MODERN DEVELOPMENTS IN POWDER METALLURGY

Volume 17
Special Materials

Edited by

Edward N. Aqua • Charles I. Whitman

MODERN DEVELOPMENTS
IN
POWDER METALLURGY

Proceedings of the 1984 International Powder Metallurgy Conference, sponsored by the Metal Powder Industries Federation and the American Powder Metallurgy Institute, June 17–22, 1984, Toronto, Canada.

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FOREWORD
P/M '84 Proceedings

The seventh international powder metallurgy conference, P/M '84, sponsored by the Metal Powder Industries Federation and the American Powder Metallurgy Institute was held June 17-22, 1984 at the Sheraton Centre Hotel in Toronto, Ontario. The proceedings of this conference, Volumes 15, 16 and 17 of "Modern Developments in Powder Metallurgy," contain the reports of original research and the status of current technologies as presented in technical sessions and workshops. These papers are supplemented by plenary presentations on the status of the industry around the world.

The theme of the conference, "Sharing the World of Powder Metallurgy," was personified by the activities of more than 2,000 participants from thirty-five countries at the technical sessions, workshops, seminars and exhibition. Manuscripts were sent from five continents and were selected by a Technical Program Committee having international membership. The technical program ultimately contained 235 presentations throughout the week-long gathering.

The success of special topics focus at previous meetings led the Technical Program Committee to schedule four seminars at P/M '84. The seminar topics included Atomization Processes, Superalloys, High-Density P/M and Coatings and Hard Alloys. The lectures presented at these seminars were provided as preprints at P/M '84 and may be published as monographs and in the "International Journal of Powder Metallurgy."

An innovation at P/M '84 was the eight workshops which focused upon Research in Progress, Energy Technologies and six special topics. Authors have selectively submitted their papers for publication and most have been included in these volumes along with papers from the Technical Sessions.

The editors wish to thank the members of the Technical Program Committee for their diligence and criticism throughout the three-year period - from conception to publication.

The three volumes collect the papers in a traditional partition. Volume 15, "Principles and Processes," contains 51 technical papers on powder products, consolidation, fundamentals and applications. The papers from the Plenary Session and the report on the 1984 P/M Part-of-the-Year Winners are included. Volume 16, "Ferrous and Nonferrous Materials," contains 41 technical papers concerned with iron, copper, aluminum, titanium, nickel and cobalt materials. Volume 17, "Special Materials," contains 49 papers on refractions, carbides, steel, magnetic materials, friction and wear, and quality. The Welcoming Luncheon address and the P/M Pioneer Award presentation are also included in this volume.

Edward N. Aqua

Charles I. Whitman

PREFACE

1984 International Powder Metallurgy Conference

P/M '84 offered an unique opportunity to its participants to communicate with one another, to exchange ideas and to establish an international relationship that makes an industry grow and a technology move forward. The recording of the formal presentations in these three volumes preserves for posterity those contributions and makes them available to a still wider audience in perpetuity.

The sponsors of P/M '84, the Metal Powder Industries Federation and the American Powder Metallurgy Institute, as well as all those who were present, owe a debt of gratitude to the many individuals who served on committees; to the professional staff of the Federation and the Institute at Princeton who were responsible for all of the details and their coordination; to those companies whose contributions made the event a pleasant and memorable one; to the exhibitors who displayed the latest examples of advances in powder metallurgy technology; and, certainly, to the authors and moderators and those associated with the technical aspects of the conference. As are all Federation sponsored technical conferences, P/M '84 was conducted under the auspices of the Federation's Technical Board. We acknowledge and thank all.

Below are the names of those who expended such efforts, who devoted the time and that of their company, and gave of themselves to make P/M '84 a memorable, worthwhile and pleasant event for all who were privileged to be there:

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These companies played a major part in contributing to the success of P/M '84 by providing the special gifts and sponsoring the special events. Their generosity is greatly appreciated. These are often what makes the difference between an ordinary meeting and a special memorable and worthwhile event.

