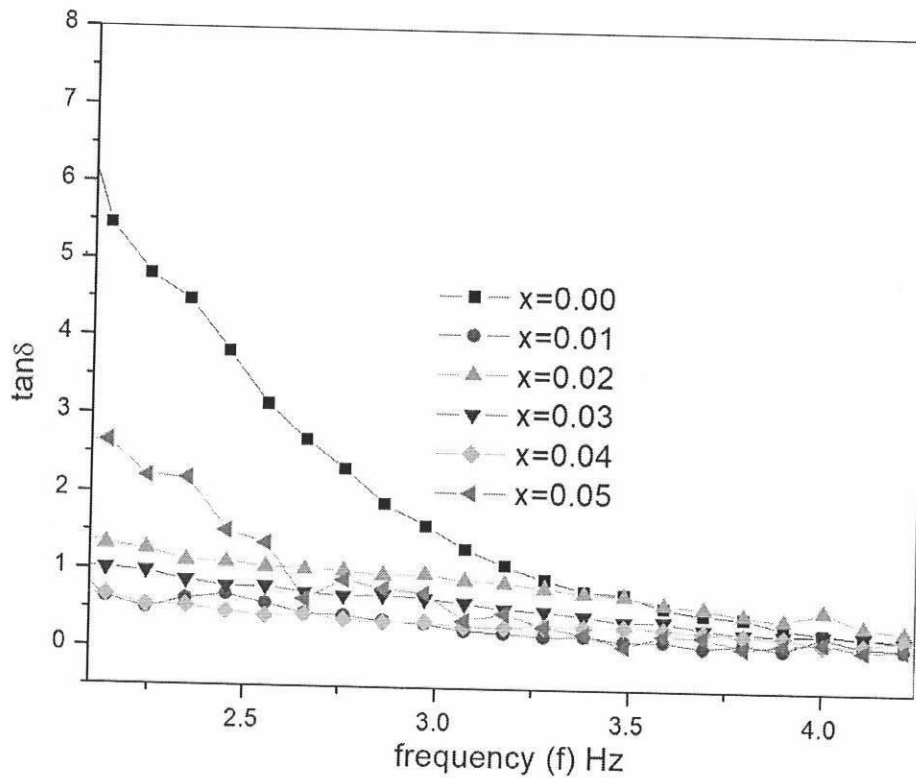
The values of ε' in bulk Mn-Zn ferrites synthesized by ceramic method are found [39] to be of the order of (17 -32) x10[5] at room temperature. In case of nanocrystalline Mn-Zn ferrites, the reported [40] values of ε' are reduced by an order of 10[4]. The values of ε' are found to be 85, 79, 395 for crystallite sizes of 65nm, 59nm and 11nm, respectively. In case of Ti[4+] doped nanocrystalline Ni-Zn ferrites, the values of ε' are reported [41] to be of the order of 10[3]. In our previous work of Sb[5+] and Nb[5+] doped nanocrystalline Mn-Zn ferrites synthesized [24,26] by hydrothermal method, the order of magnitude of ε' is 10[2]. At room temperature, Ti[4+] doped Manganese ferrites are found [42] to have values of ε' ranging from 10 to 100 at1 MHz

frequency. In the present case, the values of ε' are as low as of the order of 29 – 18. As reported [27] by other researchers earlier, in the present case also, Titanium is found to improve the values of ε' and make these materials viable for high frequency microwave applications.

The variation of dielectric loss factor (tanδ) as a function of frequency for varying Ti^{4+} concentrations in nanocrystalline Mn-Zn ferrites is provided in Figure – 5. The values of tanδ exhibited by Ti^{4+} doped Mn–Zn ferrites at 1MHz frequency are presented in Table – 1. At lower frequencies, the values of tanδ exhibit a steep decrease and as the frequency increases, a marginal decrease in tanδ is observed. Dielectric loss at lower frequencies will be more due to various factors like [43] predominance of Fe^{2+} ions, interfacial dislocations pile-ups, oxygen vacancies, grain boundary defects. As the frequency increases, the species contributing to polarizability lag behind the applied field thereby decreasing the dielectric loss.

From the values of tanδ (Table – 1), a non-linear variation of tanδ with magnitude of dopant (x) is observed. However, the values of tanδ of all the Ti^{4+} doped Mn-Zn ferrites are less than that of undoped one. The decrease in tanδ with Ti^{4+} doping can be attributed to decrease in dielectric constant. The magnitude of values of tanδ is found [39] to range from 1.59 – 2.31 at room temperature in case of bulk Mn-Zn ferrites. In nanocrystalline Mn-Zn ferrites, the improved values of tan δ are reported [40] to be in the range of 0.2 – 0.4. Our earlier reports [24,26] on higher valency .Sb^{5+} and Nb^{5+} doped nanocrystalline Mn-Zn ferrites sintered at 800^{0}C, lower values of tan δ of the order of 10^{-2} are obtained. Similar, loweredvalues oftan δ, i.e., of the order of 10^{-2} are also exhibited by the present Ti^{4+} doped nanocrystalline Mn-Zn ferrites. Observed lower values of tanδ leads to reduced power losses and thus make these materials to be utilized for high frequency applications.Thus, we can conclude that the values of tanδ areimproved with Ti^{4+} doping in nanocrystalline Mn-Zn ferrites.
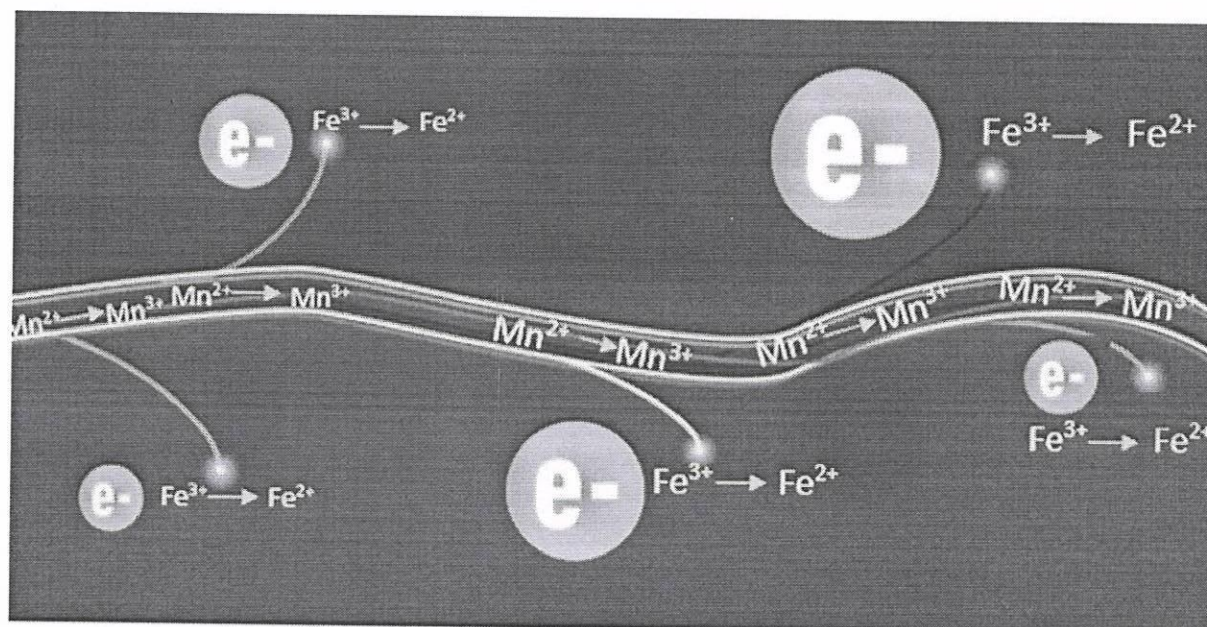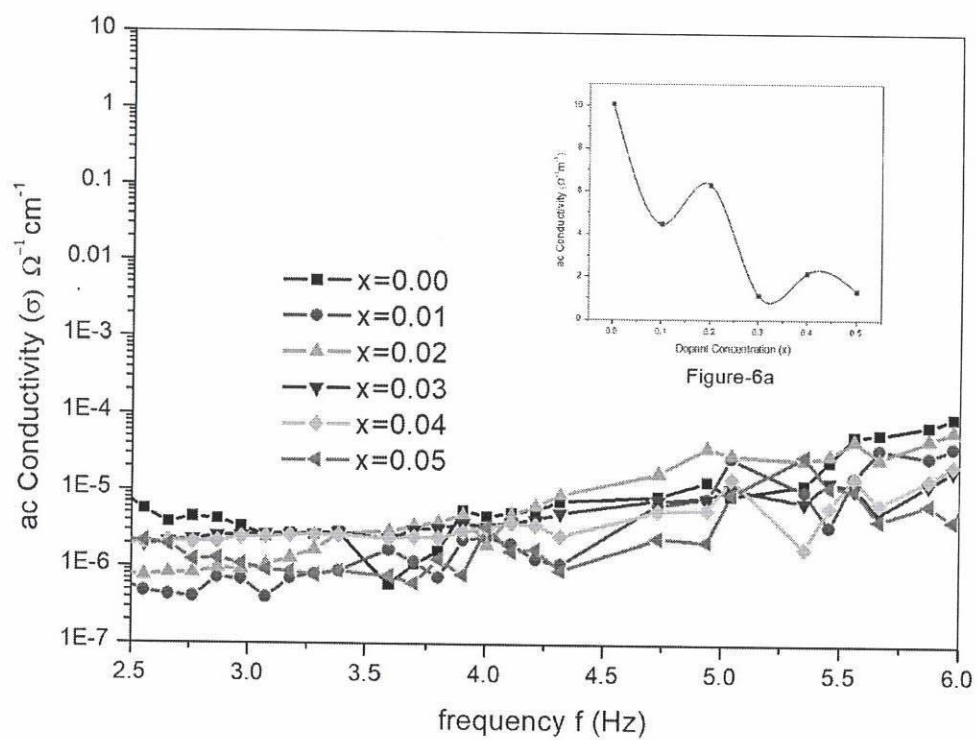
Conductivity Mechanism

