CONTENTS

Description

PART I

1. **Title of the project**: Synthesis and Development of Broadband EMI Shielding Materials using Magneto-Dielectric-Nanoparticles

2. **Objective of the Project:**

   - Development of EMI shielding material with enhanced shielding effectiveness (SE) that extends over a broad range of microwave frequencies.
   - Emphasis will be on thin and light weight EMI shielding materials for application in combat/stealth aviation technology.
   - Use of cost-effective natural materials to reduce overall cost.
   - Durability, corrosion resistant and thermal stability are additional issues to be addressed.

3. **Implementing organization**: Tezpur University, Assam

4. **DIT sanction No. and Date**: Administrative Approval No. 1(1)/2010/M&C dated 24.05.2010

5. **(a) Total budget outlay, revised if any:** Rs. (79.49+7.49= 86.98) lacs
   (b) Duration of project: May 2010 – November 2013
   (c) **Date of completion and reasons for delay, if any:** 23rd November, 2013

6. **Total Funds spent (Details at Annexure I):** Rs. 86.98 lacs

7. **Details of equipment/assets:** Refer Table1

8. **Details of manpower (Details at per Annexure II):** Refer Annexure IV

9. **Details of year-wise audited statement of accounts and utilization certificates (As per CFR 19/UC):** Refer page (15-22)

PART II

1. Project work and Achievements

a) Executive Summary

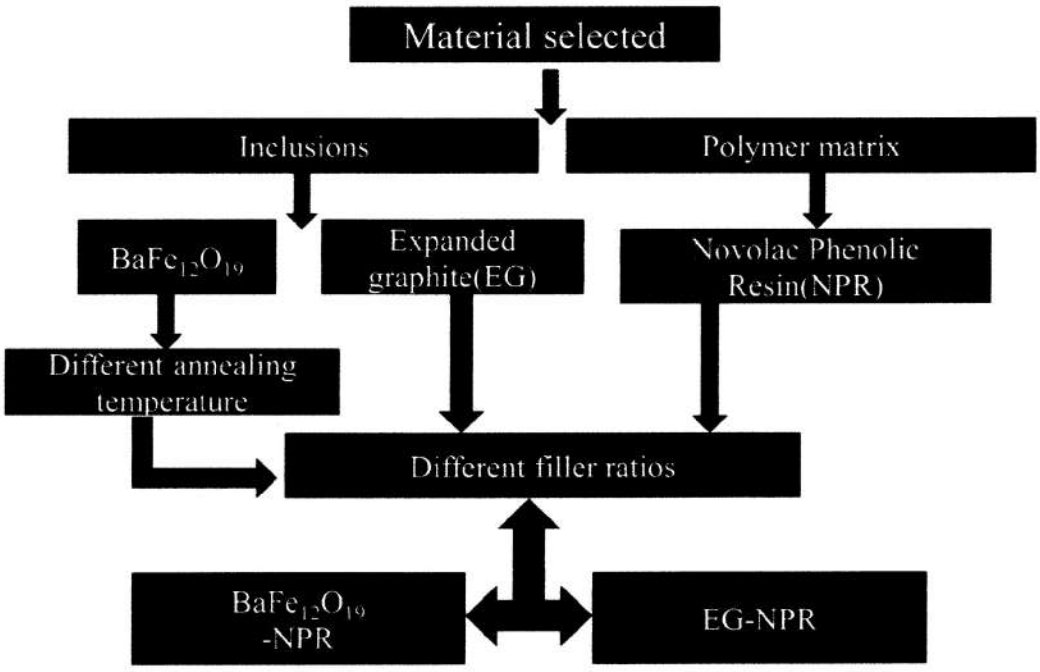
Scope of the project:

The project was aimed to develop light weight, broadband electromagnetic interference shielding material in the frequency ranges of 8.2-12.4GHz. The developed product will have use in mobile communications system, anechoic chamber, handheld devices, transmitting and receiving antennas, protecting circuits, strategic installations, human body from external radiation etc

Project output:

