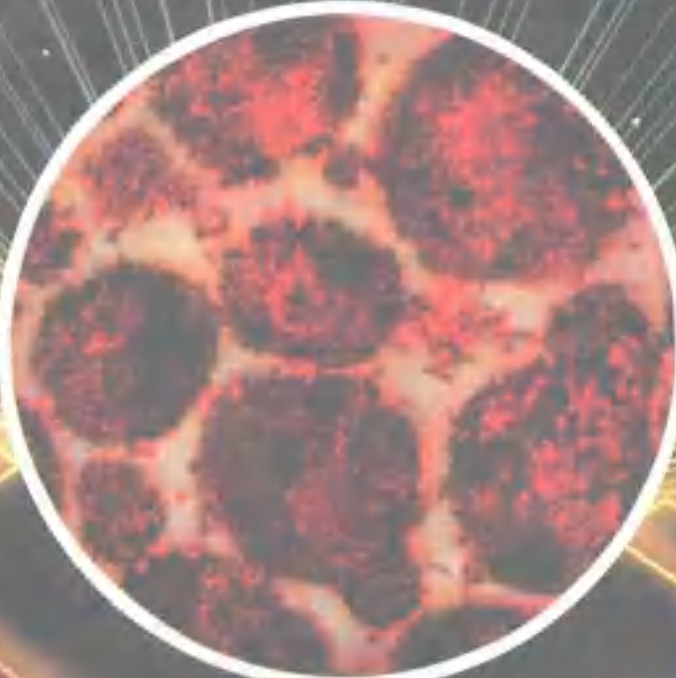
MICROSTRUCTURE OF CERAMICS



诸培南 翁臻培 王天颐 主编

无机非金属材料显微結构图册

无机非金属材料显微结构图册

Microstructure of Ceramics

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无机非金属材料
显微結构图册

嚴東生

一九九四年六月

中国科学院院士
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【内容简介】

这是一部无机非金属材料方面的专著。本图册收集了我国无机非金属材料显微结构研究和生产检验过程中所积累起来的具有代表性的图片，根据无机非金属材料科学原理，论述了无机非金属材料显微结构的形成和物相结合分布等特征，为无机非金属材料质量评价、生产工艺控制、增产降耗、新材料设计与研制等提供了必要的理论基础。

全书卷首有绪论(中英文对照)，阐述了显微结构研究的原理、方法以及当前的进展。接着按陶瓷、耐火材料、玻璃、铸石、烧结矿与球团矿、燃料灰渣与工业废渣、复合材料、水泥与混凝土、非均质材料的显微结构与性质等列成九章，分别从显微结构的形成和各类材料的显微结构特征和性质作了结合实际的讨论和说明。最后第十章为"分形学在无机非金属材料显微结构研究中的应用"。第九、第十两章体现了本学科在近年来发展的重要方面。全书图文并茂，形象直观。

本书适合于从事无机非金属材料研究和生产的广大科技人员阅读参考；亦可供高等学校作为"无机非金属材料科学与工程"专业的教材或参考书。

Summary of content

This is a monograph of inorganic non-metallic materials or ceramics. In this book, the authors have collected a definite quantities of representative pictures accumulated from the research works and testings works concerning the microstructural studies of ceramics. Based on the principles of ceramic science, the authors described and discussed the formation of microstructure and the characteristics of binding or distributing state of ceramics, that may be provided as the theoretical basis in the evaluation of qualities of products, technological control of process and in improving the quality of production, lowering the consumption as well as in the designing or in researching and manufacturing of new ceramics.

At the beginning of this book, in introduction (in Chinese and English), the authors expounded the essential principles and methods as well as the developments of microstructural studies, then according to the chapter series including ceramic, refractory, glass, casting rock, sinter and pellet, fuel ash and industrial slag, composite cement and concrete, as well as microstructure and property of inhomogeneous materials, all of the above nine chapters are enumerated respectively. Depending on the pratical uses of different materials, the formation of microstructure and the microstructural characteristics with repect to their properties were discussed and illustrated. Finally, the chapter 10 titled as "Fractology and its applications in the microstructural investigation of ceramics" was added. Here, chapter 9, 10 may be regarded as the important respects concerning the recent development of the discipline itself. It is indicated that in this book, both in pictures and writing articles parts are excellent.