Figure – 6 exhibits the variation of ac Conductivity (σ) with frequency for pure and Titanium doped nanocrystalline Mn-Zn ferrites. All thesamples are found to exhibit an increase in conductivity withfrequency in agreement with the reports [44,45] in other ferrite samples. The conductivity mechanism is explained basing on hopping of charge carriers i.e., mainly between Fe^{2+}– Fe^{3+} ions. As the frequency increases, the carriers get effectively transported which leads to an increasing trend of σ with frequency.
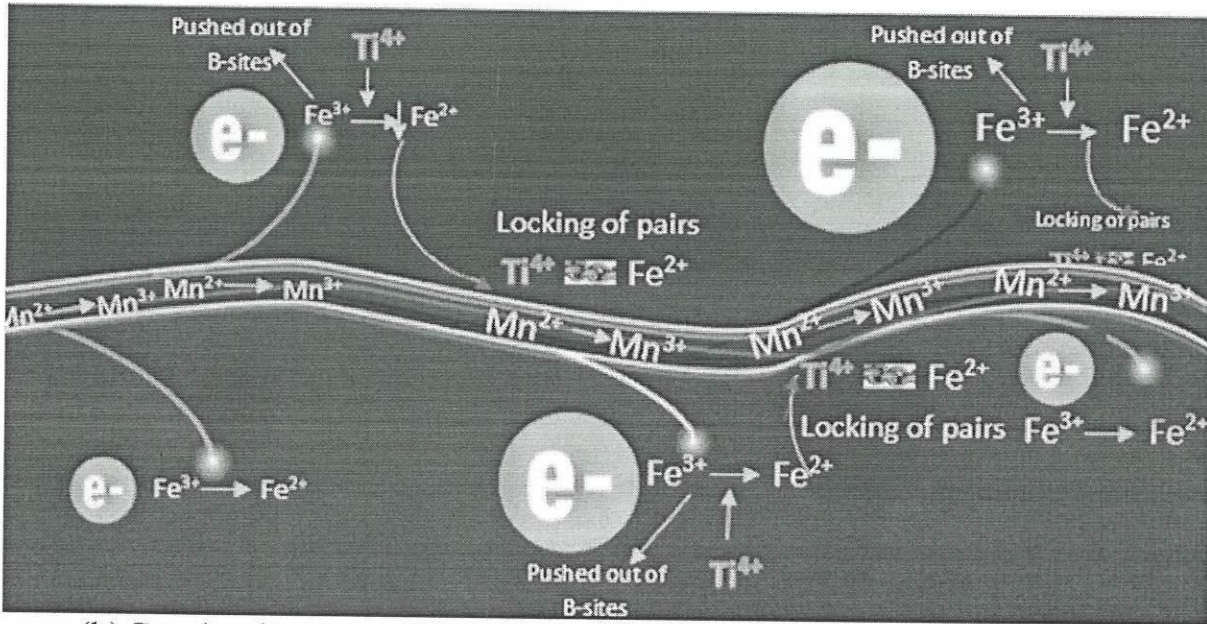
Variation of ac Conductivity (σ) with dopant concentration (x) is presented in inset Figure – 6a and the values are provided in Table – 1. An overall decreasing trend of σ with x can be viewed from Figure – 6a. The value of undoped nanocrystalline Mn-Zn ferrite is 1.01x $10^{-6}\Omega^{-1}$-cm^{-1}. The values of σ.for Ti^{4+} doped nano crystalline Mn-Zn ferrites are found to be of the order of $10^{-7}\Omega^{-1}$-cm^{-1}. Thus, electrical resistivity (ρ) is found to increase with increase in Ti^{4+} concentration. Similar increase in ρ is witnessed with increase in Ti^{4+} concentration in Ni-Zn ferrites also [46].It is reported [34] that as the concentration of Ti^{4+} ions increases, it pushes away some Fe^{3+} ions from B-sites to A-sites and reduce the hopping mechanism thus resulting in decrease of ac conductivity. It is reported [17] that electrical resistivity of Titanium doped Manganese Zinc ferrites gets enhanced by formation of Fe^{2+} - Ti^{4+} pairs at lower Ti^{4+} doping concentrations, while for higher doping levels, it is due to scattering of electrons. As lower concentrations of Ti^{4+} ions (i.e., for x=0 – 0.05) are doped in the present case of nanophased Mn-Zn ferrites, the increase in values of $\rho$is attributed [17] to the formation of Fe^{2+} - Ti^{4+} pairs. Conductivity is found to exhibit an overall decreasing trend with in Ti^{4+} doped ferrites, as Ti^{4+} ions form [47] locking pairs of Ti^{4+}– Fe^{2+}ions to reduce conductivity. From the overview of above reports, the authors sincerely attempted in presenting the conductivity mechanism in Ti^{4+} doped nanophased Mn-Zn ferritesin a pictorial formin Figure – 7.Thus, the increase in values of ρ results in improved power losses required for pushing the operating frequency of the devices to higher

frequencies.Also, several underlying reasons for enhanced power losses with Ti^{4+} doping are also discussed. Under an applied ac field, low-melting insulating phases at the boundaries [18] are formed due to reduction of ohmic currents causing improved power losses. Generally, the electrical conductivity of Mn-Zn ferrites is reported [48]to increase monotonously with increase in concentration of Fe^{2+} ions. Moreover, at higher sintering temperatures, partial reduction of Fe^{3+} to Fe^{2+}ions take place. Hence, from the above factors, the values of ac conductivity should witness an increase in the present case of ferrites sintered at relatively higher temperature of 1000^0C. But, on the contrary, the electrical conductivity is found to decrease. The possible reason may be due to formation [49] of very small amount of Fe^{2+} ions owing to evaporation of Zinc at relatively higher sintering temperatures.

In our previous reports of similar high valency doped Sb^{5+} and Nb^{5+} doped nanocrystalline Mn-Zn ferrites, both sintered at 800^0C, the values of σ are found to vary from $1.65 \times 10^{-7} \Omega^{-1} cm^{-1}$ – $1.83 \times 10^{-6}\Omega^{-1} cm^{-1}$, $1.66 \times 10^{-8} \Omega^{-1} - cm^{-1}$ $-0.03 \times 10^{-8}\Omega^{-1} - cm^{-1}$, respectively.In case of nanophased Mn-Zn ferrites synthesized [18] by ball mill method and codoped with TiO_2 (varying from 0 - 3000 ppm), $CaCO_3$ (500ppm) and SiO(100ppm), the values of dc resistivity are found to get enhancedwith increasing TiO_2 content.The ac values of electrical conductivity in the present Ti^{4+} doped nanocrystalline Mn-Zn ferrites sintered at 1000^0C are found to decrease from $1.09 \times 10^{-6}\Omega^{-1}cm^{-1}$ – $1.15 \times 10^{-7} \Omega^{-1} cm^{-1}$.As the sintering temperature of the present ferrites is obviously lower than that of the reported [24,26] Sb^{5+} or Nb^{5+} doped Mn-Zn ferites, larger values of $tan\delta$ and σ are expected due to zinc losses prevailing [48] at relatively higher sintering temperatures of 1000^0C. On the contrary, improved values of $tan\delta$ and ρ are obtained. Thus, improved dielectric properties and reduced power losses of present nanophased Mn-Zn ferrite samples is attributed to the substitution of Ti^{4+} ions.