- Single and multi layer dielectric microwave absorber composed of expanded graphite –Phenolic resin composite showing -10dB microwave absorption bandwidth>3GHz in the X-band.
- Single layer absorber composed of $BaFe_{12}O_{19}$/NPR composites of 50 wt. % gives -20dB absorption bandwidth of 3.76GHz in the X-band

Flow chart of processes/product development



Product name with composition	Product specifications					
	Layer design	Thickness (mm)	RLm at frequency	Absorption bandwidth		S_{21}(dB) over the X-band
				-10dB	-25dB	
5 wt. % EG-NPR	Single	4	-43dB at 12.4GHz	12.4-11.5= 0.9GHz	N/A	-2.7
7 wt. % EG-NPR	Single	4	-19dB at 12.0GHz	12.4-11.0= 1.4GHz	N/A	-2.8
8 wt. % EG-NPR	Single	4	-16dB at 10.39GHz	10.4-9.8= 0.6GHz	N/A	-4.0
10 wt. % EG-NPR	Single	4	-12dB at 9.72GHz	10.23-9.39= 0.84GHz	N/A	-5.5
20 wt. % EG-NPR	Single	4	-2.0dB over X-band	N/A	N/A	-31.0
30 wt. % EG-NPR	Single	4	-1.4dB over X-band	N/A	N/A	-38.0
40 wt. % EG-NPR	Single	4	-1.0dB over X-band	N/A	N/A	-41.0
50 wt. % EG-NPR	Single	4	-1.0dB over X-band	N/A	N/A	-44.7
(10-8) wt. % EG-NPR	Double	3.2	-46dB at 11.09GHz	8.2-12.4= 4.2GHz	12.3-9.84= 2.46GHz	N/A
(7-10-8) wt. % EG-NPR	Triple	3.1	-44dB at 10.99GHz	8.2-12.4= 4.2GHz	12.0-9.95= 2.05GHz	N/A

Product/process specifications: Table 1

Other output: Single layer circularly perforated absorber design on EG-NPR composites showing -10dB absorption bandwidth ~3GHz in the X-band. The absorber is relatively light weight as most of the substrate is removed.

Status of technology: The developed technology is in research scale only, however showing promising microwave absorption for commercialization in the frequency range of 8.2-12.4GHz.

Justification for the project: X-band frequency region finds wide use in wireless communication purposes like military communication satellites (7.9 to 8.4 GHz for uplink & 7.25 to 7.75 GHz for downlink), precision approach radar (PAR) (9.0-9.2 GHz), terrestrial communication and networking (10.15 to 10.7 GHz), motion detectors (10.525 GHz), traffic light crossing detectors (10.4 GHz), weather radars (9.3-9.5 GHz) and also in medical sciences etc. The prolific usage of X-band frequency spectrum creates interference among themselves as well as for devices working in other operating frequency bands. To reduce the X-band interference, efficient microwave absorber is required.