This book is recomended to the readers who are undertaking research works or technical works of production. It may be also recomended that this book may be used as teaching material or reference book for the students who are studying in the colleges majoring in materials science and engineering.

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序

无机非金属材料占材料科学中的一大部分。近年来世界各国都极为重视它的开发和应用。我国非金属矿产品及非金属制成品资源丰富，种类繁多，材料科学的发展亦极其迅速。它们的应用具有广阔的前景。很自然，与无机非金属材料有关的学科与理论，也就相应地丰富起来。

为了满足这方面的迫切需要，《无机非金属材料显微结构图册》一书，由许多位知名专家、教授集体编撰而成。书中就陶瓷材料、耐火材料、玻璃材料、铸石材料、烧结矿与球团矿材料、工业废渣材料、复合材料、水泥及混凝土材料等还包括该学科理论部分的新发展，进行分章论述。

本书主要内容是先从与各章都有关系的相律图谈起。继而是显微结构研究及晶体的生长。此后即各种材料的分论。统观各节，取材新颖，内容翔实。

本书名为显微结构图册，强调了显微结构的方面。但实际内容不仅包括显微结构，读者可以从中获得许多方面的有关知识。例如，书中对于不少种无机非金属材料制品中可能遇到的缺陷进行了核验和分析；书中阐明了热工窑炉受侵蚀的机理，提出了改进工艺的措施，从而得以延长其使用寿命，以及研究设计新材料的方向等等，统统都有所涉及。

本书是一本较为完善的研究工作手册；也是一部很好的教材及参考书籍。

苏良赫

1994 年 6 月

序　言

材料是技术进步的物质基础和先导。当代各种新型材料的出现和传统材料的改进，大大促进了工业、农业、国防、科技等国民经济各部门的快速发展，其中，无机非金属材料的发展和成就令人瞩目。显微结构研究作为一门独立的学科，在无机非金属材料研究领域中，起着不同寻常的作用。这是由材料的性能与其自身的显微结构密切相关所决定的。材料的显微结构研究，在与材料性能相关的诸领域，包括：材料性能的评价；改善材料性能的途径的研究；材料生产工艺过程的控制；新材料设计与研制等许多方面，都发挥了重要作用。可以说，在无机非金属材料研究领域，没有哪一项成果不和显微结构研究有着某种内在的联系。同时，材料科学研究的进展，超细粒子的出现，把显微结构研究推向更新的阶段，从而促进了这一学科的更深入发展。

《无机非金属材料显微结构构图册》作为我国第一部系统全面论述无机非金属材料显微结构的学术著作，为读者展示了700余幅典型的无机非金属材料显微结构图片，并以文字的说明阐述了显微结构研究的原理、方法，并分别揭示了陶瓷、耐火材料等各种无机非金属材料的显微结构特征与材料品种、性能、生产工艺条件之间的关系。这些珍贵图片和相关的文字说明，归纳和融汇了这一领域数十年的研究成果和生产实践积累的资料，同时也涉及了本领域的最新研究动向，如非均质材料的显微结构效应，分形学在无机非金属材料显微结构研究中的应用等。因此，本书不仅是一部对指导生产和科学研究具有重要实用意义的工具书，而且是一部代表了本领域这个时代水平的宏篇巨篇，对推动无机非金属材料学科发展具有重要的学术价值。

本书于1986年底始由国家建材局教材办公室和全国无机非金属材料类专业教材编审委员会（即今全国无机非金属材料学科教学指导委员会的前身）确定选题并策划组织，由我国工艺岩石学的奠基人苏良赫教授任顾问，由长期从事显微结构研究的同济大学诸培南教授、上海科技大学翁臻培教授、华南理工大学王天頔教授任主编，由中国科学院上海硅酸盐研究所陈显求教授任主审，由十几位教授组成编委会，有十多位专家撰稿或提供图片资料，这一作者阵容足见本书的编写实力和所具备的权威性。本书于1992年被列为新闻出版系统国家级"八五"重点图书，也足见出版社对本书的重视和所做的贡献。