Figure-6a

(a) Conduction Mechanism in nano Mn-Zn ferrites

(b) Conduction Mechanism in Titanium doped nano Mn-Zn ferrites

3.5 Magnetic Studies:

The variation of Saturation Magnetisation (M_s) with applied field is shown in Figure – 8 and the variation of M_s with dopant concentration (x) is exhibited in Figure – 9. The values of Saturation magnetization (M_s), Coercivity (H_C) and Retentivity (M_r) are given in Table – 2. From Figure – 8, it is observed that M_s is found to increase with x upto x=0.02 and beyond x = 0.02, it witnesses a decrease with x. Magnetisation of a material is reported [27] to depend mainly upon nature of the dopant, grain size and density or porosity of a material. Initially, the variation of M_s with x can be explained using Neels' two sublattice model. As the magnetic moment of Ti^{4+} ($1.73\mu_B$) ions is less than that of Fe^{3+} ($2.3\mu_B$) ions, an overall decrease in M_s is witnessed with increase in x. It is reported [32] that Ti^{4+} ions have a tendency to enter A-sites for lower concentrations and at higher concentrations, they subsequently occupy B-sites.

For x $\leq$ 0.02, Ti^{4+} ions tend to replace Fe^{3+} ions at A-sites resulting in decrease of magnetization at A-site. So, the net magnetization ($M_B - M_A$) increases thereby causing a rise in the values of

M_s. At higher doping levels, i.e., for $x \geq 0.02$, Titanium ions are argued to replace Fe^{3+} ions at B-sites causing a reduction in net Magnetisation ($M_B - M_A$). Thus, the values of M_s witness a decreasing trend with x. The values of magnetic moment of bulk $Mn_{0.58+x/2} Zn_{0.37+x/2}Ti_xFe_{2.05-2x}O_4$ (for x varying from $0.0 - 0.4$ in steps of 0.1) synthesized by conventional ceramic technique are found [32] to decrease from $3.15\mu_B - 0.92\mu_B$. In the present case of Ti^{4+} doped nanocrystalline Mn-Zn ferrites, the values of magnetic moment are comparatively lower than their bulk counterparts i.e., of the order of $0.001\mu_B - 0.0125\mu_B$. Eventhough Ti^{4+} ions are found to be successful in improving the loss properties, several researchers reported deteriorating values of M_s with Ti^{4+} doping. Ni-Cu-Zn doped with Ti^{4+} ions are found to witness a sudden fall in values of M_s (i.e., from 1.5emu $- 0.1$ emu/gm) and it is attributed to the breaking of domain structure of the material due to introduction of tetravalent titanium ions. A decrease in M_s is also reported [50] in $Mg_{0.95}Mn_{0.05}Fe_{2-2x}Ti_{2x}O_4$ with increase in substitution of Ti^{4+}ions and it is due to dilution of sublattice by non-magnetic Ti^{4+} ions resulting in weakening of exchange interaction in the system. With increase in TiO_2 additions in Ni-Zn soft ferrites, the values of M_s are found [51] to reduce with decrease in Fe^{3+} ions due to their replacement by Ti^{4+} ions. Similar decreasing trend of M_s with x is also observed in the present case Ti^{4+} doped nanophased Mn-Zn ferrites. Secondly, the lower values of M_s are correlated tonanosized grains obtained in Mn-Zn ferrites with Ti^{4+} doping. In nanoparticles an inert or dead layer is formed at the surface which prevents the spins to align along the field direction. In addition to this due to large surface to volume ratio in nanomaterials there will be spin glass like layer (canted spins)[52,53] at the surface which reduces the values of M_s. These canted spins are representative of triangular spin arrangements on the B-Siteresulting in a fall of M_s. Finally, the observed lower values of M_s are also explained interms of density. Generally, the magnetic properties of a material are reported [54] to be dependent on the porosity or density of the material. The demagnetizing factor is directly proportional to porosity of the material. Hence, the materials with relatively higher

values of porosity or lower values of density are prone to exhibit lower values of M_s. Thus, the values of M_s for the present Ti^{4+} doped nanocrystalline Mn-Zn ferrites are also attributed to relatively lower values of density obtained in the present samples. Thus, the competing factors like nature of the dopant, grain size and density can be considered as the major factors for the observed lower values of M_s.

The nanosized Ti^{4+} doped nanocrystalline manganese zinc ferrite powder are found to exhibit super- paramagnetic behaviour. Complex domain structure is exhibited [55] by large-grain-size ferrites. As the grain size is reduced, the number of domains decreases, and a transition from a polydomain to a single-domain occurs. At a still smaller grain size, the ferrite particles undergo a phase change to a superparamagnetic solid. These particles may show a type of magnetic Brownian movement and tend to behave similar to a paramagnetic atom with a very large magnetic moment. In a single-domain particle, all the spins are aligned in the same direction and the particle is uniformly magnetized. Because there are no domain walls to move, the magnetization will be reversed through spin rotation rather than through the motion of domain walls. This results in large coercivity of the nanoparticles. Relatively larger values of H_c are observed in the present case of Ti^{4+} doped nanocrystalline Mn-Zn ferrites. The variation of H_c with crystallite size (**D**) is provided in Figure – 10. An overall view of Figure -10 reveals that the values of H_c are found to increase with D upto a critical size of 50nm. Beyond **D** = 62 nm, the values of H_c are found to increase with **D**. Under this critical diameter which typically lies in the range of a few tens of nanometers (and depends on the type of material), the particle will consist of a single domain. Beyond **D** = 62 nm, the Ti^{4+} doped Mn-Zn ferrites exhibit multidomain structure. From Figure-10, the values of H_c are approaching to nearly zero values at a critical size of ~49 nm exhibiting superparamagnetim. The particle size limit incase of $MnFe_2O_4$ is reported [8,9] to be 42nm and 42.9 nm, respectively, below which the ferrites exhibit