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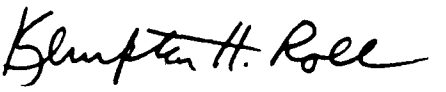
Apart from the editors of the conference proceedings whose names will forever be apparent through these volumes, Teresa F. Stillman, in coordinating the efforts of the program committee, and organizing the sessions and subsequently in conjunction with the editors in preparing the proceedings for publication, is acknowledged with appreciation. Special thanks also to Weston H. Feilbach, Jr. who undertook the task of generating a complete subject index column—an invaluable aid to researchers and others who use these volumes for reference.

Our thanks also to the editors for their arduous and meticulous labors in preparing and proofreading the manuscripts that comprise the proceedings of this conference, whose preparation and publication was the responsibility of the Federation's Publications Department.



Kempton H. Roll
Conference Chairman

Princeton, New Jersey

INTRODUCTION

VOLUME 17

Volume 17 covers in 40 papers what are normally known as "P/M Special Materials" and also covers as well high speed steels, friction and wear phenomena, electrical and magnetic materials, and finally, quality management.

Chapter One emphasizes the powder metallurgy of tungsten with papers ranging from blue oxide characteristics to properties of sintered tungsten and tungsten alloys. Information on molybdenum and zirconium based materials is also presented.

Carbides and cutting tools are covered in Chapter Two with discussions on bonding, wear resistance, and spraying of these composite materials.

High speed steels, a relative newcomer to press and sinter P/M is the subject of Chapter Three. Emphasis is on the relation between toughness and processing conditions for sintered high speed steels as well as improved materials made possible by the P/M process.

Chapter Four, in eleven papers, covers friction and wear phenomena in a wide range of P/M materials, including sintered bronze, aluminum, steel, carbides and boride based coatings.

Electrical and magnetic properties form the subject of Chapter Five with papers on capacitors, thermoelectric devices, rare earth based permanent magnets, and improved soft magnetic Fe-Si alloys.

In Chapter Six, ten papers discuss various aspects of quality management and quality techniques, an area of increased focus by the P/M industry in efforts to improve its competitive position vis a vis other fabricating methods. Information is presented on the trends toward statistical process control and fitness for use criteria in powder products and on new techniques such as quantitative image analysis to improve our understanding and control of properties.

Edward N. Aqua

Charles I. Whitman

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SPECIAL CONFERENCE PRESENTATIONS

Chapter One

Refractory Metals

FORMATION OF TUNGSTEN BLUE OXIDE AND ITS PHASE CONSTITUTION*

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ABSTRACT

By means of X-ray diffraction structure analysis, SEM observation, chemical analysis and particle specific surface analysis etc., an investigation was made in order to determine the regularity of tungsten blue oxide formation during reductional calcine process of APT. It was found that the oxygen index (OI) decreased continuously with increasing calcine temperature. The decrease rate of OI variated as the calcine atmosphere being changed, the stronger the reductivity of the atmosphere is, the more OI decreases. The deammonia-dewater process and the phase constitution variation during calcine was studied, some idea for description of phase transformation path was suggested. It was found that the most important parameter affecting phase constitution and transformation is calcine temperature. At the temperature lower than 450°C, the main formed phase was ATB, while at higher temperature, the different phase like $W_{20}O_{58}$, WO_3 etc., could be formed by different ways depending on the atmosphere reductivity. The composition and the OI of ATB are changeable. An experiment for some blue oxides reduction at low temperature was carried out. It was found that OI and the constitution of blue oxide strongly affected the particle size of the formed W-powder.

*Project Supported by the Science Fund of the Chinese Academy of Science

INTRODUCTION

It is well known that the blue oxide of tungsten has a high chemical activation, as it has a high developed specific surface and metastable structure. It is easier to reduce by hydrogen than yellow oxide (WO_3). Using blue oxide to produce W-powder can more exactly control the particle size and size distribution. This is very impotant for some types of hard metal production.

