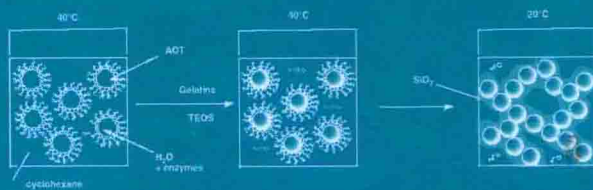


# VOLUME I: SOL-GEL PROCESSING

VOLUME EDITOR: **HIROMITSU KOZUKA**



## HANDBOOK OF SOL-GEL SCIENCE AND TECHNOLOGY Processing Characterization and Applications

**EDITOR** Sumio Sakka



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# HANDBOOK of SOL-GEL SCIENCE and TECHNOLOGY

*Processing,  
Characterization  
and Applications*

*edited by*

**Sumio Sakka**

Professor Emeritus of Kyoto University  
Hirakata, Osaka, Japan

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## VOLUME I *SOL-GEL PROCESSING*

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KLUWER ACADEMIC PUBLISHERS  
Boston/Dordrecht/London

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**Distributors for North, Central and South America:**

Kluwer Academic Publishers  
101 Philip Drive  
Assinippi Park  
Norwell, Massachusetts 02061 USA  
Telephone (781) 871-6600  
Fax (781) 871-6528  
E-Mail: [kluwer@wkap.com](mailto:kluwer@wkap.com)

**Distributors for all other countries:**

Kluwer Academic Publishers Group  
Post Office Box 322  
3300 AH Dordrecht, THE NETHERLANDS  
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E-Mail: [services@wkap.nl](mailto:services@wkap.nl)



Electronic Services (<http://www.wkap.nl>)

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**Library of Congress Cataloging-in-Publication Data**

Handbook of sol-gel science and technology: processing, characterization, and applications/edited by Sumio Sakka.

p. cm.

Includes bibliographical references and indexes.

Contents: v. 1. Sol-gel processing / volume editor, Hiromitsu Kozuka – v. 2.

Characterization of sol-gel materials and products / volume editor, Rui M. Almeida – v. 3.

Applications of sol-gel technology / volume editor, Sumio Sakka.

ISBN 1-4020-7969-9 (set: acid-free paper) – ISBN 1-4020-7966-4 (v. 1: acid-free paper) – ISBN 1-4020-7967-2 v. 2: acid-free paper) – ISBN 1-4020-7968-0 (v. 3: acid-free paper)

1. Ceramic materials. 2. Colloids. I. Sakka, Sumio.

TP810.5.H36 2004

666—dc22

2004054888

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Printed in the United States of America.

9 8 7 6 5 4 3 2 1

SPIN 11052715

[springeronline.com](http://springeronline.com)

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**VOLUME I**  
***SOL-GEL PROCESSING***

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# Preface to the Handbook (Sol-Gel Science and Technology)

This three-volume Handbook “Sol-Gel Science and Technology” was planned with the purpose of providing those who are interested in processing, characterization and application of materials with comprehensive knowledge on sol-gel science and technology.

Around 1970, three different groups in the field of inorganic materials published research results on preparation of glass and ceramics via solutions or sol-gel route. H. Dislich prepared a pyrex-type borosilicate glass lens by heating a compact of metal alkoxide derived powder at temperatures as low as 650°C. R. Roy prepared a millimeter-size small piece of silica glass via sol-gel route at temperatures around 1000°C. Mazdiyasi et al. showed that well-sintered, dense ferroelectric ceramics can be obtained at temperatures as low as 900°C, when sol-gel powders prepared from solutions of metal alkoxides are employed for sintering.

Those works stimulated people's interest in sol-gel preparation of inorganic materials, such as glasses and ceramics. Materials scientists and engineers paid attention to the possibility of this method in giving shaped materials directly from a solution without passing through the powder processing and the fact that the maximum temperature required for processing is very low compared with conventional technology for preparing glasses and ceramics. Thus, many efforts have been made in preparing bulk bodies, coating films, membranes, fibers and particles, and many commercial products were born.

The significant characteristics unique to the sol-gel method became evident, when organic-inorganic hybrid materials were prepared by H. Schmidt and silica materials containing functional organic molecules were prepared by Avnir in early 1980's. Such materials are produced at low temperatures near room temperature, where no decomposition of organic matter takes place. Low temperature synthesis and preparation of materials is the world of chemists. Therefore, the sol-gel method was propagated to the wide area including not only glasses and ceramics, but also organic and biomaterials.

In 1990, an excellent book entitled “Sol-Gel Science” was written by Brinker and Scherer, obtaining a very high reputation. However, the remarkable scientific and technological development and broadening in the sol-gel field, together with an enormous increase in sol-gel population, appeared to demand publication of a new, comprehensive Handbook on sol-gel science and technology.

Thus, it was planned to publish the present Handbook, which consists of the following three volumes:

## Volume 1 Sol-Gel Processing

Volume editor: Prof. Hiromitsu Kozuka

## Volume 2 Characterization and Properties of Sol-Gel Materials and Products

Volume editor: Prof. Rui M. Almeida

## Volume 3 Applications of Sol-Gel Technology

Volume editor: Prof. Sumio Sakka

Volume 1 compiles the articles describing various aspects of sol-gel processing. Considering that the sol-gel method is a method for preparing materials, the knowledge on sol-gel processing is of primary importance to all those who are interested in sol-gel science and

technology. Articles describing processing of some particular property as well as general basics for sol-gel processing are collected.

