MATERIALS SCIENCE MONOGRAPHS, 45

SOLID STATE ELECTROCHEMISTRY AND ITS APPLICATIONS TO SENSORS AND ELECTRONIC DEVICES

KAZUHIRO SYLVESTER GOTO

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Tokyo Institute of Technology, Tokyo 152, Japan

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Dedicated to Harumi Maria

PREFACE

This monograph is based on lecture notes for the course "Physical Chemistry of Oxides at High Temperature" conducted at the Graduate School of the Tokyo Institute of Technology. The course has been taught since 1971.

Three chapters of the original course notes have been left out because the topics presented in them have been adequately covered in other books. Those three chapters were on the crystal and ionic structure of oxides and the nonstoichicmetry of oxides in both solid and liquid states.

Every author must feel humble when he thinks about all the help he has received from others. I would like to express my deep thanks to the late Professor Carl Wagner,Professor Wolfgang Pluschkell,and Professor Hermann Schmalzried for their valuable comments and intensive discussions on the contents of the lecture notes.

Gratefully, I acknowledge the assistance of three associates who reviewed the English-language manuscript: Dr. Ken C. Mills, Senior Researcher at the National Physical Laboratory of Great Britain; Dr. George W. Orton, a former professor with the University of Puerto Rico,and Dr. James E. Battles, Senior Researcher of Argonne National Laboratory.

Equally appreciated is the assistance of Mr. S. Ushigome of Tokyo Yogyo Co., Ltd. and Mr. M. Matsuoka of Yamari-Electronite Co., Ltd. for their financial support for typing and art work.

Heartfelt thanks are extended to Professors Sven Eketorp, Zhou Rong-Zhang and Ji Chunlin for affording me the opportunity to present the lectures on the Physical Chemistry of Oxides at High Temperatures to the Royal Institute of Technology in Stockholm in 1972, the Peking University of Iron and Steel Technology in 1979 and the North East University of Technology in Shenyang in 1981.

Many students and colleagues have given either direct or indirect help. I am especially grateful to Professor S. Matoba, my advisor for the Bachelor and Master Degrees at Tohoku University, to Professor G. R. St. Pierre, my advisor at The Ohio State University,and to Professor Y. Matsushita, my advisor for the Doctorate of Engineering at the University of Tokyo. All have been a source

of inspiration to me in conducting my research. In particular, Professor St. Pierre wisely advised me to undertake experimental studies using new solid electrolytes with oxygen anions. This was the beginning of my investigations in the solid state electrochemistry of oxides.

It is a pleasure to acknowledge my gratitude to Professor M. Someno and to Professor S. Haruyama for teaching me the fundamentals of experimental science and electrochemical theory and to Professor R. A. Rapp for coordinating with me the USA-Japan joint work on hot corrosion of alloys.

Essential parts of the research of many former students are included in this monograph. I want to express my thanks to all of them, especially Professors M. Sasabe, M. Kawakami, K. Nagata and Doctors H. Itaya, Y. Ukyo and S. Yamaguchi.

The appendix was prepared by Mr. M. Susa and to him I am very grateful.

I take pleasure in acknowledging the continued encouragement and moral support of my wife, Harumi Maria.

Finally and most sincerely, I am grateful to Miss H. Higuchi and Miss C. Ishikawa for their patience and care in typing the manuscript and to Mr. Zhang Li-Wei for his skillful drawings.

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Chapter 1

INTRODUCTION

1.1 THE SCOPE OF THE BOOK

The scope of this book has been restricted to an examination of the electrochemistry of solid and liquid oxides at high temperatures and to its applications in various electronic devices. Electrochemistry is an interdisciplinary subject covering both chemistry and physics. Research in this area has progressed mainly through studies of aqueous solutions and molten salts, both of which have predominantly ionic bonding. In this book, electrochemical theories have been applied to both solid and liquid oxides at high temperatures. The application of the electrochemistry of oxides to various electronic devices, including chemical sensors, is discussed in the later chapters.

In the first part of the book, Chapter 2 to Chapter 5, a discussion of the micromechanisms of electric conduction and diffusion in the oxides is presented. The theoretical relation between conductivity and diffusivity is criticized with experimental results. The transport properties of multicomponent oxide systems are also discussed.

In Chapters 6 and 7, solid state galvanic cells are constructed, based on the transport properties of the oxides discussed in the previous chapters. Various types of solid state galvanic cells used for many thermodynamic studies are introduced.

In Chapters 8 and 9, the overpotential in galvanic cells and its applications to kinetic studies are discussed.