b) Project targets and achievements

Project targets	Achievements
a) Procurement and setting of equipments.	Procured Agilent E8362C VNA along with accessories and 85071E material measurement software
b) Calibration of instruments using standard samples.	Calibrated and tested with microwave absorber samples provided by Emerson and Cuming, Hyderabad.
c) Synthesis of filler particles & characterization.	Expanded graphite (EG) and $BaFe_{12}O_{19}$ as fillers are synthesized and characterized for confirmation.
d) Thermal and magnetic characterization of the magneto-dielectric nano particles.	Thermo-gravimetric analysis shows EG composites have thermal stability up to 250 °C. Magnetic characterization could not be performed due to unavailability of VSM.
e) Development of new magneto-dielectric polymer composite material.	Composites of expanded graphite-novolac phenolic resin (EG-NPR) and $BaFe_{12}O_{19}$-NPR are developed as absorbing materials.
f) Determination of complex permittivity and complex permeability.	Complex permittivity and permeability of the developed composites are determined using 85071E material measurement software in the X-band.
g) Development of test module for measuring reflection loss of the materials.	Free space measurement setup is developed employing a pair of spot focusing horn lens antennas.
h) Prototype studies to determine SE.	Single layer 50 wt. % EG-NPR composites of 4mm thickness show S_{21} (dB) values of -45dB.
i) Optimization of the reinforcers properties to improve absorption parameters.	Variation of filler percentages in the composites to change the shielding mechanism from reflective to absorptive. (5-10) wt. % EG-NPR composites shows absorptive behavior, whereas (20-50) wt. % EG-NPR composites are highly reflecting in nature. $BaFe_{12}O_{19}$/NPR composites of 50 wt. % gives -20dB absorption bandwidth of 3.76GHz.
j) To develop tailored made conformal Shielding materials by manipulating the geometry and online testing.	Double and triple layer structure design is optimized with EG-NPR composites to get a broadband absorption. Double layered (10-8) wt. % EG-NPR combination of thickness 3.2mm gives a -25dB absorption bandwidth of (12.3-9.84= 2.46GHz). Similarly, triple layered (7-10-8) wt. % EG-NPR of thickness 3.1mm shows -25dB absorption bandwidth of (12.0-9.95= 2.05GHz).
k) Analysis of results.	Completed. Please refer the progress report

2. Additional Information

i) **Details of patents registered, if any**: Not applied

ii) **Technological spin offs,**

- **seeding of a major activity and how the project has helped in enhancing the technological base/capabilities in the country:** N/A

iii) **Future areas for work**: Adaptive microwave absorber, high and low temperature sustains of microwave absorber for aerial applications, designing of different RCS absorber using the synthesized light weight EG-NPR and barium ferrite-NPR composites.

ADDITIONAL INFORMATION REQUIRED FOR COMMERCIAL EXPLOITATION

1. **Product/process developed:** Two type of microwave absorber are developed viz. dielectric microwave absorber (DMA) and magnetic microwave absorber (MMA).

DMA is composed of expanded graphite (EG)-novolac phenolic resin (NRP) composite. EG is synthesized from natural graphite flakes as follows: EG flakes are mixed with saturated acid consisting of sulfuric acid and concentrated nitric acid in a volume ratio 3:1 for 12 hours to form graphite intercalated compound (GIC).The chemically treated flakes are then thoroughly rinsed with water until the pH level of the solution reaches 7 and dried at 60°C, in vacuum oven, for 5 hrs. GICs are then suddenly exposed to high temperature in a muffle furnace maintained at temperature 800-900°C to form EG. This synthesized EG is mixed with NPR in different weight ratios to obtain desired EG-NPR combination as microwave absorbing materials.

MMA is composed of nanosize $BaFe_{12}O_{19}$ mixed with NPR in different weight ratios. Initially, $BaFe_{12}O_{19}$ is synthesized by co-precipitation method using barium nitrate, ferric nitrate and sodium hydroxide as raw materials. Solution of $Ba(NO_3)_2$ and $Fe(NO_3)_3$ in distilled water is mixed together and heated at 70°C with continuous magnetic stirring condition for one and half hour. 4M solution of NaOH is slowly added to the salt solution drop wise. A specified amount of oleic acid is added to the solution as a surfactant and coating material. The product is cooled at room temperature. To get free particles from sodium and chlorine compounds, the precipitate is than washed twice by distilled water and than by ethanol to remove the access surfactant from the solution. Finally it is dried and crumbled to form the precursor powder. This precursor powder is annealed at different annealing temperatures (700°C, 800°C and 900°C) for two hours.

2. **Specific use of the product/process:** EG-NPR composites of (5 to 10) wt.% can be used as absorbing material in the X-band and 20 to 50 wt% EG-NPR composites as reflecting shielding material giving S21~-43dB over the X-band. $BaFe_{12}O_{19}$/NPR composites of 50 wt. % give -20dB absorption bandwidth of 3.76GHz in the X-band.

