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Mechanical Properties and Performance of Engineering Ceramics and Composites III

Ceramic Engineering and Science Proceedings

Volume 28, Issue 2

Edited by
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Jonathan Salem
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The American Ceramic Society

Mechanical Properties and Performance of Engineering Ceramics and Composites III

A Collection of Papers Presented at the 31st International Conference on Advanced Ceramics and Composites January 21–26, 2007 Daytona Beach, Florida

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WILEY-INTERSCIENCE
A John Wiley & Sons, Inc., Publication

Published by John Wiley & Sons, Inc., Hoboken, New Jersey.
Published simultaneously in Canada.

Library of Congress Cataloging-in-Publication Data is available.

ISBN 978-0-470-19633-5

Printed in the United States of America.

10 9 8 7 6 5 4 3 2 1

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Preface

This volume contains papers presented at the *Symposium on Processing, Properties and Performance of Engineering Ceramics and Composites* during the 31st International Conference & Exposition on Advanced Ceramics & Composites held on January 21-26, 2007 at Daytona Beach, Florida. These papers from researchers in 12 different countries, address core fundamentals as well as timely topics on the science and technology of ceramics and ceramic composites in the best tradition of the International Conference & Exposition on Advanced Ceramics & Composites.

The papers in this volume are organized in the following sections:

- Processing of Ceramics and Composites
- Silicon-Based Ceramics
- Properties of Monolithic Ceramics
- Fibers and Interfaces
- Fiber-Reinforced Ceramic Matrix Composites
- Particulate-Reinforced and Laminated Ceramics
- Environmental Effects
- NDE, Test Methods and Modeling
- Fracture
- Joining & Brazing

The International Conference & Exposition on Advanced Ceramics & Composites is truly the premier international forum to present information on emerging ceramic technologies and on the processing, properties, behavior and application of structural ceramics, functional ceramics and ceramic composites. There are many reasons for the great success of this meeting, including its venue (Daytona Beach for the first time after 30 meetings in Cocoa Beach), its size (the collision frequency and mean-free path is perfect to foster interactions among the attendees), its schedule (there are not many places that beat Florida in January), and the dedication of many volunteers and the staff of The American Ceramic Society. In particular, we thank the attendees, the authors, the session chairs and session organizers, as well as those who helped us review the manuscripts contained in this volume.

EDGAR LARA-CURZIO
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Introduction

2007 represented another year of growth for the International Conference on Advanced Ceramics and Composites, held in Daytona Beach, Florida on January 21-26, 2007 and organized by the Engineering Ceramics Division (ECD) in conjunction with the Electronics Division (ED) of The American Ceramic Society (ACerS). This continued growth clearly demonstrates the meetings leadership role as a forum for dissemination and collaboration regarding ceramic materials. 2007 was also the first year that the meeting venue changed from Cocoa Beach, where it was originally held in 1977, to Daytona Beach so that more attendees and exhibitors could be accommodated. Although the thought of changing the venue created considerable angst for many regular attendees, the change was a great success with 1252 attendees from 42 countries. The leadership role in the venue change was played by Edgar Lara-Curzio and the ECD's Executive Committee, and the membership is indebted for their effort in establishing an excellent venue.

The 31st International Conference on Advanced Ceramics and Composites meeting hosted 740 presentations on topics ranging from ceramic nanomaterials to structural reliability of ceramic components, demonstrating the linkage between materials science developments at the atomic level and macro level structural applications. The conference was organized into the following symposia and focused sessions:

- Processing, Properties and Performance of Engineering Ceramics and Composites
- Advanced Ceramic Coatings for Structural, Environmental and Functional Applications
- Solid Oxide Fuel Cells (SOFC): Materials, Science and Technology
- Ceramic Armor
- Bioceramics and Biocomposites
- Thermoelectric Materials for Power Conversion Applications
- Nanostructured Materials and Nanotechnology: Development and Applications
- Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT)
- Porous Ceramics: Novel Developments and Applications

- Advanced Dielectric, Piezoelectric and Ferroelectric Materials
- Transparent Electronic Ceramics
- Electroceramic Materials for Sensors
- Geopolymers

The papers that were submitted and accepted from the meeting after a peer review process were organized into 8 issues of the 2007 Ceramic Engineering & Science Proceedings (CESP); Volume 28, Issues 2-9, 2007 as outlined below:

- Mechanical Properties and Performance of Engineering Ceramics and Composites III, CESP Volume 28, Issue 2
- Advanced Ceramic Coatings and Interfaces II, CESP, Volume 28, Issue 3
- Advances in Solid Oxide Fuel Cells III, CESP, Volume 28, Issue 4
- Advances in Ceramic Armor III, CESP, Volume 28, Issue 5
- Nanostructured Materials and Nanotechnology, CESP, Volume 28, Issue 6
- Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials, CESP, Volume 28, Issue 7
- Advances in Electronic Ceramics, CESP, Volume 28, Issue 8
- Developments in Porous, Biological and Geopolymer Ceramics, CESP, Volume 28, Issue 9

The organization of the Daytona Beach meeting and the publication of these proceedings were possible thanks to the professional staff of The American Ceramic Society and the tireless dedication of many Engineering Ceramics Division and Electronics Division members. We would especially like to express our sincere thanks to the symposia organizers, session chairs, presenters and conference attendees, for their efforts and enthusiastic participation in the vibrant and cutting-edge conference.

ACerS and the ECD invite you to attend the 32nd International Conference on Advanced Ceramics and Composites (http://www.ceramics.org/meetings/daytona2008) January 27–February 1, 2008 in Daytona Beach, Florida.

JONATHAN SALEM AND DONGMING ZHU, Volume Editors
NASA Glenn Research Center
Cleveland, Ohio

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Processing

SYNTHESIS AND CHARACTERIZATION OF $Ba_3Co_2Fe_{24}O_{41}$ AND $Ba_3Co_{0.9}Cu_{1.1}Fe_{24}O_{41}$ NANOPOWDERS AND THEIR APPLICATION AS RADAR ABSORBING MATERIALS

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ABSTRACT

There has been a growing and widespread interest in the development of radar absorbing materials (RAM) to reduce the radar signatures of navy platforms. Z-type barium hexaferrite is one of the most complex compounds in the family of hexagonal ferrites that due to its good magnetic properties, it is a promising candidate to be used as RAM.

In this work, the nanosized Z-type barium hexaferrite powders ($Ba_3Co_2Fe_{24}O_{41}$ and $Ba_3Co_{0.9}Cu_{1.1}Fe_{24}O_{41}$) were synthesized at 950 °C by the citrate sol-gel process to be used as RAM in polychloroprene (CR) matrices. X-ray diffraction and X-ray fluorescence (XRD and XRF, respectively) were used to characterize these materials. Magnetic properties of the Z-type barium hexaferrites were also evaluated by using the vibrating sample magnetometer (VSM). The Cu^{2+} ions were incorporated into the structure of $Ba_3Co_2Fe_{24}O_{41}$ and consequently, low temperature sintering and good magnetic properties were achieved. The microwave reflectivity levels (dB) of the Z-type barium hexaferrite:polychloroprene composites were determined for the frequency range 8.0 - 16.0 GHz. The permittivity (e) and permeability (m) values were measured by using the transmission/reflection (T/R) method in a waveguide medium. The nanocomposite 80:20 of $Ba_3Co_{0.9}Cu_{1.1}Fe_{24}O_{41}$:CR, 3.0 mm thick, showed the best performance as RAM for the X-band, with a microwave absorption of 99.50 % (reflectivity of - 22.5 dB) in 9.5 GHz, which can be attributed to the increase in the magnetic properties due to the Cu addition.

INTRODUCTION

Radar absorbing materials (RAM) play an important role on the stealth technology, which corresponds to the invisibility of military platforms to the different systems of detection (radar, acoustic, infrared, etc), by suppressing microwaves reflected from metallic structures and so, reducing the radar signatures of the targets.

Recently, a great deal of attention is devoted to hexagonal ferrites as microwave materials for the 1-100 GHz band and a wide range of chemical methods have been used to obtain ultrafine particles. All of these methods require a low-temperature process in order to control the particles' size, as a first synthesis stage. In an attempt either to promote the formation of Z-type

ferrite and to improve the magnetic properties, we report the synthesis of these nanostructured materials by the sol–gel–citrate precursor method [1, 2].As magnetic materials, the barium hexaferrites are not generally replaced by any other magnetic material because they are relatively inexpensive, stable and have a wide range of technological applications. Barium hexaferrites have been classified according to their structures, into five main classes: $BaFe_{12}O_{19}$ (M-type), $BaMe_2Fe_{16}O_{27}$ (W-type), $Ba_2Me_2Fe_{28}O_{46}$ (X-type), $Ba_2Me_2Fe_{12}O_{22}$ (Y-type) and $Ba_3Me_2Fe_{24}O_{41}$ (Z-type), where Me represents a divalent ion from the first transition series.