The state and the behavior of blue oxide greatly affects the properties and the quality of reduced tungsten powder. In order to control the blue oxide struture rationally, a great deal of fundamental research work has been carried out by some researchers since the sixties. For example, J. Neugebauer et al investigated ammonium-tungsten bronze (ATB)— one of the main phase constitution of blue oxide(1). Later, P. G. Dickens did some ATB structure analysis(2) and pointed out that it belongs to hexagonal crystal having a chemical composition of $(NH_4)_{0.25}WO_3$ approximately. A. K. Basu , F. R. Sale carried out a lot of research about hot decomposition of APT(3.4.5.). Especially in their recent work(6), they revealed the whole features of APT decomposion step-by-step process during ignition in nitrogen atmosphere and suggested a hot decomposition mechanism. M. Dahl studied the decomposition process of APT in different atmosphere (7), the experiment result indicates that the main phase constitution in formed blue oxide are ATB and

$WO_{2.9}$, then WO_3, at higher temperature in reductional atmosphere, $WO_{2.72}$, even WO_2 may be formed. It is worth pointing out the valuable work by Hungarian researchers(8), they took the relationship between the decrease of oxygen index (OI or ratio o/w) and the change of composition structure and the properties of blue oxide into consideration, and emphasized the great interest in searching for the best phase constitution of formed blue oxide during APT reductional calcine.

It should be indicated the industrial blue oxide of tungsten, in fact, is some sort of composition including many compounds, either its consitution or structure is changeable, it varied with different calcine conditions, the OI may be changed in the range of 2.96 to 2.75. In different companies, blue oxide structure and constitution control and the producing technology are different, depending on the requirement of the final products. A universal guidance for blue oxide production is still unavailable.

This paper is a part of earlier stage work on tungsten blue oxide investigation, trying to determine the regularity of the structure and property variation of tungsten blue oxide during reductional calcine process of APT, especially to determine the influence of the temperature and atmosphere on the structure and OI variation, and to find out the relationship between OI and the composition.

EXPERIMENT

Raw Material

APT ($5(NH_4)_2O \cdot 12WO_3 \cdot 5H_2O$) was supplied by Zhuzhou Hard Metal Co. Produced by distillate crystallizing industrally. It has an equiaxed grain belonging to monoclinic crystal system. The sample purity is better than 99.5%, the impurity content is shown in table 1.

Table 1. Impurity content of APT

Element	Pb	Sn	Bi	Sb	Fe	Co	Ni	Cr
ppm(<)	1	1	0.9	10	10	10	5	10
Element	Mn	Mg	Ti	V	Al	Cu	Si	Mo
ppm(<)	5	5	10	9	10	5	10	100

Reductional Calcine

The reductional calcine was carried out in a tube furnace, the temperature fluctuation was ±1°C, the powdery sample was put in stainless steel boats, the layer thickness was ~15 mm, the boats were pushed continuously against atmosphere flow, every boat spent ~40 min. in high temperature zone. The H_2 or (H_2 + N_2) was used for reductional atmosphere, which was purified by 5A type molecular sieve and palladium molecular sieve, the gas purity was better than 99.97% and had a dew point of -40°C. The flow rate was 50 ml./min. cm^2. The calcine temperature was chosen in the range of 350 - 600°C.

Blue Oxide Reduction

The blue oxide reduction was also carried out in tube furnace, the reduction temperature was 700 and 800°C, layer thickness was 4 mm, the boats were pushed continuously along gas flow. The reduction time at high temperature was 2h. High purified H_2 was used for reductional atmophere. The flow rate was 440 ml./ min. cm^2.

Properties Measurement

1. The weight decrease of APT after reductional calcine was measured accurately, and the weight increase of produced blue oxide after additional ignition at 700°C in air was also measured.

2. The OI of blue oxide was measured by means of chemical volumetric method, which was founded by B. A. Kiss(9), and improved by Tan et al(10). The deviation of the measurement only took place on the 3rd digid after decimal point.

3. The phase constitution was analysied by X-ray diffraction.

4. The microstructure of blue oxide was observed by SEM.

5. The powder specific surface was measured by BET.

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