3. **Background information on the project (existing technology and alternatives in India):** The recent revision in international standard (CISPR 16-1-4) of anechoic chamber testing up to 18GHz than the existing 30MHz to 1 GHz has compelled to modify the absorbing materials to meet the new regulations. In particular, the X-band spectrum is mostly used in modern wireless communication system as discussed above and consequently electromagnetic interference occurs. EMI shielding materials finds versatile applications in reducing electronic pollution, camouflaging strategic defense system etc. By the very nature of the technology which is critical for defense applications no nation will share this technology with other nations. Indigenous development therefore is unavoidable. Regarding the existing technology most of the organization imports the absorber sheets from company like Emerson and Cumming, Belgium etc.

4. **Status of product/area in India:**

The data of exact status of the microwave absorber being produced commercially in India is not well documented; however there are some research groups who are working in isolation [1-3]. The product developed in this project work has the potential to be developed in mass scale because of its ease in processing, low cost, light weight and good broadband performance.

References:

[1] Goyal, R.K., Jagadale, P.A., and Mulik, U.P. Thermal, mechanical, and dielectric properties of polystyrene/expanded graphite nanocomposites, *J. Appl. Polym. Sci.* 111, 2071-2077, 2009.

[2] S.M Abbas, A K Dixit, R Chatterjee, T C Geol. "preparation of nanosize polyaniline and its utilization for microwave absorbers", *J Nanosci Nanotechnol*,vol. 7, No. 6, pp.2129-33, 2007

[3] K.H. Prema et al. "Permittivity Characteristics in the X- and S-Band Frequencies of Microwave Absorbers Based on Rubber Ferrite Composites", *Journal of Elastomers and Plastics*, Vol. 40, No. 4, pp 331-346 (2008)

5. **International status of technology/products/process**

Most of the literature survey does not give details of the development process as this being a strategic research area. We acquired samples from Emerson and Cuming, Belgium from their agents in Hyderabad, and compared our products with them. The details are given below:

6. **Suggested end users:** DRDO, ISRO, Electronic and Communication product related Industries, Navigation Industries.

7. **Similar products/process in India:** Some private companies do advertise for EMI shielding materials, but the standardization of product is not known. It is not clear whether they are importing it or developing themselves. As mentioned in 4. Some research paper from NPL, IIT Delhi, College of Engineering, Shivaji Nagar, Pune regarding different absorbers were found.

8. **Whether the product is for import substation:** Till date the product was developed more for R & D with the possibility of mass production. In second phase mass production of the developed absorbers will be carried out, with provision of low and high temperature testing and custom designing to cater the needs of National requirements after which development of import will be considered.

9. **Name & address of prospective entrepreneurs:** We are looking for the entrepreneurs. Communication in this regard has been made with an entrepreneur group from Guwahati and Vidyut Yantra, Modi Nagar

10. **Safety and pollution aspects**

Most of the ingredients of the developed EG-NPR and $BaFe_{12}O_{19}$are non-toxic. However, some amount of NO_2 and SO_2 is produced while intercalating graphite flakes which are within toxic permissible limit (NO_2~25 ppm and SO_2 ~ 5.0 ppm) of the government [4, 5]. Care has to be taken for mass production. The absorber developed is corrosion resistant and inert to water and common reagents at room temperatures, but bio-degradability have to be studied in long run.

Reference: [4] http://www.atsdr.cdc.gov/mmg/mmg.asp?id=394&tid=69

[5] http://inspectapedia.com/sickhouse/gashaz.htm

11. **Suggestion regarding terms for transfer of technology:** After contacting the end-users and intermediate manufacturers the terms of transfer of technology will be accordingly developed in consultation with DIETY.

12. **Preparation of achievability report**

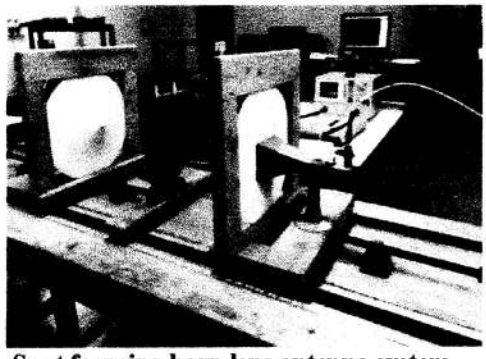
The EMI shielding product specifications are given in Table 1. For transferring technology has to be developed case to case depending upon the end-user.

