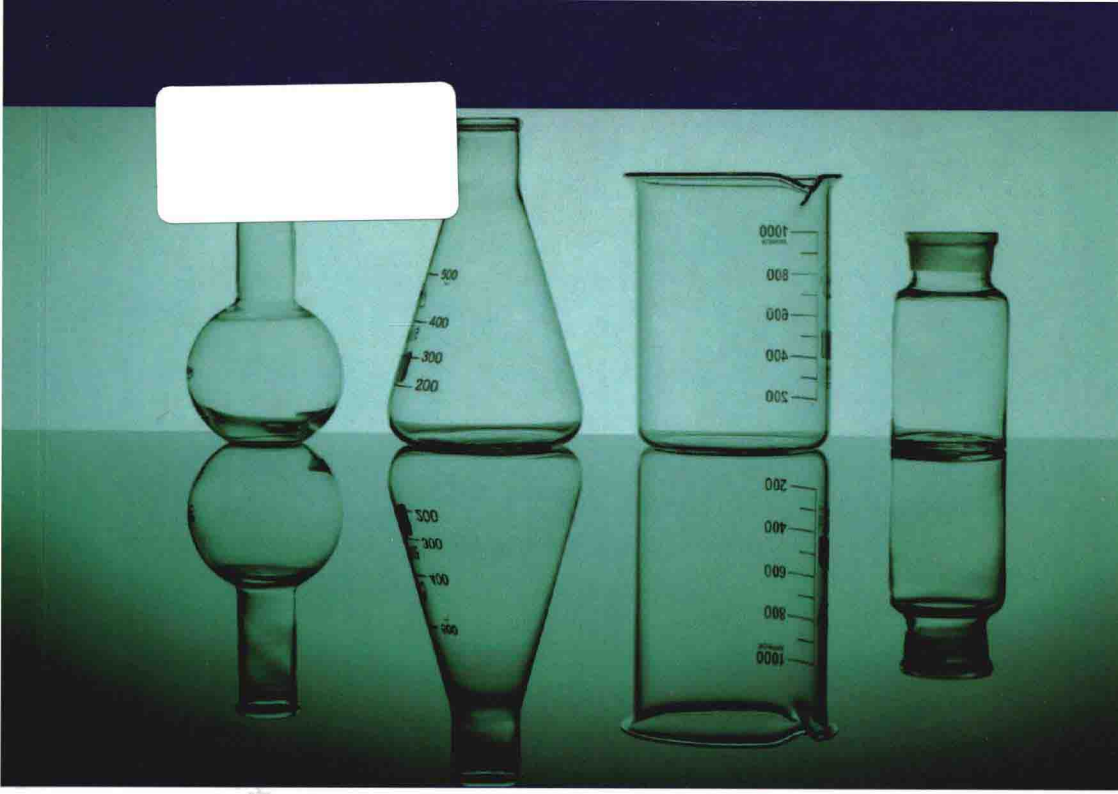

K. Venkateswara Rao
C.S. Sunandana

Combustion & Chemical Bath Deposition of Nanopowders and Thin Films

MgO, NiO, Mg1-xMxO(M=Ni, Co; 0≤x≤1)and Co3O4 Nanopowders and Thin Films

LAP LAMBERT Academic Publishing

K. Venkateswara Rao
C.S. Sunandana

Combustion & Chemical Bath Deposition of Nanopowders and Thin Films

MgO, NiO, Mg1-xMxO(M=Ni, Co; 0≤x≤1)and Co3O4 Nanopowders and Thin Films

LAP LAMBERT Academic Publishing

Impressum / Imprint
Bibliografische Information der Deutschen Nationalbibliothek: Die Deutsche Nationalbibliothek verzeichnet diese Publikation in der Deutschen Nationalbibliografie; detaillierte bibliografische Daten sind im Internet über http://dnb.d-nb.de abrufbar.

Bibliographic information published by the Deutsche Nationalbibliothek: The Deutsche Nationalbibliothek lists this publication in the Deutsche Nationalbibliografie; detailed bibliographic data are available in the Internet at http://dnb.d-nb.de.