superparamagnetic behavior. Hydrothermally synthesized [56] nanosized $Mn_{0.5}Zn_{0.5}Fe_2O_4$
exhibit superparamagnetism for grain sizes less than 100 nm beyond which they exhibit
ferromagnetism. Superparamagnetism is witnessed [11,12] in La^{3+} doped Mn-Zn ferrites for a
critical size of nearly 57.14 nm and for Gd^{3+} doped Mn-Zn ferrites for a critical size of around 45
nm. Hence, the critical range of **D** (~49nm)in Ti^{4+} doped nanocrystalline Mn-Zn ferrites is found
to agree with reported values. Thus, the phenomena of superparamagnetism observed at
relatively higher critical sizes in the present Mn-Zn-Ti ferrites make these materials usable for
technological and biomedical applications.
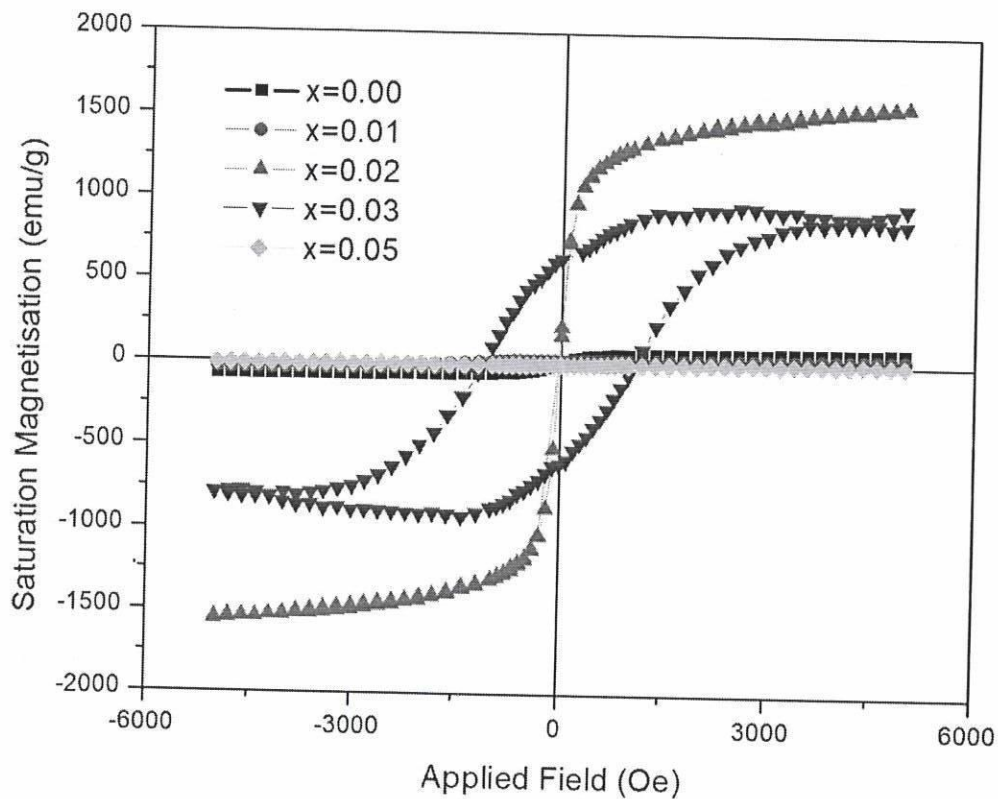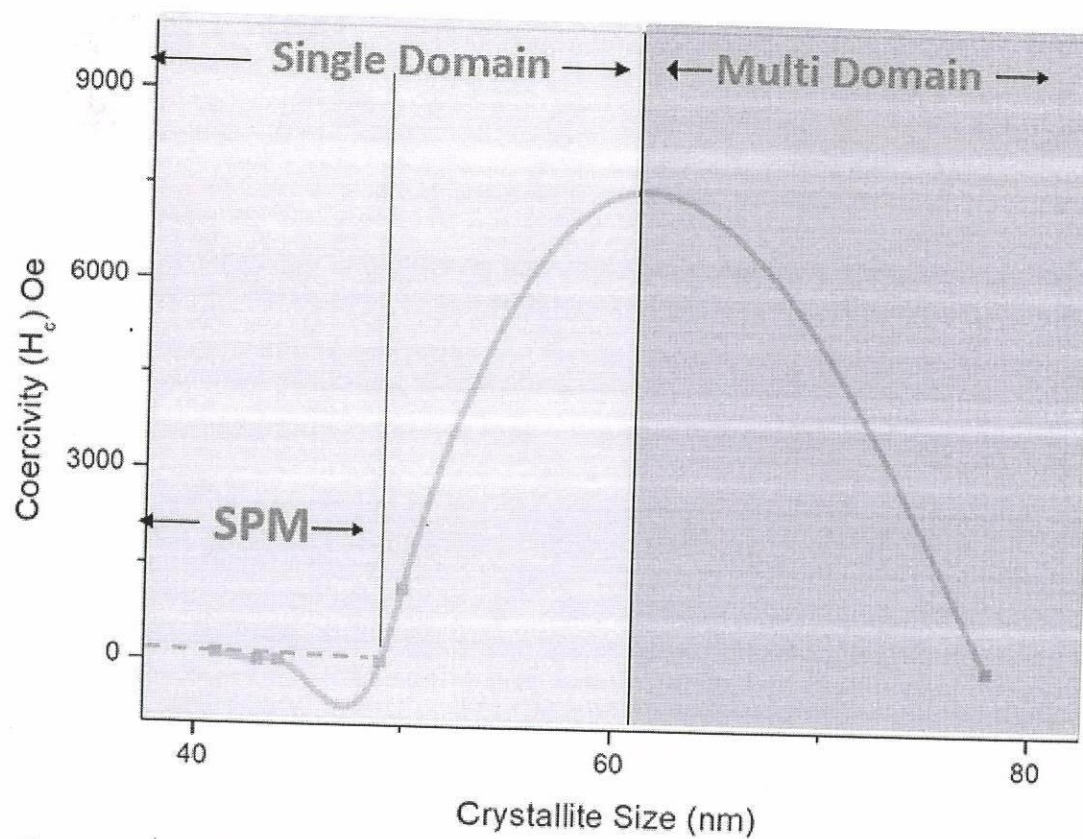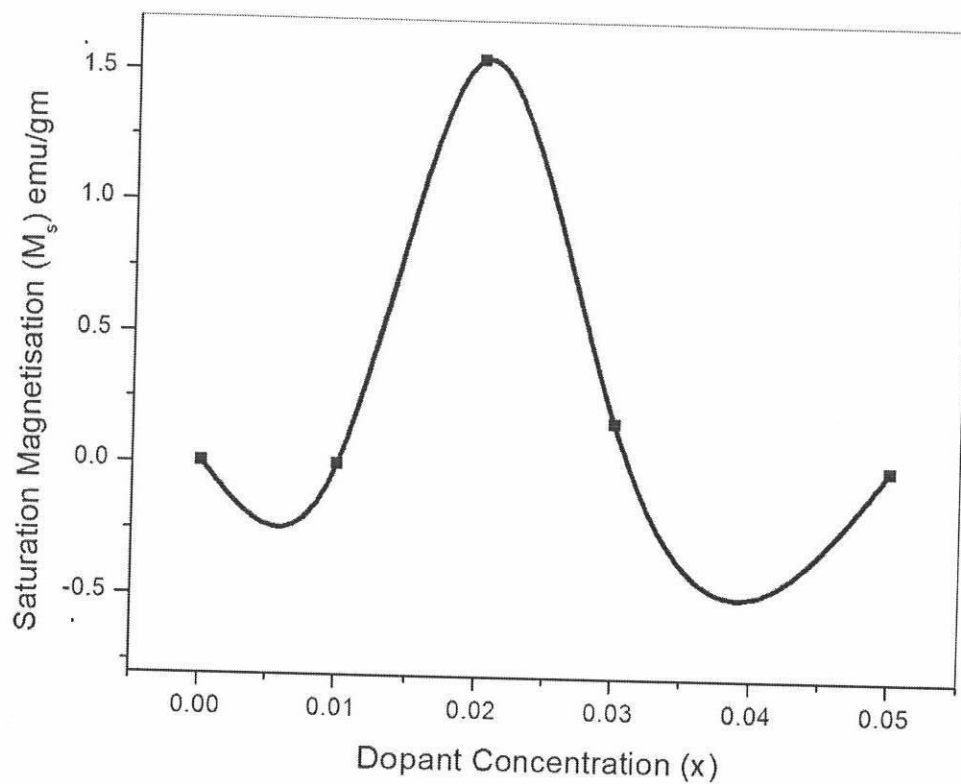
SPM = Superparamagnetism

Composition	M_s x 10^{-1} (emu/gm)	H_c (Oe)	M_r x 10^{-2} (emu/gm)	Magnetic Moment (n_B)
x = 0.00	0.70	0.05	0.31	0.0006
x = 0.01	0.27	0.36	1.94	0.0002
x = 0.02	15.5	0.40	2.19	0.0112
x = 0.03	16.9	1140	61.8	0.0153
x = 0.04	1.92	111	5.17	0.0018
x = 0.05	1.48	0.06	0.33	0.0001

3.6

Conclusions

Influence of Ti^{4+}ions on Structural, dielectric and magnetic properties of nanophased Mn-Zn ferrites synthesized by hydrothermal method infer that

- Formation of pure spinel phase of nanophased Mn-Zn-Ti ferrites without any extra peaks isascribed to the relatively higher sintering temperature of 1000^0C.

- Non-linear variation of lattice parameter (a) with x is explained by occupancy of Ti^{4+} ions in both tetrahedral and octahedral sites.

- Replacement of Ti^{4+} ions with Fe^{3+} ions in Mn-Zn ferrites results in an overall decreasing trend of D with x.

- Dielectric parameter $\varepsilon'$is found to exhibit grain size (D) dependent behavior.Improved dielectric properties and reduced power losses anticipated with increasing Ti^{4+} concentration are attributed to hindered hopping mechanism by locking of $Ti^{4+} - Fe^{2+}$ pairs.