Volume 2 consists of the articles dealing with characterization and properties of sol-gel materials and products. Since materials exhibit their functional properties based on their microstructure, characterization of the structure is very important. We can produce useful materials only when processing-characterization-property relationships are worked out. This indicates the importance of the articles collected in Volume 2.

The title of Volume 3 is "Applications of Sol-Gel Technology". The sol-gel technology is one of the methods for producing materials and so there are many other competitive methods, whenever a particular material is planned to be produced. Therefore, for the development of this excellent technology, it is important to know the sol-gel science and technology in producing new materials as well as already achieved applications. This is the purpose of Volume 3.

Sol-gel technology is a versatile technology, making it possible to produce a wide variety of materials and to provide existing materials with novel properties. I hope this three-volume Handbook will serve as an indispensable reference book for researchers, engineers, manufacturers and students working in the field of materials.

Finally, I would like to express my sincere thanks to all the authors of the articles included in the Handbook for their efforts in writing excellent articles by spending their precious time. As general editor I extend my thanks to Prof. H. Kozuka and Prof. R. Almeida for their difficult work of editing each Volume. I have to confess that this Handbook would not have been realized without enthusiastic encouragement of Mr. Gregory Franklin, senior editor at Kluwer Academic Publishers.

**Sumio Sakka**

# Preface to Volume 1

## ("Sol-Gel Processing")

Volume 1 entitled "Sol-Gel Processing" has 27 chapters, concerning techniques for sol-gel processing of materials of specified shapes, structure, and chemistry, including chemistry of precursors and special processing techniques.

Sol-gel technology has already long history, starting with processing of oxide materials including glass and ceramics about 30 years ago. However, since then, the technology has been employed in preparation not only of oxides, but also of non-oxide materials including nitrides, carbides, fluorides, and sulfides as well as oxynitride and oxycarbide glasses. Processing of organic-inorganic materials is now a very active field of research, which has been expanded even in the field of biotechnology as is represented by research on encapsulation of enzymes, antibodies and bacteria.

The sol-gel technology started with processing of dense, bulk materials, and great efforts have been made on how to densify porous gels into glasses and ceramics. However, recently sol-gel processing of mesoporous and macroporous materials has also attracted much attention, including materials with well-controlled pore characteristics and highly porous materials, which have excellent chemical and photonic functions. As far as the shape of the products are concerned, powders and fibers are also important products via sol-gel processing, the techniques of which are still in progress. Thin films or coatings are other shaped materials that can be prepared by sol-gel method, which also has already long history. Although dip- and spin-coating methods are very familiar techniques, and science on sol-gel thin film deposition seems to have been established already, there are still technical issues to be solved scientifically for practical fabrication of industrial products. Deposition techniques have now a variety, such as ultrasonic pulverization of aerosols and electrophoretic deposition, and those allowing coating of plastic materials and self-standing thick films have also been developed. There are also special techniques like non-hydrolytic sol-gel technique, which produces unique materials, and UV irradiation that activates the chemical bonds of organic and inorganic components in sol-gel films.

Metal alkoxides are the most important precursors employed in sol-gel processing. The development of synthesis technique is often the key for preparing materials of excellent functions with sophisticated nanostructures. Then, how about other precursors than alkoxides? Sometimes people say that "sol-gel method" exclusively represents processing that undergoes hydrolysis and polycondensation of metal alkoxides, ending up with formation of metalloxane bonds. And also some people say that "sol" represents "colloidal solutions," not "polymer solutions." Prof. Pierre-Gilles de Gennes, who was awarded the Nobel Prize in Physics in 1991, wrote a famous book entitled "Scaling Concepts in Polymer Physics" (Cornel University Press, Ithaca, 1979). In that book, first he precisely describes theoretical aspects of "polymer solutions," which he never calls "sols." Then, he describes the conversion of "polymer solutions" into "gels," which he calls "sol-gel transition," however. No other terms than "sol-gel transition" can represent this kind of conversion, and we should recognize that "sol-gel method" is a processing that passes through "sol-gel transition" irrespective of the kinds of precursors. Science and technology have been greatly developed on (i) metal salt routes for thin film deposition, (ii) polymeric

gel precursors for materials with high homogeneity, and (iii) aqueous precursors ideal for material production in industries.

The current state of science and technology on all of these are covered in Volume 1, and the chapters are grouped in eight parts; (1) Sol-gel precursors, (2) Processing of powders and bulk materials, (3) Processing of non-oxide materials, (4) Processing of thin films, (5) Processing of fibers and monodisperse particles, (6) Encapsulation of organic materials, (7) Processing of catalysts, porous materials and aerogels, and (8) Special techniques used in sol-gel processing. Each chapter has been written by a leading expert in the field. I hope that Volume 1 will provide great information on the current state of sol-gel processing of materials of specified shapes, structure, and chemistry, including chemistry of precursors and special processing techniques.

Finally I would like to thank all the authors for spending their precious time, and making much efforts to make great contribution to the Handbook. I also thank Mr. Gregory Granklin, senior editor in Kluwer Academic Publishers and Prof. Sumio Sakka for continuous encouragement.

**Hiromitsu Kozuka**



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