由于本书的作者和武汉工业大学出版社的编辑人员共同的努力，反复推敲，从修改定稿到编辑、出版，每一步都克服了许多困难，终于我们看到了这部凝聚着众多人们的共同心血和情谊的巨著将要问世了。它在材料科学发展长卷中，又将增添一幅美丽多姿的画卷，我为此感到由衷的欣喜。于是，应本书主编诸培南教授和武汉工业大学出版社曹文聪副总编辑之约，欣然命笔，成此序言，表达诚挚的祝贺，表达对为本书作过各种贡献的专家和所有的工作人员的敬意和感谢。

我愿向所有从事无机非金属材料研究、生产、教学工作的科研人员、技术人员、大专院校师生推荐本书，不仅因为它所具有的实用价值，也不仅因为它在学术界的地位，还因为在本书策划、编写和制作过程中所渗透着的奉献精神，让我们所有的无机非金属材料科学工作者，以及后来的人们，以这种奉献精神和科学态度迎接21世纪材料科学发展的新时代。

袁润章

1994年6月

编 者 的 话

《无机非金属材料显微结构图册》终于付印出版，即将问世了，在付印前夕，作为本书的编者，心中感到无比欣慰。同时想把本书在编辑出版过程中的某些情况，简略地写上几句话，让各位关心过这本书的人们和今后接触本书的读者，对此有所了解，也许并不是没有意义的。

全国无机非金属材料专业教材编审委员会是国家建材局领导下组成的一个学术性组织，该委员会于 1986 年在南京召开会议讨论确定编写教材和专著的选题，材料测试编审组即确定了本书的选题；并聘请了国内大专院校、中国科学院有关研究所、工业部有关研究院、所的多位专家组成了本书的编委会。其后，根据编写出版的进程，先后分别在洛阳、上海、宜昌、西安召开了 5 次编委会。由于大家的努力，每次会议按计划进行了预定的工作，并得到了领导的关怀。编委会和出版社亦相互配合支持。国家建材局人才开发司，根据历次会议的纪要精神，对本书的出版寄予了很高的期望，并在人力和物力上给予了很大的支持和帮助。承接本书出版工作的武汉工业大学出版社，经过申请上报手续，终于被批准，以本书作为全国“八五”重点图书出版，落实了本书出版的基本有利条件。出版社把本书的出版质量放到了首要位置上，每一步骤都严格把关。编委会除了组织专家分头撰稿外，对本书的内容安排，还不断同主审和有关专家研究，推敲调整，逐步更新，对已经撰成的有关章节初稿，认真修改补充。因此，本书在学科内容上，除了反映传统的基础和收集国内的科学成就之外，还陆续收进了当代无机材料显微结构研究的最新内容，体现了当代的先进水平，从而构成了国内第一本全面介绍无机非金属材料显微结构研究的专著，对生产控制和新材料的研制都有重要的指导意义。本书从开始策划到正式出版，历时长达 8 年，它是在领导关怀下，全体撰稿专家以及为本书提供珍贵图片资料的专家、科技工作者和出版社领导、编辑人员通力合作的成果，反映了我国无机非金属材料显微结构研究领域的水平和成就，编者在此向各位表示衷心的敬意和感谢！

其次，本书之所以能顺利出版，是同参加编写和编辑工作的各位同志的严谨作风和高尚的精神风貌分不开的。由于每一个人都认识到编写出版这本书的重要性和它的深远意义，领会到它将在推动材料科学研究与开发中所起的巨大作用，因此，大家都不计较个人名利得失，宁守清俭，甘于奉献，为促成本书的早日问世，作出不懈的努力，这在我国学术界可以称得上是一项盛举。

最后，编者还应向严东生教授为本书封面题签，扉页题词；苏良赫教授、袁润章教授为本书作序；陈显求教授为本书封面提供珍贵的显微结构图片，在此一并致以衷心的感谢！

由于经验不足，同时限于编者的学识和水平，本书中对某些问题的叙述讨论和图片的选用，可能不够深入，或者挂一漏万，文字的表达（包括中英文）也可能有不妥之处，甚至还可能存在谬误，敬希海内外专家和本书读者不吝指正。

编 者

1994 年 6 月

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MICROSTRUCTURE OF CERAMICS

INTRODUCTION

1. GENERAL ILLUSTRATION

In the progress of materials science, a great attention has been paid on inorganic materials, which in combining with metals and polymers constitute the three main pillars of modern science and technology.