- Reduced values of M_s with doping x due to spin canting mechanism and triangular spin arrangementin nanosized grains and lowered density of the samples.

- Attainment of zero value of $H_c$upto a critical size of ~49nm denotes the superparamagnetic nature of nanophased Mn-Zn-Ti ferrites.

- Lowered values of ε', tanδ and higher values of ρ render these materials usable for high frequency applications.

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PAPER

Dielectric properties of superparamagnetic titanium doped nanophased Mn–Zn ferrites for high frequency applications

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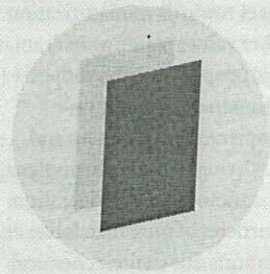
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zero is referred to as superparamagnetism (SPM). Beyond the critical size, coercivity decreases with increase in crystallite size reflecting upon the multi-domain nature of the ferrites. Occurrence of SPM is reported [8–12] in $MnFe_2O_4$, $MnZnFe_2O_4$, Lanthanum and Gadalonium doped Mn–Zn ferrites by other researchers.

Among a wide variety of techniques that exist for the synthesis of ferrites, Hydrothermal synthesis is proven [13] to be relatively user friendly and cost-effective technique which needs comparatively lower sintering temperatures, less energy and has an advantage of producing less agglomerated particles of controlled size. It is well recognized [14] in the field of electroceramics that the nature and the amount of dopant is one of the most important operational parameters towards tailoring or improving properties and performance of the product.

TiO_2 is an effective additive in improving [1] electromagnetic properties by replacing Fe^{3+} ions with Ti^{4+} ions at octahedral sites of the spinel lattice.Simultaneous codoping of TiO_2, $CaCO_3$ and SiO in the Mn–Zn ferrite lattice is found [15] to lead to the formation of Fe^{2+}–Ti^{4+} pairs replacing Fe^{3+} ions in the octahedral B-sites hindering the electron hopping, thus resulting in an increase of dc electrical resistivity and improved power losses of the material. An overall increase in ac resistivity and decrease in saturation magnetisation is observed [16] with increase in Ti^{4+} concentration in $Mn_{1-x}Fe_{2-2x}Ti_xO_4$ ferrites. Addition of Ti^{4+} ions in Ni–Zn ferrites is reported [17] to exhibit improved values of electrical resistivity while it is found to exhibit an adverse effect on saturation magnetization.Higher values of electrical resistivity are obtained [18] in Cobalt and Titanium doped Mn–Zn ferrites.

Incorporation of tetravalent ions like TiO_2 or SnO_2 into ferrites is reported [19] to be capable of development of high resistivity ferrites as they form stable bonds with Fe^{2+} ions and they impede the process of electron hopping. Moreover, it is also reported [1] that addition of pentavalent ions is useful in producing high conductivity ferrites whereas tetravalent ions have the ability to produce ferrites of comparatively higher values of electrical resistivity. In our previous work, for both the cases of Sb^{5+} and Nb^{5+} doped nanocrystalline Ni-Zn ferrites synthesized [20, 21] by hydrothermal method, we have obtained XRD patterns with pure spinel phase, exhibiting relatively larger values of saturation magnetisation (M_s), higher values of ac resistivity (ρ) and lower loss factor ($\tan \delta$) values which are viable for high frequency applications. When similar dopants, i.e., Sb^{5+} and Nb^{5+} are introduced [22, 23] into nanophased Mn–Zn ferrites, XRD patterns show certain hematite peaks along with spinel phase accompanied by higher values of ρ and lower values of $\tan \delta$ and M_s.Titanium ions dissolve [24] in the lattice and occupy regular tetrahedral and octahedral positions of the lattice and change the dielectric and magnetic properties. Electrical resistivity is found to be improved by formation of a liquid phase at the grain boundaries but addition of Ti^{4+} has a negative effect on the values of M_s. Even though extensive research work is carried out on bulk Ti^{4+} doped Mn–Zn ferrites, synthesis of nanophased Ti^{4+} doped Mn–Zn ferrites by hydrothermal method and their dielectric and magnetic response to investigate the property of superparamagnetism has not been elaborated so far.In view of the above reports on Ti^{4+} doped ferrites and other pentavalent Sb^{5+} and Nb^{5+} doped ferrites, a humble attempt is made to synthesize nanophased$Mn_{0.5}Zn_{0.5}Ti_xFe_{2-4x/3}O_4$ ferrites by hydrothermal method at lower dopant concentrations i.e., for x varying from 0–0.05 insteps of 0.01 and characterize them for possible applications.

2. Experimental

2.1. Preparation

Nanophased $Mn_{0.5}Zn_{0.5}Ti_xFe_{2-4x/3}$(x = 0.0, 0.01, 0.02, 0.03, 0.04 and 0.05) are prepared using hydrothermal method. The starting precursors are A.R.Grade $Mn(NO_3)_2,6H_2O$, $Zn(NO_3)_2, 6H_2O$, $Fe(NO_3)_3, 9H_2O$ and TiO_2.Stoichometric quantities of the required materials are dissolved in deionised water and using Ammonia solution,P^H of 8 is obtained. The solution is then thoroughly mixed using magnetic stirrer for nearly one hour and is then heated in a autoclave at a temperature of 200 °C for four hours. After allowing to cool naturally, the solution is taken out and cleaned using deionised water. Hot air oven is used to dry the sample at 60 °C for 24 h. The material is then taken out and powdered using an agate mortar. The powder is then sintered at a temperature of 500 °C for 6 h. Pellets of 12 mm diameter are prepared at a pressure of 5Tons. The pellets are then sintered at a temperature of 1000 °C for 6 h. The sintered pellets are used for structural,electrical and Magnetic characterization.

2.2. Structural Studies

XRD studies were carried out with a Bruker (Germany, Model: D8) x-ray Diffractometer operated with CuK_α radiation with $\lambda = 1.5406$ Å. The lattice parameter (**a**) corresponding to the observed prominent reflections (identified with the help of standard JCPDS Card No. 10–0467) is estimated using the equation
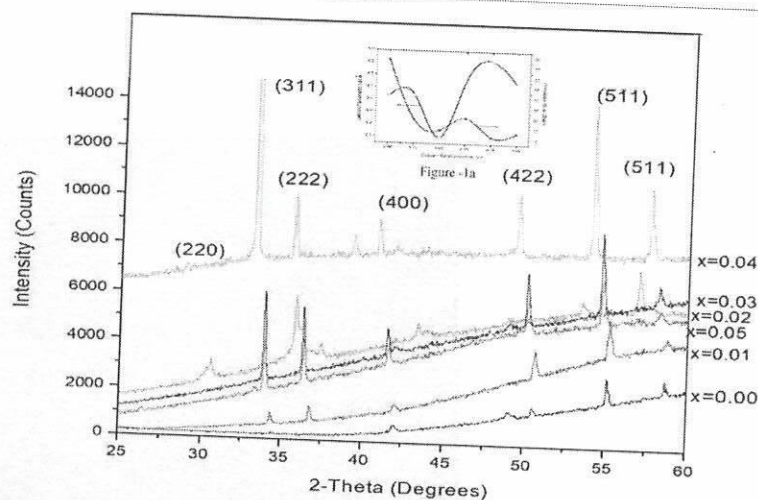
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Figure 1. XRD patterns for Titanium doped Mn–Zn nano ferrites. (a) Variation of Lattice parameter (a) and Grain size (D) with ·Titaniumconcentration (x) in nano phased Mn–Zn nano ferrites.

