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Powder Metallurgy of Superalloys

G H Gessinger

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G. H. Gessinger

BBC Brown Boveri & Co. Ltd., CH-5401 Baden, Switzerland

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Preface

In the late 1960s, powder metallurgy was almost synonymous with a low-cost manufacturing technique of mass produced precision parts. Superalloys, on the other hand, have been a somewhat mysterious subject to the powder metallurgist. Within less than five years a series of completely new technologies evolved to what has become known as powder metallurgy of superalloys. Inert gas powder atomization, hot isostatic pressing, isothermal forging, and oxide dispersion strengthening were established as important and often novel steps in superalloy processing. The main driving force for all these new developments has been to meet the demands of increased strength and temperature capability of superalloys to be used as turbine disks, and turbine blades and vanes in aircraft and industrial gas turbines.

This book summarizes the most significant developments in this field of the past 15 years. It is the first monograph dedicated to this subject, and it has been written at a moment when the technology has reached a mature stage from which further progress will come only in much smaller steps.

The basic underlying principles of superalloys produced via powder metallurgy are simple, although the variety of suggested processing routes appears to be confusing. The original processing route followed mostly in the United Kingdom has been vacuum sintering, but this route has been abandoned almost completely. The modern processing routes can be rather compared with casting and forging on a microscale, with sintering playing only a minor role. There also exists a development trend to convert directly from the liquid droplet stage into a solid compact, sidestepping the conventional consolidation techniques.

The key to a better understanding of powder production, powder consolidation and thermomechanical processing routes is the central role of microstructure and its correlation with mechanical properties. Simply speaking there exists a cross-over of mechanical properties as a function of grain size at intermediate temperatures, which can vary slightly from alloy to alloy. Below the cross-over temperature a fine grain size is favoured for optimum mechanical properties, above that temperature a coarse, often elongated grain structure is needed. This book describes ways to manipulate the microstructure and thereby also the mechanical properties.

Wherever possible, metric units have been used. Clearly, this led to some very 'accurate' figures for temperature values which had been converted from °F to °C (e.g. 649 °C for 1300 °F).

There are a large number of experimental or commercial alloys which have been developed or are in use. The designations of these alloys, their manufacturers and their chemical compositions are listed in a number of appendices at the end of the book.

In compiling this book the needs of both metallurgical and mechanical engineers and of universities were borne in mind. Thus it is expected to serve both as a useful textbook in university courses on high-temperature materials and as a reference source for those working in this field.

The book has been written in a relatively short time, which is necessary in a rapidly progressing field. That such an exercise became possible at all, is principally due to the generous attitude of Brown Boveri & Co. Ltd. in general, and to Professor A. P. Speiser, Director of Brown Boveri Corporate Research, in particular. The author, who was then Head of the Department of Physical Metallurgy at the BBC Research Centre in Baden-Dättwil, is indebted to him for letting him spend a substantial amount of time preparing the book and to spend a sabbatical leave at the National Research Council of Canada in Ottawa. I am also indebted to Dr W. Wallace, Head of the Structures and Materials Laboratory at NRC, for providing a secluded office, a helpful staff and financial assistance in the very important task of library work. I would also like to thank Dr E. Jenny, General Manager, Division Turbocharging and Central Laboratories of Brown Boveri, for his understanding and encouragement.

Many colleagues have helped me by direct contributions, by discussions or by assisting in visits. The three principal contributors to chapters in this book have done excellent jobs to maintain the general style of the book. They are: Dr W. Hoffelner and Dr R. F. Singer, BBC and Mr R. Thamburaj, National Research Council.

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Last, but not least, I would like to thank my wife, Beth, who has had to sacrifice many evenings and weekends during the past year.