The most successful application of the solid state electrochemistry of oxides is generally considered to be in oxygen sensors. These electronic devices are discussed in Chapters 10 and 11.

Applications of solid oxides in various electronic devices are discussed in Chapters 12 and 13. Gas sensors with semiconductor oxides, humidity sensors, gas sensors with MOS transistors, electrochemical timers, potential memory cell, switching device, display device, and solid state batteries with high energy density are covered in detail.

The first part of the book is designed to give the reader an insight into the micromechanism of transport phenomena of ions

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and electrons in solid and liquid oxides. In the second part, various solid state galvanic cells are discussed in relation to the transport properties of oxides. Then, it is demonstrated how these galvanic cells are used for thermodynamic and kinetic studies at high temperatures.

In the last part of the book, many electronic devices using various solid oxides are introduced as examples of the application of solid state electrochemistry to electronic devices.

Solid and liquid oxides of metals are discussed because of their great importance in various fields of industry.

The oxides of metals have long been used by mankind as raw materials for pottery, refractories, cement, and ceramics. Furthermore, oxides are important as the ores from which metals can be extracted.

In contrast to these classic fields of industry, oxides are now being very extensively studied as promising functional materials for a rapidly advancing technology.

This new technology can be classified into the following; 1) the technology of new functional materials, 2) microelectronic technology (LSI, MOS-IC, optoelectrics, laser devices, etc), 3) sensor technology, 4) energy saving and converting technology, 5) life science and biotechnology, 6) robots for factory automation and 7) space or ocean technology.

In these new technologies, the oxides are used as components of the devices. In the following, the author would like to illustrate the importance of the oxides with two examples from industry; one from a classic field and the other from a newly advancing field.

1.2 IMPORTANCE OF OXIDES IN METALLURGY

The art of extraction and refining metals has been developed over the centuries in a wide variety of locations throughout the world. The crust of the earth is composed of the oxides of metals, which have been and will continue to be the original source of most metals.

Table 1.1 shows the oxide ores along with their mineralogical names. This table was made by rearranging the order and eliminating the sulfide ores from Table 1-1 of the excellent textbook, "Unit Processes of Extractive Metallurgy" by R.D. Pehlke(1).

TABLE 1.1
Oxide ores for common metals

Metal	Formula	Name of Mineral
Iron	Fe_3O_4	Magnetite
	Fe_2O_3	Hematite
	$2Fe_2O_3\cdot3H_2O$	Limonite
Copper	Cu_2O	Cuprite
Aluminum	$Al_2O_3\cdot H_2O$	Diaspore
	$Al_2O_3\cdot3H_2O$	Gibbsite
	$Al_2O_3\cdot2SiO_2\cdot2H_2O$	Kaolinite
Zinc	ZnO	Zincite
	$ZnO-FeO-MnO$	Franklinite
Magnesium	MgO	Magnesite
Tin	SnO_2	Cassiterite
Manganese	MnO_2	Pyrolusite
	$MnSiO_3$	Rhodonite
Chromium	$FeCr_2O_4$	Chromite
Titanium	$FeO\cdot TiO_2$	Ilmenite
	TiO_2	Rutile
Zirconium	ZrO_2	Baddeleyite
	$ZrSiO_4$	Zircon
Vanadium	$K_2O\cdot2UO_2\cdot V_2O_5\cdot3H_2O$	Carnotite
Molybdenum	MoO_3	Molybdite
Tungsten	$FeWO_4$	Wolframite
	$CaWO_4$	Scheelite
Beryllium	$3BeO\cdot Al_2O_3\cdot6SiO_2$	Beryl
Uranium	Complex Oxide	Pitchblende

(Rearranged from Table 1-1 of the book by R.D. Pehlke(1))

The modern science of extraction and refining of metals has a relatively short history, spanning less than two centuries. The common principle of the extraction and of the purification of metals is based on selective reduction and oxidation, usually at high temperature. Scientific theories of thermodynamics and kinetics at high temperature have been used to produce metals with precise composition at the lowest cost.

Table 1.1 indicates the importance of oxides in metallurgy. It is evident that many useful metals are produced from oxide ores.

Furthermore, oxides used in refractories and ceramics are also very important in refining metals. This will now be concisely discussed with help of an example of metal refining. Figure 1.1 shows a schematic picture of a modern method of producing steel from the oxide ores in Table 1.1.

In the blast furnace, iron is extracted from the oxide by selective reduction with carbon monoxide gas. The separation of metallic iron is accompanied by the migration of ionic species, and hence electrochemical processes are involved in the blast furnace.

