UNIVERSITI PUTRA MALAYSIA

ESTABLISHMENT OF OPTIMUM PROCESSING CONDITIONS FOR SOME TECHNOLOGICAL MANGANESE-ZINC FERRITES

ROHAIDA TAMIN

FSAS 2002 4

ESTABLISHMENT OF OPTIMUM PROCESSING CONDITIONS FOR SOME TECHNOLOGICAL MANGANESE-ZINC FERRITES

By

ROHAIDA TAMIN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, In Fulfilment of the Requirements for the Degree of Master of Science

January 2002

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of requirement for the degree of Master of Science

ESTABLISHMENT OF OPTIMUM PROCESSING CONDITIONS FOR SOME TECHNOLOGICAL MANGANESE-ZINC FERRITES

By

ROHAIDA TAMIN

January 2002

Chairman : Associate Professor Dr. Mansor Hashim

Faculty : Science and Environmental Studies

This research work is an initial step to produce manganese-zinc ferrites in the laboratory using the solid-state ceramic preparation method with controlled atmospheres. The first part of the work was to develop the required material by manipulation of composition. Then, the specimens were sintered at 1350°C in an oxygen partial pressure. At this stage, a systematic crucial approach was used to produce high permeability ferrites with suitable oxygen partial pressure. This involved a gradual introduction of nitrogen gas into the furnace during sample cooling giving an optimum equivalent isocompositional line. This work has successfully produced materials (samples MRQ1 and MRQ2) suitable with the permeability in the range of 2000 to 2500 when an average particle size was reduced to ~2.5μm and zinc oxide was added to minimize zinc loss. Then the sintering cycle was made to follow the isocompositional line.

In conclusion, an optimum processing technique has been established for producing ferrite materials with the desired magnetic properties.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia bagi memenuhi keperluan ijazah Master Sains

PENGUJUDAN KEADAAN PEMPROSESAN YANG OPTIMA BAGI BEBERAPA BAHAN FERIT MANGAN-ZINK UNTUK KEGUNAAN TEKNOLOGI

Oleh

ROHAIDA TAMIN

Januari 2002

Pengerusi : Profesor Madya Dr. Mansor Hashim

Fakulti : Sains dan Pengajian Alam Sekitar

Kerja penyelidikan ini adalah satu langkah awal untuk menghasilkan ferit mangan-zink di dalam makmal dengan menggunakan kaedah penyediaan seramik keadaan pepejal dengan atmosfera yang dikawal. Penyelidikan dimulakan dengan menghasilkan bahan yang dikehendaki melalui manipulasi terhadap komposisi. Seterusnya, pensinteran bahan spesimen pada suhu 1350°C di dalam tekanan separa oksigen dilakukan. Pada peringkat ini, suatu pendekatan yang sistematik dan amat perlu dilakukan supaya menghasilkan ferit dengan tekanan separa oksigen yang sesuai. Ini melibatkan pemasukan beransur gas nitrogen ke dalam relau semasa penyejukan sampel serta memberikan satu garisan isokomposisi setara yang optima. Penyelidikan ini berjaya menghasilkan bahan (sampel MRQ1 dan MRQ2) yang sesuai dengan julat ketelapan di antara 2000 ke 2500 apabila purata saiz zarah

dikurangkan sehingga ~2.5μm dan zink oksida ditambah untuk meminimakan kehilangan zink. Kemudian, kitaran pensinteran dijalankan supaya mengikut garisan isokomposisi tersebut.

Kesimpulannya, suatu teknik pemprosesan yang optima telah dapat diujudkan untuk menghasilkan bahan ferit dengan sifat-sifat magnet yang diperlukan.

ACKNOWLEDGEMENTS

I would like to extend my deepest gratitude and appreciation to my supervisor, **Associate Professor Dr. Mansor Hashim** and my co-supervisors, **Dr Jamil Suradi** and **Dr Jumiah Hassan**, for their invaluable guidance, advice and constant support throughout the course of this research.

I am very grateful to all my dearest friends especially Kak Ana, Zolman and Masdhiah for sharing their knowledge, advice and help towards the completion of this research.

Finally, my dearest thanks to my family members for their encouragement and most importantly to my husband for his support, care and understanding.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF PLATES

LIST OF SYMBOLS AND ABBREVIATIONS

μ_i	initial permeability
Q	quality Factor
TC	Temperature coefficient
D	disaccommodation
M_s	Saturation magnetization
M_A	Saturation magnetic moment for A site
M_B	Saturation magnetic moment for B site
B	Induction
H	Applied field
μ_o	Magnetic constant
t	Thickness
D_o	Outer diameter
D_i	Inner diameter
N	Number of wire turn
L	Inductance
μ'	Real part of permeability or magnetic loss
μ''	Imaginary part of permeability or magnetic loss

f_r	Loss resonance frequency
γ	Gyromagnetic ratio
Tan δ	Loss tangent
RLF	Relative Loss Factor
μ_m	Maximum permeability
B_s	Saturated induction
B_r	Remanence induction
H_c	Coercive force
K_1	Crystal anysotropy
μ^R	Intrinsic rotational permeability
μ^W	Wall permeability
σ	Internal stress
μ_B	Bohr magneton
P_{Zn}	Pressure of zinc
H_{max}	Magnetic field for effective saturated induction

CHAPTER I

GENERAL INTRODUCTION

Historical Overview

Magnetite or ferrous ferrite (Fe_2O_4) is an example of a naturally occurring ferrite. It has been known since more than 2000 years ago and its weak permanent magnetism found application in the compass of the early navigators. Nevertheless, there was hardly any progress in scientific research concerning ferrites until the 19th century (Ishino, 1987). Ferrite came into prominence only at the end of the Second World War (Goldman, 1990).

