SOL-GEL SILICA

Properties, Processing and Technology Transfer

by
Larry L. Hench

NOYES PUBLICATIONS

SOL-GEL SILICA

This page intentionally left blank

SOL-GEL SILICA

Properties, Processing and Technology Transfer

by

Larry L. Hench

Imperial College of Science, Technology and Medicine
London, England

np NOYES PUBLICATIONS
Westwood, New Jersey, U.S.A.

Library of Congress Catalog Card Number: 98-19933
ISBN: 0-8155-1419-0
Printed in the United States

Published in the United States of America by
Noyes Publications
369 Fairview Avenue, Westwood, New Jersey 07675

10 9 8 7 6 5 4 3 2 1

Library of Congress Cataloging-in-Publication Data

Hench, L. L.
Sol-gel silica : properties, processing, and technology transfer / by Larry L. Hench.
p. cm.
Includes bibliographical references and index.
ISBN 0-8155-1419-0
1. Ceramic materials. 2. Colloids. I. Title.
TP810.5.H45 1998
620.1'4--dc21 98-19933
CIP

MATERIALS SCIENCE AND PROCESS TECHNOLOGY SERIES

Series Editors

Rointan F. Bunshah, University of California, Los Angeles
Gary E. McGuire, Microelectronics Center of North Carolina
Stephen M. Rossnagel, IBM Thomas J. Watson Research Center

Electronic Materials and Process Technology

CHARACTERIZATION OF SEMICONDUCTOR MATERIALS, Volume 1: edited by Gary E. McGuire

CHEMICAL VAPOR DEPOSITION FOR MICROELECTRONICS: by Arthur Sherman

CHEMICAL VAPOR DEPOSITION OF TUNGSTEN AND TUNGSTEN SILICIDES: by John E. J. Schmitz

CHEMISTRY OF SUPERCONDUCTOR MATERIALS: edited by Terrell A. Vanderah

CONTACTS TO SEMICONDUCTORS: edited by Leonard J. Brillson

DIAMOND CHEMICAL VAPOR DEPOSITION: by Huimin Liu and David S. Dandy

DIAMOND FILMS AND COATINGS: edited by Robert F. Davis

DIFFUSION PHENOMENA IN THIN FILMS AND MICROELECTRONIC MATERIALS: edited by Devendra Gupta and Paul S. Ho

ELECTROCHEMISTRY OF SEMICONDUCTORS AND ELECTRONICS: edited by John McHardy and Frank Ludwig

ELECTRODEPOSITION: by Jack W. Dini

HANDBOOK OF CARBON, GRAPHITE, DIAMONDS AND FULLERENES: by Hugh O. Pierson

HANDBOOK OF CHEMICAL VAPOR DEPOSITION: by Hugh O. Pierson

HANDBOOK OF COMPOUND SEMICONDUCTORS: edited by Paul H. Holloway and Gary E. McGuire

HANDBOOK OF CONTAMINATION CONTROL IN MICROELECTRONICS: edited by Donald L. Tolliver

HANDBOOK OF DEPOSITION TECHNOLOGIES FOR FILMS AND COATINGS, Second Edition: edited by Rointan F. Bunshah

HANDBOOK OF ION BEAM PROCESSING TECHNOLOGY: edited by Jerome J. Cuomo, Stephen M. Rossnagel, and Harold R. Kaufman

HANDBOOK OF MAGNETO-OPTICAL DATA RECORDING: edited by Terry McDaniel and Randall H. Victora

HANDBOOK OF MULTILEVEL METALLIZATION FOR INTEGRATED CIRCUITS: edited by Syd R. Wilson, Clarence J. Tracy, and John L. Freeman, Jr.

HANDBOOK OF PLASMA PROCESSING TECHNOLOGY: edited by Stephen M. Rossnagel, Jerome J. Cuomo, and William D. Westwood

HANDBOOK OF POLYMER COATINGS FOR ELECTRONICS, 2nd Edition: by James Licari and Laura A. Hughes

HANDBOOK OF REFRACTORY CARBIDES AND NITRIDES: by Hugh O. Pierson

HANDBOOK OF SEMICONDUCTOR SILICON TECHNOLOGY: edited by William C. O'Mara, Robert B. Herring, and Lee P. Hunt

HANDBOOK OF SEMICONDUCTOR WAFER CLEANING TECHNOLOGY: edited by Werner Kern

HANDBOOK OF SPUTTER DEPOSITION TECHNOLOGY: by Kiyotaka Wasa and Shigeru Hayakawa

HANDBOOK OF THIN FILM DEPOSITION PROCESSES AND TECHNIQUES: edited by Klaus K. Schuegraf

HANDBOOK OF VACUUM ARC SCIENCE AND TECHNOLOGY: edited by Raymond L. Boxman, Philip J. Martin, and David M. Sanders