In order to investigate and improve the properties and the quality of inorganic materials, various kinds of methods and techniques based on physics and chemistry have been used to solve the key problems, and a great development has been reached. It has been realized that the microstructure study is one of the important links in practice. It should be noted that different technologies can be used for making the inorganic materials with more excellent properties, however, these properties are closely related to the microstructure. For example, the formation of glass-ceramics and transparent ceramics was discovered to be dependent on the change of their microstructures, and the special properties of mica glass —ceramics which broke the limit of mechanical property of all these materials, such as glass, ceramic, metal, and polymer, was also discovered to be dependent on their particular microstructure. Another example, the ceramic based composite, toughened by zirconia, used as a part of machine which possesses outstanding properties covering high temperature resistance, thermal shock resistance, high mechanical strength and toughness is one of the new materials with special distribution of stress, caused by the presence of additives that make the phase transformation of zirconia only partially due to the control in technology.

Therefore, the microstructure study can contribute a deep influence on the inorganic materials science, it can also play an important role in the breakthrough of material properties and so should not be depressed, or even neglected.

As we have mentioned above, sometimes, microstructure may be confused with the microscopic structure of atoms. Actually, in the former, the particle size studied lies in the range above several tens or at least several atomic radii, i. e. from 1nm to several tens nm, or even as large as 0. 1mm. Generally, in each particle there should be an independent phase. Whereas in the latter the dimension of the atomic microstructure to be discussed should be limited under the size of lattice cell. According to above standard, it seems to be suitable to take the size scope as a distinction between them. But if one wishes to know the images in details, during observation of minerals, rocks and inorganic materials or their products, satisfactory results can not be obtained from the low resolution optical microscope. Along with the progress of research techniques and the development of modern instruments, they give new energetic forces to activate the microstructure study of inorganic materials. Among them the adaption of some special techniques of microscopy (such as dark field illumination method)and simultaneously the creative application of phase contrast microscope, interference microscope and interference phase contrast microscope can improve the working ability of optical microscope so greatly that it could even provide such information where the electron microscope could not yet. As a matter of fact, the application of transmission and scanning electron micros-cope have become the main method in microstructure investigation. In addition, the cooperative application of electron microprobe, various energy spectrometers, X-ray diffractometer, electron diffraction, differential thermal analysis and differential scanning calorimerty can contribute such a good effect that the understanding of the relationship between microstructure and its composition, phase as well as the property of inorganic materials reaches a new level.

It is well known that the history of microstructure development started from petrology. But due to the complexity of structure involved in the silicate system, studies can hardly approach the physicochemical level, so it is not so progressive as that in metallography. Many studies were limited in the identification of phases or the description of binding state in the structure, and so too simple to interprete the formation of rocks. In the later period, the microstructure study was treated as one of the important subject in the technological petrology research. However, the study on the formation of the phases and the microstructures in inorganic materials has not really proceeded from theory to practice systematically. So, researchers must do much more works in the interpretation and evaluation of micrographs for enhancing their study to a certain-logical level.

For approaching this purpose, the researchers started from the principles of physical chemistry, which may be possible to be applied in the micrograph interpretation. Then, according to the images, they can give a series of programs and methods based on every imaginable thinking, so as to feasibly hold the essential rules needed to use in the microstructure interpretation. It can be indicated that it is effective to use the comprehensive knowledge of physical chemistry, crystallography, mineralogy, petrology, materials technology, and various analyzing and measuring techniques as the foundation to interprete the micrographs and virtually make it clear, i. e. how the material structure formed just as the micrographs represented and which properties this material will show. It is revealed that the detail characteristics can be recognized to realize the crystal aspects and the phase transformation processes in qualitative deduction and to understand the relation between phase to phase combination from stereology.