In an early work in 1909, Hilpert published the first systematic study of the relation between the chemical and magnetic properties of a number of the binary iron oxides but experienced difficulty in identifying the magnetic phase of his preparation. He claimed that ferrites had caused high-energy loss when subjected to alternating magnetic fields and thus had no commercial values. Around 1928 Forestier in France and Hilpert and Wille in Germany made quantitative investigations into the relation between the chemical composition, the saturation magnetization and the Curie temperature.

Japanese workers between 1932 and 1935 also studied magnetic oxides. Practical utilization of the ferrites began after the research of Kato and Takei. In 1936, Snoek was studying magnetic oxides in the Netherlands. Snoek and his co-worker, Six, realized that the most important property of a material intended as a core for an inductor is the loss tangent divided by the permeability, the so-called loss factor. This is because the loss can always be reduced by the introduction of an air gap provided the resultant permeability remains sufficient. This led Snoek to the development of manganese-zinc-ferrous ferrite in which low loss and high permeability were combined by minimizing the magnetocrystalline anisotropy and the magnetostriction. By 1945 Snoek had laid the foundations of the physics and technology of practical ferrites and a new industry came into being. Another important discovery concerning ferrites was given by Neel 1948, in the theory of ferrimagnetism, which brought about a great advance in the magnetic investigation of ferrites.

The large-scale introduction of the television in the 1950s was a major opportunity for the new ferrite industry. Ferrite cores were the material of choice in television sets for the high-voltage transformer and the picture-tube deflection system. In the 1970s, ferrite cores were used widely in telecommunication and electronic equipment such as mainframe computer memories, recording heads, etc. (Hirota et al., 1980). Since the early 1980s, ferrite cores have been used in high-frequency power supplies (Roess, 1982; Bracke, 1983).

Soft Magnetic Materials

Soft ferromagnetic or ferrimagnetic materials are those which have been developed with technical applications in view, to allow changes in magnetization to occur easily in weak magnetic fields. When the applied fields is removed, they return to a state of relatively low residual magnetism. Important magnetic properties of a soft magnetic material are high permeability, high saturation induction and low coercive force. The converse, the need for high magnetizing field and high remnant magnetism is true for hard magnetic materials.

Ferrites may be defined as magnetic materials composed of oxide containing ferric ions as the main constituent. They are hard, brittle, ceramic-like materials and are classified as ferrimagnetics. Ferrites are polycrystalline and are

generally dark grey or black in appearance. Ferrites have three distinct crystal structures: The hexagonal magnetoplumbite, dodecahedral garnet and the spinel structure (Crangle, 1991; Standley, 1972). The first structure is that of hard ferrites, the later two being those of soft ferrites.

Some of the applications for soft ferrites are for low signal, memory-core, audio-visual, and recording head applications. At low signal levels, soft ferrite cores are used for transformers and low-energy inductors. A large tonnage usage of soft ferrites is for deflection-yoke cores, flyback transformers, and convergence coils for television receivers. For these materials their survival in the intense competition from the growing technologies and their ability to enter newer areas of applications have promoted them to many disciplines. Consequently, the growing information-oriented society and the expanding roles of ferrites in electronic gadgets and other industrial pursuits have, in return, motivated this study.

Objective of Work

Besides Nickel-Zinc (NiZn) ferrites, Manganese-Zinc (MnZn) ferrites are well known as a class of ferrites showing good soft magnetic properties up to the MHz frequency range because of their high magnetic permeability and low electric losses. The development and the continued success of electronics industry have created an expanding commercial market. This market is continually challenging

the ferrite industry to produce high-quality ferrite cores capable of operating in increasingly higher frequency. MnZn ferrite is the most important ferrites for such application and constitutes a substantial portion of present-day soft ferrite production.

This project may be considered as a basic research in processing and preparation method of MnZn ferrite. Further research can be done to improve the magnetic permeability of certain composition of MnZn ferrite due to the general formula $Mn_xZn_{(1-x)}Fe_2O_4$ in order to produce better properties close to commercial specifications.

The objective of this project is to prepare the MnZn ferrites with high initial permeabilities in the range of 2000 to 5000 which are now commercially available, with the higher permeabilities limited to small toroids. Attaining such high permeabilities on a commercial scale has been a technological challenge. As such, this experiment at work can lead to the beginning of important efforts at UPM to attain high permeability soft magnetic materials, starting with MnZn ferrites.

Evolution of Magnetic Properties

Figure 1 and 2 have shown the values of maximum permeability and μQ product as a function of the year that the values were attained (Slick, 1980). This