Coverbild / Cover image: www.ingimage.com

Verlag / Publisher:
LAP LAMBERT Academic Publishing
ist ein Imprint der / is a trademark of
AV Akademikerverlag GmbH & Co. KG
Heinrich-Böcking-Str. 6-8, 66121 Saarbrücken, Deutschland / Germany
Email: info@lap-publishing.com

Herstellung: siehe letzte Seite /
Printed at: see last page
ISBN: 978-3-659-35939-2

Zugl. / Approved by: Hyderabad, University of Hyderabad, Ph.D, 2008

K. Venkateswara Rao
C.S. Sunandana

Combustion & Chemical Bath Deposition of Nanopowders and Thin Films

Combustion & Chemical Bath Deposition of Nanopowders and Thin Films

MgO, NiO, $Mg_{1-x}M_xO$(M=Ni, Co; $0 \leq x \leq 1$)and Co_3O_4 Nanopowders and Thin Films.

K.Venkateswara Rao
Associate Professor & Head,
Centre for Nano Science & Technology, Institute of Science & Technology,
JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY HYDERABAD
Hyderabad, Andhra Pradesh, India.

&

C.S.Sunandana
Professor , School of Physics,
UNIVERSITY OF HYDERABAD
Hyderabad , Andhra Pradesh, India.

DEDICTATION

This is dedicated to

My parents

Kalagadda. Lutherpaul and Lurdhamma

as a mark of honor, for their love and special efforts that they devoted towards my studies

&

My wife

Smt K.Rama Laxmi

ACKNOWLEDGEMENTS

With great pleasure and much indebtedness, I express my heartfelt and sincere gratitude to my research supervisor **Prof.C.S. Sunandana**, who has been a constant source of inspiration, encouragement and guidance to me throughout my research. He is simple, committed and a fair minded humanist which we find rarely in the society. These qualities of him motivated and helped me for completion of work smoothly.

I thank Dr. Ghanashyam Krishna, member of the doctoral committee for his valuable and critical comments.

I am very much thankful to all my teachers who thought at ANDHRA LOYOLA COLLEGE for graduation and at HYDERABAD UNIVERSITY, School of Physics for post graduation.

I would like to extend my heart felt thanks to all the Central Instrumentation Laboratory staff members especially

Mr.C.S.Murthy, Dr.Manjunath, Mr.Suresh and in school of physics who helped me directly or indirectly during my experimental works.

The academic enjoyment imported during the seminar sessions with Professor V.Seshubai, deserves a special mention in my carrier, all the DEAN and faculty members of the School of Physics are thankfully acknowledged for providing constant support during my research period.

I thank Professor **K. Rajagopal** Vice-Chancellor, JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY for encouraging me and granting permission for doing the research at HCU, who always supports research.

I am also thankful to the Rector Dr.B. C. Jinaga and the Registrar Dr. K.Lal Kishore and the Director of evaluation DR. K. Vijaya Kumar Reddy, Earlier Principal Dr.G.Tulasiram Das and present Principal Dr.A.K.Asthana and Vice-Principal Dr.A.Damodaram for their continues encouragement and support .

I am very much thankful to Director of admission Dr. M. Chandra Shekar and Dr. P. Appalanaidu, Coordinator, DAP for their moral support and academic discussions.

I am thankful to the Head of the department physics JNTUCEH Dr.S.Chandralingam and my colleagues Dr.Y.Aparna, Mr.S.Venkaateswara Rao and Dr.P.Dakshina Murthy(JNTUCEK) for their cooperation and support.

I thank my PG classmate G.Baskar who helped me when ever I am need.

I shall be ever thankful to my Father-in Law Dr. Chinta. Venkateswara Rao Controller of Examination, HCU with out whose future vision about my carrier, my mind would'nt have gone towards the research for a Ph.D degree.

I am thankful to my well wishers and senior Colleagues Father Antony, Dr.GSR and colleagues Mr.KRR, Mr.DVS at Andhra Loyola College. I am also thankful to all my colleagues at VKR College

I thank my senior lab mate BHARTHI MOHAN especially for his continuous help and also my other lab mates.

I will be falling my responsibility if I don't acknowledge the remarkable support extended by my better half Smt. Rama laxmi for her support and patience and taking pains and family burden and spending most of the time without my presence. And my sons Master. Lalith Chandra and Master. Lalith Surya who made us to be happy forgetting all the tensions and also for allowing me to concentrate on my work.