Z-type barium hexaferrite is a promising material for applications as RAM in the frequency of GHz, which require high permeability, great resistivity and good chemical and thermal stabilities. $(Co\text{-}Cu)_2Z$ barium hexaferrite is a new type of soft magnetic compound, which presents these characteristics and a ferromagnetic resonance in the GHz frequency, being useful for inductor cores or in UHF communications, in the microwave region [3, 4].

In the conventional ceramic method, a high sintering temperature is necessary to obtain this Z-type hexaferrite due to the complex crystalline structure.

By using chemical methods, the calcination temperature can be reduced and the introduction of metallic ions makes possible the use of these ferrites as microwave absorbers in different frequency ranges, simply by varying the degree of substitution [5].In this work, the citrate sol-gel process under inert atmosphere was used to obtain $Ba_3Co_2Fe_{24}O_{41}$ and $Ba_3Co_{0.9}Cu_{1.1}Fe_{24}O_{41}$ nanopowders. The introduction of Cu^{2+} ions in the structure of $Ba_3Co_2Fe_{24}O_{41}$ can also reduce the sintering temperature, because it can acts as a flux, due to its low melting point (1084.62 °C) [6].

Composites of $Ba_3Co_2Fe_{24}O_{41}$ and $Ba_3Co_{0.9}Cu_{1.1}Fe_{24}O_{41}$ with polychloroprene (CR) were obtained for the microwave absorption measurements, for the frequency range: 8.0 – 12 GHz (X-band) and 12 – 16 GHz (Ku-band). The magnetic properties of these materials are largely dependent of the sample microstructure.

X-ray diffraction (XRD), X-ray fluorescence (XRF), thermal analyses (TGA/DTA), and the vibrating sample magnetometer (VSM) were used to characterize the synthesized material.

The microwave measurements were based on the transmission/reflection method (T/R) using rectangular waveguides as the confining medium for the samples [7].

EXPERIMENTAL

Nanosized $(Co\text{-}Cu)_2Z$ structured powders were synthesized by the citrate precursor method using reagent grade $Fe(NO_3)_3.9H_2O$, $Ba(NO_3)_2$, monohydrate citric acid, $Co(NO_3)_2.6H_2O$ and $Cu(NO_3)_2.3H_2O$ in stoichiometric molar ratios to obtain $Ba_3Co_2Fe_{24}O_{41}$ and $Ba_3Co_{0.9}Cu_{1.1}Fe_{24}O_{41}$ hexaferrites. The solids were weighing and placing then into appropriate closed vessels subjected to a super dry nitrogen atmosphere to obtain the precursor solutions [8]. Distilled water was added under agitation, until total dissolution of solids.

The solutions were then transferred to a previously evacuated flask and mixed under super dry nitrogen operating as a reflux condenser, with intensive stirring. The resulting mixture was heated to 80 °C to complete the reaction under reflux, in order to keep the inert atmosphere and to allow subsequent additions of ammonium hydroxide (NH_4OH), added drop wise into the solution to render it neutral or slightly alkaline (pH 7.0 - 8.0), for subsequent precipitation of the organo metallic complex [8].

Predried ethanol was previously added drop wise under vigorous stirring into the reaction mixture, to promote the precipitation of a complex citrate gel of barium, iron, copper and cobalt.

Drying at 60 °C, leaving behind the desired solid phase, the remaining aqueous solution was eliminated. The ideal temperature for the citrate gel decomposition was determined by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Based on the results of the thermal analyses, the batch of dried solid was calcined inside a muffle furnace. TGA and DTA measurements were carried out in a TA Instruments SDT-2960. The experiments were carried out in static air, using platinum crucibles between 20 and 1,000 °C, with a heating rate of 10 °C min^{-1}.

The calcination was performed using the following heating schedule: 2 °C/min up to 410 °C, establishing a plateau for 1 hour, 10 °C/min up to the final sintering temperature with a residence time of 4 h at the sintering temperature. The material was then cooled to room temperature at a rate of 10 °C/min.

Then, X-ray fluorescence (XRF) measurements were carried out on a Philips model PW 2,400 sequential spectrometer. This quantitative method was used to determine the stoichiometry of the ferrite samples, which were analyzed in the form of a fused bead, using lithium tetraborate flux.

For the powder X-ray diffraction (XRD) analysis, the material was placed on a glass sample holder and spread out to form a thin layer. A Siemens AXS D5005 diffractometer with a dwell time of 1 °/min, in the θ-2θ Bragg-Brentano geometry, was employed.

The magnetic hysteresis loops were obtained using the vibrating sample magnetometer VSM 4,500 PAR.

The Z-type barium hexaferrite structure is illustrated in Figure 1.