January 1984

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Foreword

If one goes back far enough, for example, to the *Encyclopaedia Britannica* of 1928, then the gas turbine (jet engine) was once not considered to be viable as a machine for the propulsion of air transport. Few believed then that an aircraft could fly high enough and fast enough to make such a machine for propulsion practical. With the advent of the Whittle aircraft gas turbine engine, which energized activities in Great Britain and the United States near the end of World War II, a new era in aircraft propulsion emerged.

The era of the aircraft gas turbine engine was launched with cobalt-based alloys like Vitallium (which was derived from a dental alloy) and the alloy S 816. It was quickly realized, however, that carbide-strengthened alloys did not have the overtemperature 'recovery capability' required for prolonged durability. Therefore, although cobalt-base alloys were the materials initially chosen for gas turbine applications, subsequent alloy development work revealed that nickel-base superalloys had the desired 'recovery capability' and would predominate in gas turbine applications.

The researchers of that period, during the late 1940s and early 1950s, recognized the need to advance the science and technology of such an important application. Amongst the leaders who foresaw the future and the metallurgical needs related to high-temperature gas turbine materials were Professor Nicholas J. Grant of the Massachusetts Institute of Technology, Professor Paul J. Beck, then of the University of Notre Dame, and Dr. A. Taylor with Dr. R. W. Floyd of Great Britain who did the basic studies, especially the necessary work on phase diagrams. The concept and development of PHACOMP, by H. J. Beattie, Jr., in the 1950s and those who appreciated its significance played an important role in the chemistry control of production superalloys.

Nickel-base superalloys owe their high-temperature mechanical properties to the presence of γ' (Ni_3Al) precipitates which are coherent and remain stable to relatively high temperatures. It is important to note that through the mid-1960s alloy designers were focused on increasing the γ' volume fraction for increased high-temperature strength. γ' volume fractions of 60% are now common. Unfortunately, increasing γ' volume fractions generally brought with them the difficulty of increased macrosegregation in large ingot castings. In the extreme this made advanced superalloys, which were otherwise attractive as new turbine disk materials, virtually unforgeable.

Conceptually, powder metallurgy offers a method for overcoming the severe macrosegregation which inhibits successful ingot breakdown. Since the material is divided into small droplets while it is a homogeneous liquid, the maximum segregation distance is restricted by the size of the solidified droplets (assuming that the individual particles have the correct compositions). Early efforts using the then conventional powder metallurgy techniques to process superalloys were frustrated by the oxidation of powders during processing, which led to poor tensile and rupture ductility. The breakthrough came with inert powder processing in which the powder production, collection and densification were carried out in an inert atmosphere.

Pratt and Whitney Aircraft (USA) in cooperation with Universal Cyclops (USA), who possessed a facility for processing in a high-purity argon atmosphere, made the first attempt at inert powder processing in 1965. This early work yielded remarkable results. Gas analysis of the first powder showed less than 100 parts per million (p.p.m.) oxygen. Early superalloy vacuum metallurgy work had shown that alloys containing less than 100 p.p.m. oxygen had satisfactory tensile and creep rupture ductilities. Both microstructural studies and forging studies showed that macrosegregation had indeed been eliminated and that the alloys were forgeable. This early work provided the foundation for advanced superalloy powder metallurgy. The necessity of producing pre-alloyed powders of ever-increasing purity brought about the development of the present-day production atomization processes.

Almost concurrent with these developments in powder production was the development of the consolidation and subsequent forming of advanced superalloys by extrusion, followed by superplastic forming (GatorizingTM). Stable ultrafine grain sizes can be developed in powder-processed advanced superalloys which permit these materials to be deformed superplastically. These techniques produced the first powder metallurgy compressor and turbine disks ever operated in a jet engine, which was a consequence of the pioneering work of J. B. Moore and R. Athey and co-workers at Pratt and Whitney Aircraft. Today in just one plant of Pratt and Whitney Aircraft more than 20 000 parts have been Gatorized in the patented process that simplifies the forging of these high-strength alloys which are normally difficult to work.

Consequently, the subject of powder metallurgy superalloys is ready for comprehensive treatment and this book is dedicated to that aim.