In our earlier reports [22, 23] of Sb^{5+} doped and Nb^{5+} doped Mn–Zn ferrites synthesized by hydrothermal method, the ferrites are sintered at 800 °C and an extra hematite phase is observed along with the spinel phase. But, in the present case of Ti^{4+} doped nanocrystalline Mn–Zn ferrites sintered at 1000 °C, XRD patterns exhibit pure spinel phase with no additional hematite peaks and it is attributed [28] to high temperature sintering conditions of 1000 °C.Relative broadening of XRD peaks evince the formation of nanocrystalline ferrites. The values of lattice parameter (**a**) determined from XRD data are provided in table 1 and its variation with dopant concentration (x) is presented in figure 1(a). The values of **a**are found to vary from 8.3030 A to 8.926 A. From figure 1 (a), it is observed that at lower concentrations (i.e., upto x = 0.02), **a** is found to decrease with x. For intermediate concentrations, i.e., from x = 0.02 to x = 0.04, the values of **a**exhibit an increasing trend. Beyond x = 0.04, again a decreasing trend of **a** with x is witnessed. Mn–Zn ferrite is a mixed spinel ferrite, with Mn^{2+}, Zn^{2+} and Fe^{3+} ions are argued to occupy both tetrahedral (A-sites) and octahedral sites (B-sites), whereas Mn^{3+} and Fe^{2+} ions occupy octahedral sites only. Ti^{4+} ions are reported [29] to have a tendency to enter both A- and B-sites.The ionic radius of Ti^{4+} (0.56 A)ions is less than that of Fe^{3+} ions (0.63 A) at A—sites. So, the initial decrease of **a** with x upto x = 0.02 is attributed to the replacement of Ti^{4+} ions with Fe^{3+} ions at A-sites. As the ionic radius of Ti^{4+} (0.74 A) ions at B-sites is larger than that of Fe^{3+}(0.69 A) ions, they tend to replace Fe^{3+} ions at B-sites resulting in increase of lattice parameter. Again, the decrease in **a** for x = 0.05 is explained by the influence of repulsion parameter (b) and Madelung Constant (M). The lattice parameter **a** is proportional [30] to (b/M). At higher concentrations, more Ti^{4+} enter into B-sites, thus pushing more charge from B-sites to A-sites resulting in an increase of M. Thus, b/M decreases. Hence, lattice constant decreases for x = 0.05. It is also reported [31] that in case of ferrites that are neither completely normal spinel nor inverse spinel, the lattice parameter **a** is found to exhibit a non-linear behavior with dopant concentration (x). Thus, the non-linear behavior of lattice constant of nanocrystalline Mn–Zn ferrites with Titanium concentration in the present case can also be attributed to the mixed spinel structure of Mn–Zn–Ti ferrites.

The crystallite size (**D**) is calculated using the (311) peak and the values are provided in table 1. The values of **D** are found to be in the range of 78 nm–41 nm. An overall decreasing trend of **D** with x is presented in figure 1(a). In case of Sb^{5+} doped nanocrystalline Mn–Zn ferrites, the crystallite size is reported [22] to be in the range of 46 nm–14 nm for Sb^{5+} doped ferrites, whileincase of Nb^{5+} doped ferrites, **D** is found [23] to varyfrom 50 nm–14 nm. Relatively larger values of **D** in the present case of Ti^{4+} doped Mn–Znferrites as compared to that of reported [22, 23] values of other high valency dopedMn–Zn ferrites isattributed to higher sintering temperature of 1000 °C employed in the present case. The crystallite size is reported [32] to increase from 74 nm–137 nm in Ni–Ti dopedMn ferrites synthesized by standard ceramic method and sintered at 1300 °C. Decrease in **D** with Ti^{4+} doping in the present case of Mn–Zn ferrites vouches for the successful substitution of dopant by the present hydrothermal method adopted in the synthesis of the ferrites.

. The representative FESEM Micrographs for x = 0.00, x = 0.01 and x = 0.05are presented in figures 2(a)–(c). The images are clear and lesselectrostatically distorted. Well crystallized grains with almost uniform size can be
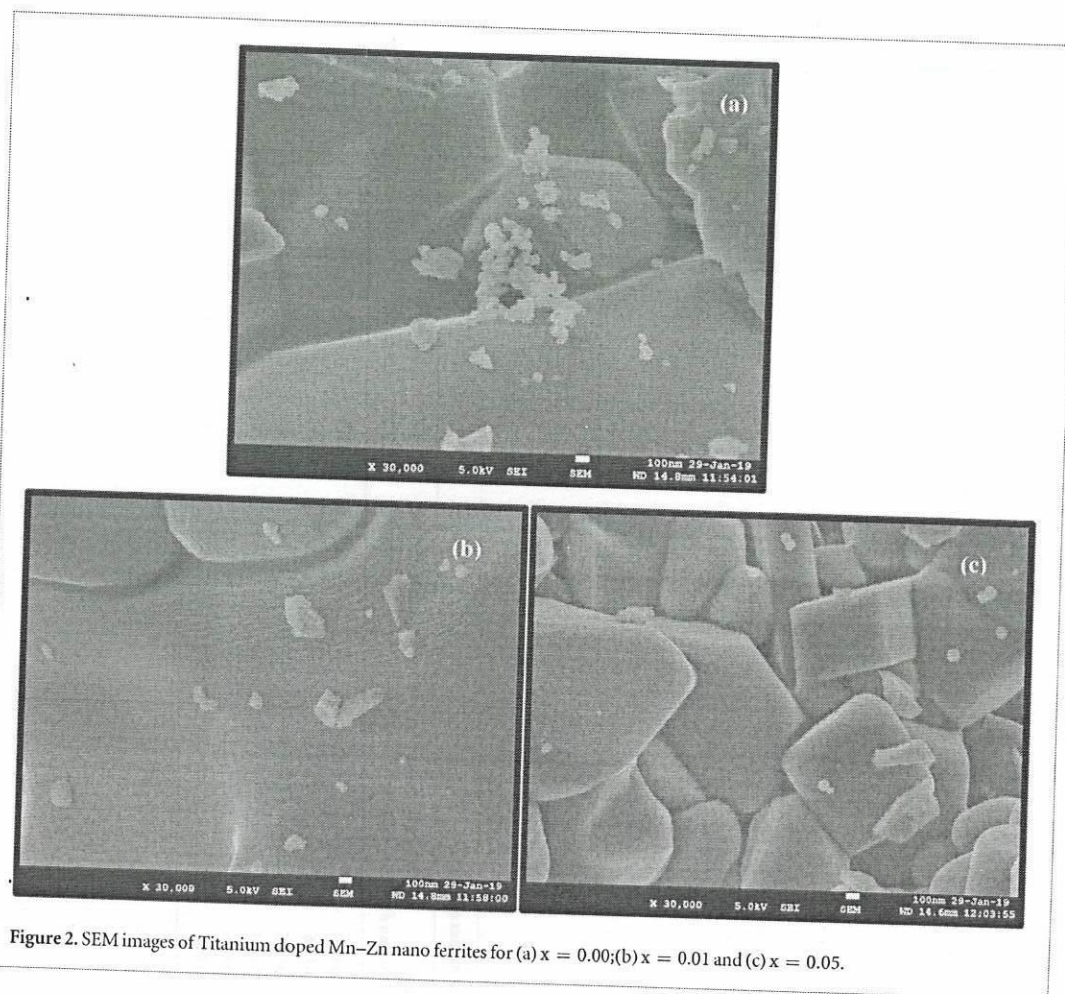
Figure 2. SEM images of Titanium doped Mn–Zn nano ferrites for (a) x = 0.00;(b) x = 0.01 and (c) x = 0.05.

observed. Decrease in particle size with increase in Ti^{4+} dopant concentration can be clearly observed from SEM micrographs. An overall view of structural properties indicate that the present hydrothermal method is successful in synthesizing single phase nanocrystalline Titanium doped Mn–Zn ferrites.

3.2. Dielectric studies

Variation of Dielectric constant (ε') is found to exhibit (figure 3) a non-linear behavior with x. However, an overall decreasing trend of $3\varepsilon'$ with increase in x can be witnessed due to reduced [31] hopping probability between Fe^{3+} and Fe^{2+} ions by replacement of Ti^{4+} ionswith Fe^{3+} ions at B-sites. Relatively lower values of ε' ranging from 29–18 are obtained in the present case of Titanium doped nanocrystalline Mn–Zn ferrites. From figure 3, a correlative trend between ε' and D with x is observed. Increasing values of ε' with Dare attributed [33, 34] to the increasing number of iron ions at octahedral sites with **D**. Thus, the anomalies in the values of ε' with x are correlated to grain size. Similar grain size dependence of dielectric constant is also reported [22, 23] in Sb^{5+} and Nb^{5+} doped Mn–Zn ferrites. Moreover, the variation of dielectric constant is found [19] to be inverse to that of lattice parameter, except for x = 0.02 which is ascribed to the relatively higher density of the sample. The values of ε' in bulk Mn–Zn ferrites synthesized by ceramic method are found [35] to be of the order of $(17-32) \times 10^5$ at room temperature while for nanocrystalline Mn–Zn ferrites, the values of ε' are reported [36] to be reducedby an order of 10^4 and they are found to be 85, 79, 395 for crystallite sizes of 65 nm, 59 nm and 11 nm, respectively. In case of Ti^{4+} doped nanocrystalline Ni-Zn ferrites, the values of ε' are reported [37] to be of the order of 10^3. In our previous work of Sb^{5+} and Nb^{5+} doped nanocrystalline Mn–Zn ferrites synthesized [21, 23] by hydrothermal method, the order of magnitude of ε' is 10^2. At room temperature, Ti^{4+} doped Manganese ferrites are found [38] to have values of ε' ranging from 10 to 100 at1 MHz frequency. In the present case, the values of ε' are as low as of the order of 29–18. As reported [24] by other researchers earlier, Titanium is found to improve the values of ε' in the present synthesized Mn–Zn–Ti ferrites and make these materials viable for high frequency microwave applications.