ANNEXURES

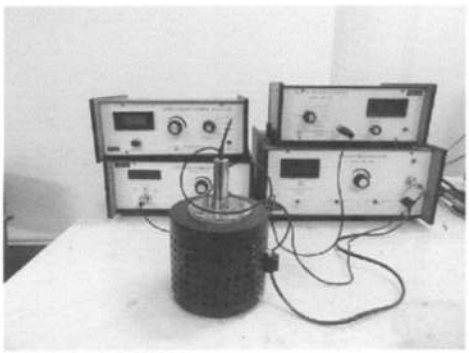
I. Minutes of the Steering committee meeting: Attached as minutes

II. Equipments photographs

Agilent E8362C Vector Network Analyzer



Spot focusing horn lens antenna system



Four probe set up

Vertical muffle Furnace

Muffle Furnace

Toledo Electronic balance

III. List of publications

1. J. P. Gogoi, N. S. Bhattacharyya, "EMI shielding characteristics of expanded graphite/novolac phenolic resin composite for applications in wireless communication", IEEE proc. ICDeCOM-2011, BIT Mesra, *doi: 10.1109/ICDECOM.2011.5738512*
2. J. P. Gogoi and N. S. Bhattacharyya, "Microwave Characterization of Expanded Graphite/Phenolic Resin Composite for Strategic Applications" *Progress In Electromagnetics Symposium-2012 Kaula Lumpur, Malaysia*
3. J. P. Gogoi and N. S. Bhattacharyya, "Expanded Graphite/Novolac Phenolic Resin Composite as Single Layer Electromagnetic Wave Absorber for X-Band Applications," *Proc. of SPIE Vol. 8760 876005-1, 2013*
4. S. Ozah and N. S. Bhattacharyya, "Nanosized barium hexaferrite in Novolac Phenolic Resin as microwave absorber for X-band application" *Journal of Magnetism and magnetic materials* 342, pp. 92-99, 2013
5. J. P. Gogoi, D. Sarmah, S. Ozah and N. S. Bhattacharyya, "Design and fabrication of conductor backed perforated single layer microwave absorber" *Under review (Electronic letters)*
6. J. P. Gogoi and N. S. Bhattacharyya, "Design Optimization of Expanded Graphite - Phenolic Resin Composites based Double Layer Microwave Absorber for X-band Applications" *IEEE transactions on electromagnetic compatibility (under review)*

IV. Chemical characterization: FTIR studies confirm the interaction between EG and NPR is physical in nature.

V. Cost calculation

Cost calculation for absorber samples	
Raw materials	**Cost Price/kg (Rs.)**
Natural graphite (NG)flakes	100
Novolac phenolic resin	200
Barium nitrate	516
Ferrite nitrate	716
Sodium hydroxide	480
oleic acid	426
Acetone	590
Sulphuric acid	464
Nitric acid	510
*Expanded graphite (EG)flakes synthesized from NG	2000
Synthesized Barium ferrite cost	5000
Absorber of dimension of 15.2 cm x 15.2cm x 0.4cm	
5wt. %EG-NPR	***1026.762***
7wt. %EG-NPR	***1030.02***
8wt. %EG-NPR	***1031.6***
10wt. %EG-NPR	***1035***
20wt. %EG-NPR	***1051.74***
30wt. %EG-NPR	***1068.37***
40wt. %EG-NPR	***1084.92***
50wt. %EG-NPR	***1101.64***
Barium ferrite-NPR	***1240.24***

*Volume of 1 kg EG flakes are 200 times greater than volume of 1 kg of NG and a percolation limit of EG is small in comparison to NG.