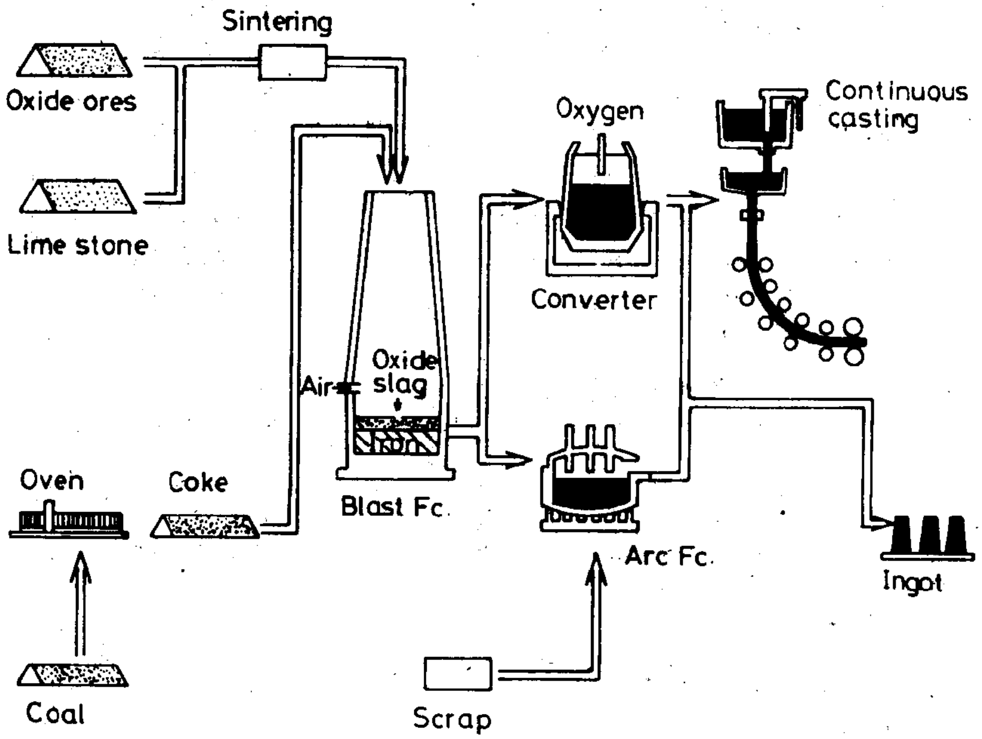

Fig. 1.1 Flow chart of production of steel from iron ores.

The gangue minerals of Al_2O_3, SiO_2, MgO, etc. float upwards and are separated from iron melt as a liquid slag. Essential portions of the impurities are removed with the liquid slag. However, the molten iron with a temperature of about 1500°C still contains a significant amount of impurities.

This crude iron is refined by the selective oxidation of impurities in the converter or in the electric arc furnace, as shown in Fig. 1.1. The refined iron is called "steel". The liquid steel is then solidified into convenient shapes for subsequent rolling and forging. Nowadays steel is usually solidified by the continuous casting process or by the traditional mould-casting method.

Refractories for furnaces are made from oxides such as Al_2O_3, SiO_2 and MgO and their life time is usually controlled by the hot corrosion of liquid slags which are also formed from oxides. As the bonding in both the refractories and the slags is predominantly ionic, hot corrosion is an electrochemical process. One more electrochemical phenomenon in steelmaking is the transfer of impurities between liquid slags and the iron or steel bath. As the slag has an electrolytic nature, the transfer of the impurities is accompanied by charge transfer reactions. This is a phenomenon similar to that observed when electrodes are dipped in aqueous electrolytic solutions.

The present author believes that the applications of electrochemical theories will be helpful in gaining a deeper understanding of natural phenomena taking place during the extraction and refining of metals at high temperature.

More details of iron and steelmaking processes are given in several books listed(2)-(17) at the end of this chapter.

1.3 OXIDES AS FUNCTIONAL ELEMENTS IN MICROELECTRONICS

Metallurgy has a long history spanning thousands of years and it will continue to be very important in the future development of mankind. In a strong contrast to this, we now have several new, rapidly developing fields of technology. Their histories are very short, a few decades at the most.

Among these rapidly-developing technologies, "microelectronics" might be considered the most important. This name of "microelectronics" is used to distinguish the new electronic technology from the classic electrical engineering.

Microelectronics can be defined as a new branch of technology, in which the art and science are being developed in order to produce elegant <u>electronic</u> devices by using new "<u>micro</u>-sized" materials.

Day after day, many electronic materials are being developed. A significant fraction of these new materials are the oxides of metals. Table 1.2 shows examples of solid oxides of metals either being studied or used as electronic materials.