      Variation of dielectric constant (ε') with varying ac frequency from100 mHz–4 MHz is shown in figure 3(a). ε' is found to witness a decreasing trend with frequency since the electron exchange between $Fe^{2+} \leftrightarrow Fe^{3+}$ of

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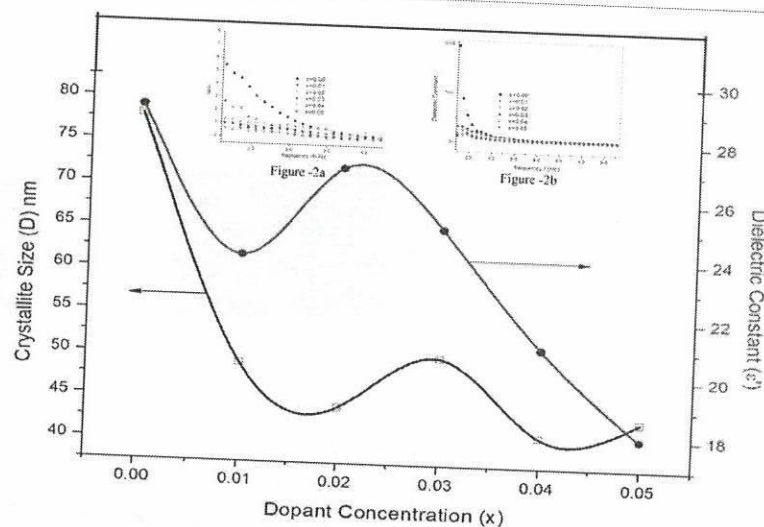
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Figure 3. Dielectric Constant and grain size D with Titanium doping in Mn–Zn nano ferrites. (a) Inset graph of Frequency variation of dielectric constant $\varepsilon'(\omega)$ in Titanium doped Mn–Zn nano ferrites. (b) Inset graph of Frequency variation of Loss factor (tan δ) with Titanium doping in Mn–Zn nano ferrites.

Mn–Zn ferrites does not follow [39] the AC fieldresulting in decrease of polarization. Variation of dielectric loss factor (tan δ) as a function of frequency for varying Ti^{4+} concentrations in nanocrystalline Mn–Zn ferrites (figure 3(b)) exhibits a steep decrease with frequency at lower frequencies while a marginal decrease is witnessed at higher frequencies, as reported [40].

From the values of tan δ (table 1), a non-linear variation of tan δ with magnitude of dopant (x) is observed and the values of all the Ti^{4+} doped samples are lower as compared to that of undoped sample. The decrease in tan δ with Ti^{4+} doping can be attributed to decrease in dielectric constant. The magnitude of values of tan δ isreported [35] to range from 1.59–2.31 at room temperature in case of bulk Mn–Zn ferrites and in nanocrystalline Mn–Zn ferrites, the values are improved [36] and are found to be in the range of 0.2–0.4. In our earlier reports [22, 23] on higher valency Sb^{5+} and Nb^{5+} doped nanocrystalline Mn–Zn ferrites sintered at 800 °C, lower values of tan δ of the order of 10^{-2}–10^{-3} are obtained. Similar, lowered values of tan δ, i.e., of the order of 10^{-2} are also exhibited by the present Ti^{4+} doped nanocrystalline Mn–Zn ferrites. Observed lower values of tan δ leads to reduced power losses and thus make these materials usable for high frequency applications. Thus, we can conclude that the values of tan δ are improved with Ti^{4+} doping in nanocrystalline Mn–Zn ferrites.

3.3. Conductivity mechanism

Figure 4 exhibits the variation of ac Conductivity (σ) with frequency for pure and Titanium doped nanocrystalline Mn–Zn ferrites. All the samples are found to exhibit an increase in conductivity with frequency in agreement [41, 42] with the reportsof other ferrite samples.

Variation of ac Conductivity (σ) with dopant concentration (x) is presented in inset figure 4(a) and the values are provided in table 1. An overall decreasing trend of σ with x can be viewed from figure 4(a). From table 1, the electrical resistivity (ρ) is found to rise by an order of 10 with increase in Ti^{4+} concentration due to hindered [34] hopping between Fe^{2+} and Fe^{3+} ions by pushing away of Fe^{3+} ions to octahedral sites by Ti^{4+} ions.Similar increase in ρ is witnessed with increase in Ti^{4+} concentration in Ni-Zn ferrites also [43]. Conductivity is found to exhibit an overall decreasing trend with in Ti^{4+} doped ferrites, as Ti^{4+} ions form [24] locking Ti^{4+}–Fe^{2+}ion pairs to reduce conductivity. From the overview of above reports [16–19, 24], the authors sincerely attempted in presenting the conductivity mechanism in Ti^{4+} doped nanophased Mn–Zn ferritesin a pictorial formin figures 4(c) and (d). Thus, the increase in values of ρ results in improved power losses required for pushing the operating frequency of the devices to higher frequencies.Also, several other underlying reasons for enhanced power losses with Ti^{4+} doping are also discussed. Under an applied ac field, low-melting insulating phases at the boundaries [15] are formed due to reduction of ohmic currents causing improved power losses. Generally, the electrical conductivity of Mn–Zn ferrites is reported [44] to increase monotonously with increase in concentration of Fe^{2+} ions. Moreover, at higher sintering temperatures, partial reduction of Fe^{3+} to Fe^{2+}ions take place. Hence, from the above factors, the values of ac conductivity
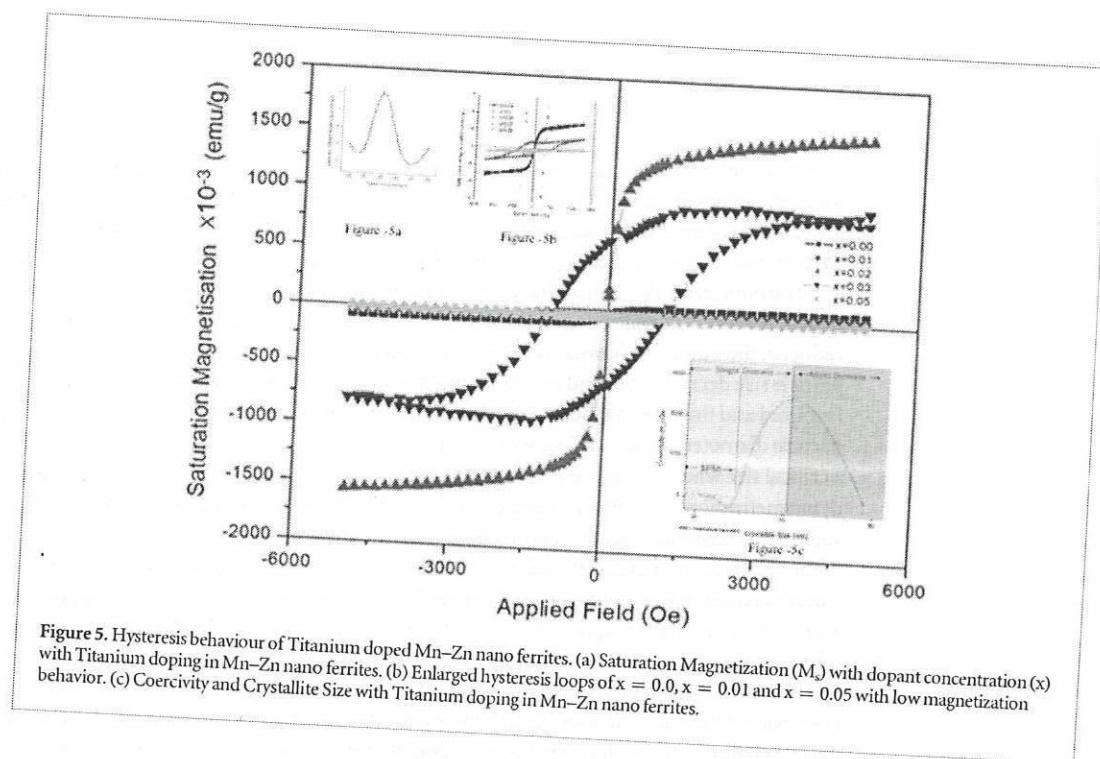
Figure 5. Hysteresis behaviour of Titanium doped Mn–Zn nano ferrites. (a) Saturation Magnetization (M_s) with dopant concentration (x) with Titanium doping in Mn–Zn nano ferrites. (b) Enlarged hysteresis loops of x = 0.0, x = 0.01 and x = 0.05 with low magnetization behavior. (c) Coercivity and Crystallite Size with Titanium doping in Mn–Zn nano ferrites.