My Mother-in Law Smt.Ch. Susila who took care of my twin children with greatest love and affection and also bearing all the pains and sufferings in seeing their welfare, with out her help my children might not be in happy moods and healthy which helped me to concentrate on my research with out any mind bothering towards my children care.

I thank my brother his wife and daughter K.Santha Babu, Jyothirmayi, papa, and K.Bikshalu, K.Jhansi (sister) who helped me

always. I thank my Brother-in Law Ch.Sandeep and sister-in laws Sunitha and Sirisha and Vijay for their support.

I owe all my success to the almighty and my mother and father who took lot of pains to brought up and whose endless sacrifice made my journey of life a meaningful one.

CONTENTS

DEDICATION

ACKNOWLEDGEMENTS

CHAPTERS	**Page No**
1. Introduction	1-56
2. Experimental techniques	57-123
3. Transition metal oxides (NiO, Co_3O_4) and MgO Structural, micro structural, optical and EPR studies	124-182
4. Diluted anti-ferromagnetic solid solutions $Mg_{1-x}M_xO$ (M=Ni, Co) ($0 \leq x \leq 1$) by chemical combustion: structural, Micro structural and EPR studies	183-239
5. Thin films of MgO, NiO, Co_3O_4 by chemical deposition Method: structural, micro structural and optical studies	240-263
6. Summary and Scope for Future work	264-274

CHAPTER-1

GENERAL THEME:

The general theme of this thesis is broadly divided into three parts

I. Synthesis of transition oxide nano materials of (NiO, Co_3O_4) and MgO by chemical combustion method for different fuel to oxidizer ratio ($0<\Psi<2$) in the steps of 0.25) and studies of their structural, electron paramagnetic resonance, optical and microscopic properties.

II. Synthesis of diluted antiferromagnetic systems $Mg_{1-x}M_xO$ (M=Ni, Co) ($0<x<1$) by chemical combustion method and studies of their structural, electron paramagnetic resonance and microscopic properties.

III. Fabrication of transition oxide nano crystalline thin films by chemical bath deposition and studies of their structural, optical and microscopic properties

Various experimental probes have been utilized for the following characterization studies such as,

(1) Structural: X-ray diffraction

(2) Microscopic: Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM)

(3) Susceptibility (magnetic studies): Electron Paramagnetic Resonance (EPR)

(4) BET surface area: Specific surface area

(5) Optical: UV-Visible spectrophotometer and FTIR

The objectives of the present investigation are

(1) Study of the formation mechanism of combustion synthesized MgO, NiO, Co_3O_4 nano powders for different fuel to oxidizer ratios (Ψ=0.25,0.5,0.75,1,1.25,1.5,1.75,2) and studies of their crystal structure and micro structure analysis.

(2) The optical studies allowed us to determine the band gap of the NiO, Co_3O_4 nanoparticles, which is changing with Ψ. FT-IR peaks confirms the functional groups

(3) EPR data allowed to us to calculate the susceptibility of the NiO, Co_3O_4 nanoparticles.

(4) Study of the formation of combustion synthesized $Mg_{1-x}M_xO$ (M=Ni, Co) (0<x<1) solid solution and their crystal structure and micro structure analysis. EPR susceptibilities of solid solution $Mg_{1-x}M_xO$ is determined at room temperature. Variable temperature EPR is allowed to check the Curie-Weiss law for solid solution and the linearity of $C_M(x)$ and $\Theta(x)$ with concentration of nickel (cobalt) has ruled out chemical clustering in the samples.

(5) Study of fabrication of nano crystalline thin films of MgO NiO, Co_3O_4 by chemical deposition method and studies of their crystal structure and micro structure analysis.

1. INTRODUCTION:

Ultra fine microstructures having average phase of grain size on the order of a nanometer (10^{-9} m) are classified as nanostructured materials [1, 2]. Currently, in a wider meaning of the term, any material that contains grains or clusters below 100 nm, or layers or filaments of that dimensions, can be considered to be nanostructured [3]. The interest in these materials has been stimulated by the fact