This table was compiled from an encyclopedia of functional materials written by M. Kitada(18) in 1984 and for the sake of brevity only examples of the solid oxides have been included. In fact, the sensor materials alone in Table 1.2 can be sub-divided into another table (Table 1.3). This table was compiled from the proceedings of International Meeting on Chemical Sensors(19)

TABLE 1.2

Examples of solid oxides used as electronic materials

Electronic Materials	Solid oxide elements
MATERIALS for IC, MOS-IC, LSI	SiO_2 in Mo|SiO_2|Si Al_2O_3 as substrate of IC
Magnetic Materials	γ-Fe_2O_3, BaO-6Fe_2O_3, MnO-ZnO-Fe_2O_3 MnO-MgO-Fe_2O_3, $YFeO_3$, $YCoO_3$, $Y_3Fe_5O_{12}$
Optoelectronic Materials	Na_2O-B_2O_3-SiO_2, Na_2O-Li_2O-CaO-SiO_2 for optical fibres. $LiNbO_3$, $LiTaO_3$, $PbTiO_3$ for optical switch.
Sensor Materials	SnO_2, In_2O_3, ZnO, NiO, FeO, $BaTiO_3$ for semiconductor gas sensors. ZrO_2-MgO, ZrO_2-Y_2O_3 for solid electrolyte gas sensors.
Laser Materials	$Y_3Al_5O_{12}$-Nd_2O_3, $CaWO_4$, $YAlO_3$, Al_2O_3-Cr_2O_3, Phosphate glass doped with Nd_2O_3
Piezoelectric Materials	$PbTiO_3$, $PbZrO_3$, $LiNbO_3$ $LiTaO_3$
Ceramic superconductors	$LiTi_2O_4$, $BaPb_{1-x}Bi_xO_3$, CuO-BaO-Y_2O_3
Condensor Varistor Registor	$BaTiO_3$, $CaTiO_3$, $ZnTiO_3$, $SrTiO_3$, $Bi_2Ti_2O_5$ for condensors. ZnO-Bi_2O_3 for varistor, RuO_2 for registors.
Others	In_2O_3-SnO_2 for transparent conductor. In_2O_3-Cu_2O for solar cell.

TABLE 1.3

Solid oxides used for sensor elements

Sensors	Examples of sensor elements
Thermo-sensors (Thermister)	NTC*: NiO, FeO, CoO, MnO, $CoO-Al_2O_3$ PTC*: $BaTiO_3$
Semiconductor gas sensors	SnO_2, In_2O_3, ZnO, WO_3, $\gamma-Fe_2O_3$ NiO, CoO, Cr_2O_3, $LaNiO_3$, $BaTiO_3$ TiO_2, $CoO-MgO$
Humidity sensors	Al_2O_3, P_2O_5, $ZnO-Li_2O$, $NiFe_2O_4$ TiO_2, $MgCr_2O_4$, Fe_3O_4, $ZnO-Cr_2O_3$ ZrO_2-MgO, $SrSnO_3$, $SrTiO_3$
Infrared optical sensors	$LiNbO_3$, $LaTaO_3$, $SrTiO_3$ $Ba_2NaNb_5O_{15}$, $LiNbO_3$
Solid electrolyte gas sensors	ZrO_2-MgO, $ZrO_2-Y_2O_3$, ZrO_2-CaO, $ThO_2-Y_2O_3$, $Na_2O-11Al_2O_3(\beta-Al_2O_3)$ $SrO-CeO_2-Yb_2O_3$, $Bi_2O_3-MoO_3$, $Na_3Zr_2Si_2PO_{12}$

* NTC and PTC mean negative and positive temperature coefficient of electric resistivity with temperature increase, respectively.

edited by T. Seiyama et al in 1983.

From Tables 1.2 and 1.3, one can see that many kinds of solid oxides are used in integrated circuits, in metal-oxide-semiconductor transistors, in magnetic materials, in optical fibers, in optical switches, in optoelectrical devices, in sensors, in laser generators, and in various other microparts such as piezoelectrics, superconductors, condensors, varistors and transparent conductors.

The oxides used in these applications are produced by sintering, by hot pressing, or by solidification from liquid state. They are often used in the form of thin films, thin wires or ultrafine powders. For such cases, they are produced by physical vapor deposition, by chemical vapor deposition, by ion sputtering, by molecular beam epitaxy, or by combinations of three methods.

Figure 1.2 demonstrates how silicon oxide, SiO_2, is used in microelectronic devices. This "MOS-FET" is a kind of transistor, the abbreviation standing for Metal-Oxide-Semiconductor-Field-