GFR 19-A
[See Rule 212(1)]

Form of Utilization Certificate
(Period: From-May, 2010-to November, 2013)

Sl. No.	Letter No and Date	Amount
1	Administrative Approval No. 1(1)/2010/M&C dated 24.05.2010, 26.06.2012 & 25.07.2012	**Rs. 8697240.00**
2.	Interest Earned	**Rs. 279251.00**
Total		**Rs. 8976491**

Certified that out of **Rs. 8697240.00 (Eighty six lacs ninety seven thousand two hundred and forty only)** of grants-in-aid sanctioned during the year 2010-2013 in favour of Tezpur University, Assam.
Under this Ministry/Department letter No. given in the margin and **Rs. 8697240.00 (Eighty six lacs ninety seven thousand two hundred and forty only)** on account of total sanctioned amount and **Rs. 279251.00 (Rs. Two lacs seventy nine thousand two hundred and fifty one only)** earned as interest amount, a sum of **Rs 8886372.00 (Rs. Eighty eight lacs eighty six thousand three hundred and seventy two only)** has been utilized for the purpose of project under Electronics Material Development programme entitled "Synthesis and Development of EMI Shielding Material using Magneto-Dielectric Nanoparticles" for which it was sanctioned and **Rs. 90119.00 (Rs. Ninety thousand one hundred and nineteen only)** remaining unutilized at the end of the project.

2. Certified that I have satisfied myself that the conditions on which the grants-in-aid was sanctioned have been duly fulfilled/are being fulfilled and that I have exercised the following checks to see that the money was actually utilized for the purpose for which it was sanctioned.

Kinds of checks exercised :

1. Stock checking for procured equipments and other expenditure
2. Physically verifying the equipments installed
3. All the vouchers related to expenditure in the project checked and deposited.

(Nidhi S. Bhattacharyya)
Principal Investigator

10.4.14
(B. Pathak)
Finance Officer
Tezpur University
Finance Officer
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(Dr B. Das)
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Principal Investigator
DIT Project
Title "Synthesis...nanoparticles"
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SD Reg No. DoRD/Phy.NSB/2040

For SURAJIT CHAKRABORTY & CO.
CHARTERED ACCOUNTANTS
02.02.2016
CA. SURAJIT CHAKRABORTY
(Proprietor)
Membership No.- 305054

Statement of Expenditure

DIT PROJECT-"Synthesis and development of broadband EMI shielding materials using magneto-dielectric nanoparticles"
PI- Prof. N. S. Bhattacharyya

Sl No	Head	Fund Received (Rs)		Total Received amount (Rs)	Spent amount (Rs)					Total spent (Rs)	Unspent (Rs)
		2010-2011	2012-2013	2010-2013	2010-2011	2011-2012	2012- 2013	2013-2014	2014-2015		
1	Equipment	5429000	699000	**6128000**	-	5140444	101303	--	1090000	**6331747**	**-203747**
2	Manpower	312000	611000	**923000**	228967	234000	390000	101600		**954567**	**-31567**
3	Consumables	94000	192240	**286240**	-	94000	96270	--	47054	**237324**	**48916**
4	Contingency	100000	200000	**300000**	68380	43907	85691	67676	34346	**300000**	**0**
5	Travel	50000	150000	**200000**	19384	25150	114714	43486		**202734**	**-2734**
6	Overhead	-	860000	**860000**	-	--	--	537500	322500	**860000**	**0**
7	Interest			**279251**							**279251**
	Total	5985000	2712240	**8976491**	316731	5537501	787978	750262	1493900	**8886372**	**90119**

(Nidhi S. Bhattacharyya)
Principal Investigator

Principal Investigator
DIT Project
Title "Synthesis...nanoparticles"
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R&D Reg No. DoRD/Phy.NSB/2040

10.11.14

(B. Pathak)
Finance Officer
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(Dr B. Das)
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Tezpur University

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For SURAJIT CHAKRABORTY & CO.
CHARTERED ACCOUNTANTS

02.02.2016

CA. SURAJIT CHAKRABORTY
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