In recent years, with the development of investigation method in stereology, more precise or quantitative determinations on materials microstructure have been established. For the same crystal with different aspects, stereology generally indicates the relation of three-dimensional growth reflecting on the two-dimensional micrograph. The arguments are derived from the process of crystal growth and influence of enviroment, and the stereology can be applied to determine the fractional weights of different phases in the specimen, so as to play an important role in developing and substantiating the content of microstructural chemistry.

As we know, the theoretical system involved in the microstructure study of inorganic materials is continuously substantiated and perfected. If we carefully creat and accumulate experiences and informations, surely, we are able to add more abundant content to the materials science.

Combining with materials science and technology, we now conclude the tasks of microstructure study as follows:

1) To evaluate their quality, according to the results obtained from inorganic materials research and the microstructures of raw materials, semi-finished products and products in the manufacturing process.

2) Through the inspection of defects in the materials or articles of products, to find out the forming reasons from the microstructure and put forward the improving and preventing treatment to control the production.

3) Though the microstructure study, to understand the corrosion mechanism with respect to the reactions among the glass or slag with refractories in kiln, in order to prolong the age of the kiln and provide the elementary criteria for selecting and using refractories.

4) Starting from the basic view poins of microstructure and physical chemistry, to study and design new materials and intermediate products (such as artificially enriched ores) in order to get the material and product with ideal microstructure and excellent properties expected.

5) Though the microstructure study of inorganic solids prepared by uncertain technology or produced from the application of some materials, to understand their forming reasons and then to rationalize their manufacturing process or to improve their usage effect as well as utilize their by-products comprehensively.

So, the inorganic materials microstructure study will develop in depth and continuously play an important role in the modern science.

2. RELATIONSHIP BETWEEN COMPOSITION, PHASE AND MICROSTRUCTURE IN THE EQUILBRIUM AND NON-EQUILBRIUM CONDITIONS

2.1 Crystallization involving in the phase equilibrium conditions and the related microstructures

Thermodynamic phase equilibrium is always the main content of materials science, but in the inorganic materials microstructure study, the question that which microstructure will appear in a certain physico-chemical equilibrium condition is always put forward.

As we know, in the preparing and manufacturing processes of in organic materials, the rate of reaction in some silicate systems is slow because the components can react only through the diffusion among solids. Perhaps, for the liquid with high viscosity, the real equilibrium state can hardly reach an expected degree in many conditions. However, phase equilibrium is still effectively applied in many practical systems (including silicate and non-silicated systems). Especially, in some special conditions, even if the system does not reach the equilibrium, the formed phases and microstructure can be still interpreted from the relation of phase equilibrium.

In order to explain the above questions, now, according to the rules with respect to the changes of microstructure involving in the transformation processes from melt to solid, some phase diagrams are summarized. The typical binary systems are given as follows. The characteristic microstructures got from different crystallization stages when the system has reached equilibrium are shown in Fig. 0-1-1 to Fig. 0-1-9. Other complicated systems can also be inferred depending on these rules.

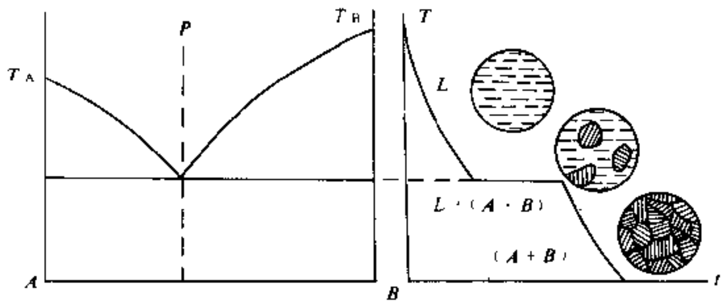

Fig. 0-1-1 Phase diagram of binary system with eutectoid and the change of microstructure for the composition of P during cooling process