For x ⩽ 0.02, Ti^{4+} ions tend to replace Fe^{3+} ions at A-sites resulting in decrease of magnetization at A-site. So, the net magnetization (M_B–M_A) increases thereby causing a rise in the values of M_s. At higher doping levels, i.e., for x ⩾ 0.02, Titanium ions are argued to replace Fe^{3+} ions at B-sites causing a reduction in net Magnetisation(M_B–M_A). Thus, the values of M_s witness a decreasing trend with x. The values of magnetic moment of bulk $Mn_{0.58+x/2}$ $Zn_{0.37+x/2}Ti_xFe_{2.05-2x}O_4$ (for x varying from 0.0–0.4 in steps of 0.1) synthesized by conventional ceramic technique are found [29] to decrease from 3.15 μ_B–0.92 μ_B. In the present case of Ti^{4+} doped nanocrystalline Mn–Zn ferrites, the values of magnetic moment are comparatively lower than their bulk counterparts i.e., of the order of 0.001 μ_B–0.015 μ_B. Eventhough Ti^{4+} ions are found to be successful in improving the loss properties, several researchers reported deteriorating values of M_s with Ti^{4+} doping. Ni–Cu–Zn doped with Ti^{4+} ions are found to witness a sudden fall in values of M_s (i.e., from 1.5 emu–0.1 emu gm^{-1}) and it is attributed to the breaking of domain structure of the material due to introduction of tetravalent titanium ions. A decrease in M_s is also reported [49] in $Mg_{0.95}Mn_{0.05}Fe_{2-2x}Ti_{2x}O_4$ with increase in substitution of Ti^{4+} ions and it is due to dilution of sublattice by non-magnetic Ti^{4+} ions resulting in weakening of exchange interaction in the system. With increase in TiO_2 additions in Ni–Zn soft ferrites, the values of M_s are found [50] to reduce with decrease in Fe^{3+} ions due to their replacement by Ti^{4+} ions. Similar decreasing trend of M_s with x is also observed in the present case Ti^{4+} doped nanophased Mn–Zn ferrites. Secondly, the lower values of M_s are correlated to nanosized grains obtained in Mn–Zn ferrites with Ti^{4+} doping. In nanoparticles an inert or dead layer is formed at the surface which prevents the spins to align along the field direction. In addition to this due to large surface to volume ratio in nanomaterials there will be spin glass like layer (canted spins) [51, 52] at the surface which reduces the values of M_s. These canted spins are representative of triangular spin arrangements on the B-Site resulting in a fall of M_s. Lower values of M_s is also attributed to the lower sintering temperature of 900 °C. Increase in sintering temperature leads to increment in density, grain size. The increase in grain size inturn results in increase of M_s. Magnetic properties in nanophased ferrites can also be correlated [53–55] to finite size effects related to the number of exchange-coupled spins within nanoparticles, reduced symmetry of atoms at the surface and inter-particle interactions of agglomerated magnetic nanoparticles. It is also reported that in case of superparamagnetic nano iron clusters, sintering temperature is reported to weaken the inter-particle interactions causing increased values of M_s. Magnetic properties of ferrites are also reported [55] to depend upon the shapes of the nanophased samples. M_s is found to decrease with increasing thickness off the sample. Hence, in the present case of Ti^{4+} doped ferrites, the values of M_s can be increased by raising the sintering temperatures to reduce the agglomerating trends which inturn reduce the interparticle interactions. Coating with insulating thick silica shell can also increase the values of M_s. Finally, the observed lower values of M_s are explained interms of density. Generally, the magnetic properties of a material are reported [56] to be dependent on the porosity or density of the material. The demagnetizing factor is directly proportional to porosity of the material. Hence, the

domestic television receivers as core materials for line time base transformers. Enhanced electrical resistance and improved dielectric losses obtained in the present TiO_2 doped Mn–Zn ferrites make them usable in switching power supply transformers, fly back transformers or deflection yoke, domestic radio transformers, various inductance elements, impedance elements for EMI countermeasure and electromagnetic wave absorbers.

3.5.2. Biological applications

The basic requirements for biotechnology applications are explained Superparamagnetism observed in the present Ti^{4+} doped Mn–Zn nanoferrites make [59] them suitable for biomedical and biotechnology applications like hyperthermia, magnetic resonance imaging contrast agents, and targeted drug delivery. Biomedical applications require particles that are biocompatible, less toxic and relatively smaller size.

In magnetic drug delivery, the drug molecules are attached to a functionalized magnetic nanoparticle and guided to a chosen site using an external magnetic field and they stay at that site until therapy is complete and after that they are removed. Nanoferrites with good superparamagnetic properties like Fe_3O_4 are gaining increasing attention in the field of targeted drug delivery and cell imaging.

Hyperthermia treatment is considered to be a treatment along with chemotherapy, radiotherapy and surgery in cancer therapy. When a varying magnetic field is applied to a superparamagnetic nanoparticle, heat is generated by magnetic hysteresis loss, Neel relaxation and the Brownian relaxation effect. This results in a rise of temperature upto nearly 40 °C–45 °C. As the tumor cells are more sensitive to heat than normal cells, they are destroyed. Nanoferrites and fluorescent magnetic nanocomposites have been widely used *in vivo* as magnetic resonance imaging (MRI) contrast agents for molecular and cell imaging. Magnetic separation can be used as a quick and simple method for capturing specific proteins or biomolecules efficiently and reliably without using expensive liquid chromatography or other techniques.

For biological applications, the nanoparticles should be biocompatible and non-toxic, preferably sufficiently small (10–50) nm leading to large surface to volume ratio that results in improving efficiency of coating (and also the attachment of ligands) in reducing agglomeration and better targeting, Smaller nanoparticles remain in the circulation after injection and pass through the capillary systems of organs and tissues avoiding vessel embolism and they avoid precipitation due to gravitational forces. They should also possess higher values of M_s to control the movement of particles,close to the target pathologic tissue in the blood with moderate external magnetic field.

3.6. Conclusions

Influence of Ti^{4+} ions on Structural, dielectric and magnetic properties of nanophased Mn–Zn ferrites synthesized by hydrothermal method infer that

- Formation of pure spinel phase of nanophased Mn–Zn–Ti ferrites without any extra peaks is ascribed to the relatively higher sintering temperature of 1000 °C.

- Non-linear variation of lattice parameter (**a**) with x is explained by occupancy of Ti^{4+} ions in both tetrahedral and octahedral sites.

- Replacement of Ti^{4+} ions with Fe^{3+} ions in Mn–Zn ferrites results in an overall decreasing trend of **D** with x.

- Dielectric parameter ε' is found to exhibit grain size (**D**) dependent behavior. Improved dielectric properties and reduced power losses anticipated with increasing Ti^{4+} concentration areattributed to hindered hopping mechanism by locking of Ti^{4+}– Fe^{2+} pairs.

- Reduced values of M_s with doping x due to spin canting mechanism and triangular spin arrangementin nanosized grains and lowered density of the samples.

- Attainment of zero value of $H_c$uptoa critical size of ~49 nm denotes the superparamagnetic nature of nanophased Mn–Zn–Ti ferrites.

- Lowered values of ε', tan δ and higher values of ρ render these materials usable for high frequency applications.

Acknowledgments

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