HANDBOOK OF VLSI MICROLITHOGRAPHY: edited by William B. Glendinning and John N. Helbert

HIGH DENSITY PLASMA SOURCES: edited by Oleg A. Popov

HYBRID MICROCIRCUIT TECHNOLOGY HANDBOOK, Second Edition: by James J. Licari and Leonard R. Enlow

IONIZED-CLUSTER BEAM DEPOSITION AND EPITAXY: by Toshinori Takagi

MOLECULAR BEAM EPITAXY: edited by Robin F. C. Farrow

SEMICONDUCTOR MATERIALS AND PROCESS TECHNOLOGY HANDBOOK: edited by Gary E. McGuire

ULTRA-FINE PARTICLES: edited by Chikara Hayashi, R. Ueda and A. Tasaki

Ceramic and Other Materials—Processing and Technology

ADVANCED CERAMIC PROCESSING AND TECHNOLOGY, Volume 1: edited by Jon G. P. Binner

CEMENTED TUNGSTEN CARBIDES: by Gopal S. Upadhyaya

CERAMIC CUTTING TOOLS: edited by E. Dow Whitney

CERAMIC FILMS AND COATINGS: edited by John B. Wachtman and Richard A. Haber

CORROSION OF GLASS, CERAMICS AND CERAMIC SUPERCONDUCTORS: edited by David E. Clark and Bruce K. Zoitos

FIBER REINFORCED CERAMIC COMPOSITES: edited by K. S. Mazdiyasni

FRICTION AND WEAR TRANSITIONS OF MATERIALS: by Peter J. Blau

HANDBOOK OF CERAMIC GRINDING AND POLISHING: edited by Ioan D. Mavinescu, Hans K. Tonshoff, and Ichiro Inasaki

HANDBOOK OF INDUSTRIAL REFRACTORIES TECHNOLOGY: by Stephen C. Carniglia and Gordon L. Barna

SHOCK WAVES FOR INDUSTRIAL APPLICATIONS: edited by Lawrence E. Murr

SOL-GEL TECHNOLOGY FOR THIN FILMS, FIBERS, PREFORMS, ELECTRONICS AND SPECIALTY SHAPES: edited by Lisa C. Klein

SOL-GEL SILICA: by Larry L. Hench

SPECIAL MELTING AND PROCESSING TECHNOLOGIES: edited by G. K. Bhat

SUPERCRITICAL FLUID CLEANING: edited by John McHardy and Samuel P. Sawan

Preface

This book summarizes fifteen years of research in sol-gel processing of silica leading to the commercial development of gel-silica optics and bioactive gel-glasses for medical applications. Each of the seven stages of sol-gel processing is described with emphasis on control of the ultrastructural variables necessary to make reproducible materials. A unique feature of the book is its emphasis on the practical outcome of the processing studies, i.e., commercially viable production of net shape, net surface, and monolithic optical components. It is shown that reliable drying of gel-silica optics requires control of gel texture, especially the distribution of pore sizes, which is a function of the kinetics of hydrolysis and condensation reactions occurring during mixing and gelation, and also the kinetics and environment of the cast component.

The environmental stability of porous gel monoliths is of special concern in the book because it can limit the utility of porous optical matrices, Type VI gel-silica, and also severely restrict the ability to transform porous gel into a fully dense silica optical component, Type V gel silica. The book shows how theory and experiment are combined to solve the problem of environmental stabilization; semi-empirical quantum mechanical calculations provide insight as to the mechanism of water attack of surface defects within the interconnected pore network. Experiments are described which determined the concentration of surface defects, the dimensions of the hydrated layers within the network and the specific thermochemical treatments required to stabilize the surface of the pores without collapsing them.

The ability to make stable porous matrices with interconnected porosity of controllable size makes it possible to impregnate the pores with optically active second phases. The processing and properties these new generations of optical composites are described in the book with numerous examples from published papers and Ph.D. theses. The production of fully dense optical silica components is also presented along with many advantages including: improved physical properties and net shape, net surface processing of microlenses, surface diffractive optics, aspheres, micro-optical arrays, and laser written waveguides.

Addition of CaO and P_2O_5 to the silica precursor during mixing leads to a new material, a biologically active gel-glass which enhances the repair and regeneration of bone. A chapter reviews the concept of bioactive implant materials and the important advances associated with sol-gel processing of bioactive glasses.

A final chapter addresses one of the most difficult topics of all: how to make the transfer of technology from the laboratory to a profitable commercial enterprise. Principles of technology transfer are presented and the separate pathways involved in moving from a research program to pilot plant are discussed using examples from various chapters in the book. Thus, this book presents an unique perspective of twentieth century research and development in materials science. It starts with the concept of using ultrastructure processing to produce a new generation of optical and medical materials and shows step by step the sequence of experiments conducted to reduce the concept to a commercial reality.

London, England
February, 1998

Larry L. Hench