2. 2 Polymorphic transformation and the related microstructure

Many phases existing in inorganic materials are characterized by the polymorphic transformation in solid states. For example, silica, alumina, zirconia, $2CaO \cdot SiO_2$, $BaTiO_3$, $Na_2O \cdot Al_2O_3 \cdot 2SiO_2$ etc. can transform in their structure under different temperatures. These processes are called under one technical term as phase transformation. It has been noted that polymorphic transformation given deep influences on the properties of materials and products, especially, it brings about volume change during the transformation. Owing to that it will also be reflected on the characteristics of microstructure, usually, one can understand through the microstructure study how the polymorphic transformations occur, and they will be proved by the morphology of phases, optical properties of crystals and the phenomenon that whether twins appear or not, and so on.

For SiO_2 system, the different products of polymorphic transformation (with mineralizer or without) are shown in the following equations:

$$\beta\text{-Quartz} \xrightleftharpoons{573℃} \alpha\text{-Quartz} \xrightleftharpoons{870℃} \alpha\text{-Tridymite}$$

(with mineralizer)

(1050℃

β-Quartz ——→ α—Cristobalite)

(without mineralizer)

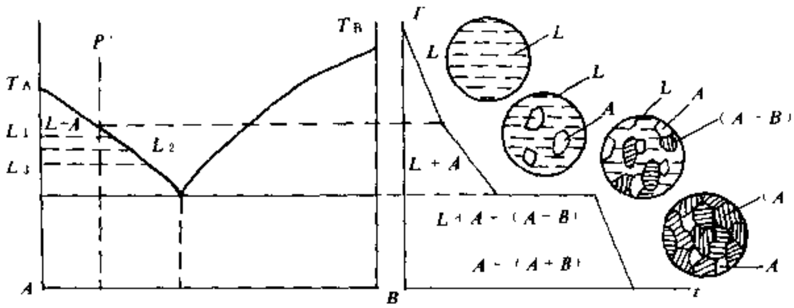

Fig. 0-1-2 The change of microstructure for the composition of P during the cooling process in the binary system with eutectoid

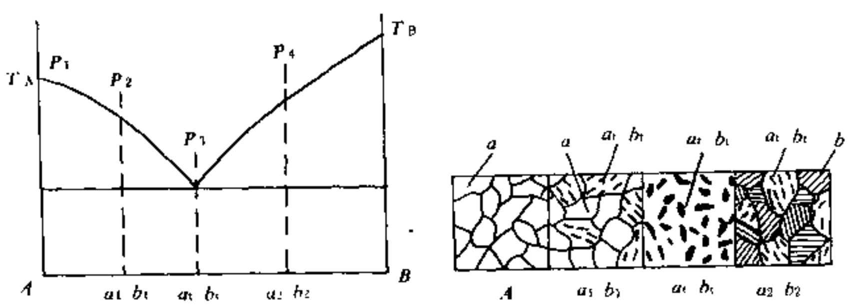

Fig. 0-1-3 Differences on the microstructure for different compositions condenced from melts in binary system with eutectoid

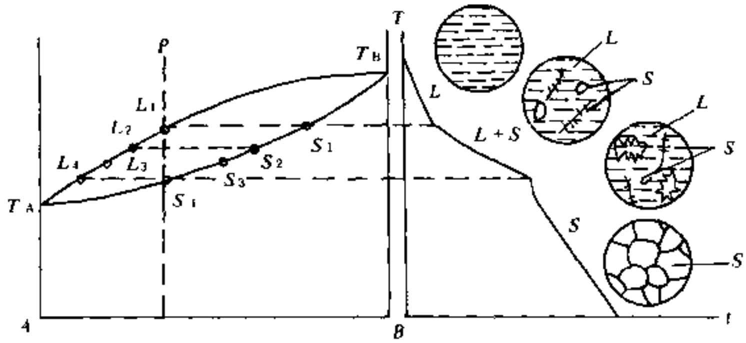

Fig. 0-1-4 Change of microstructure for the composition of P in the cooling process of binary system forming in a continuous solid solution