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Foreword

Starting with a hesitant romance in the early 1960s, the marriage of superalloy technology with powder processing was consummated during the late 1970s. It must be counted among the most exciting events in the recent development of high-performance materials. To the superalloys, powder processing has brought vistas of higher property levels and better production economics; to powder metallurgy, the superalloys have become the main spur for progress in technical sophistication.

The full extent of these possibilities, however, has only been known to a small circle of devoted pioneers. Safeguarding of industrial property rights has put a blanket on the dissemination of the new ideas, and among those who could have drawn inspiration from the new achievements too few individuals were sufficiently versed in both technologies to accept the message easily.

Dr. Gessinger, himself an important contributor to the progress of superalloy powder metallurgy, is one of the very few individuals who could have produced such a book at the present time. Based on his experience in both parent fields, and with seasoned judgement of technical and economical feasibility, he has accomplished a systematic treatise which will lead the reader from the fundamentals up to the present frontier. The work not only makes a complicated new field accessible to the materials and engineering community at large, but it is also the type of book which will stimulate the imagination of many readers into ventures of their own. To them, and to the book, the best of success!

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Director,
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Stuttgart, West Germany

Contents

Foreword by F. L. VerSnyder

Foreword by H. F. Fischmeister

Part I Introduction 1

Chapter 1 Introduction 3

- 1.1. Structure and chemistry of superalloys 3
- 1.2. Historical development of superalloys 7
- 1.3. Historical development of powder metallurgical techniques in superalloys 9

Part II Pre-alloyed dispersion-free powders 17

Chapter 2 Powder production and characterization 19

- 2.1. Pre-alloyed powders with 'normal' solidification rates 19
- 2.2. Physical metallurgy of pre-alloyed powders 35
- 2.3. Alloy development considerations for pre-alloyed powders 40
- 2.4. Defect control and defect removal in pre-alloyed powders 42
- 2.5. Pre-alloyed powders with 'rapid' solidification rates (RSR powders) 49

Chapter 3 Powder consolidation methods 59

- 3.1. Cold compaction and sintering (conventional powder metallurgy) 59
- 3.2. Hot consolidation techniques 68
- 3.3. Theories of hot consolidation of powders 80
- 3.4. Forging of fully dense preforms 89
- 3.5. Direct melt–solid conversion techniques 97
- 3.6. Dynamic compaction 107

Chapter 4 Thermomechanical processing principles	112
4.1. Microstructural parameters in superalloys	112
Chapter 5 Mechanical properties of dispersoid-free P/M superalloys	132
5.1. Effect of powder microstructure on microstructure and mechanical properties of consolidated material	132
5.2. Effects of minor alloying elements on mechanical properties	134
5.3. Effects of microstructure on static mechanical properties	141
5.4. High-temperature low-cycle fatigue (HTLCF)	165
5.5. High-temperature fatigue crack growth and creep crack growth	182
Chapter 6 Quality control and non-destructive evaluation of P/M superalloys	202
6.1. Introduction	202
6.2. NDE and quality control of new components	203
6.3. Evaluation of remaining life	206
Part III Oxide-dispersion-strengthened superalloys	211
Chapter 7 Oxide-dispersion-strengthened superalloys	213
7.1. Introduction	213
7.2. Strengthening mechanisms	214
7.3. Powder production	232
7.4. Powder consolidation	237
7.5. Thermomechanical processing	241
7.6. Mechanical properties	258
7.7. Oxidation and hot corrosion	280
7.8. Development trends for ODS superalloys	286
Part IV Joining	293
Chapter 8 Joining techniques for P/M superalloys	295
8.1. Liquid-phase bonding	295
8.2. Solid-state bonding	306
8.3. Transient liquid-phase (TLP) bonding	315
8.4. Applications	317
Chapter 9 Practical applications and economic aspects of P/M superalloys	328
Appendix 1 Nominal composition of superalloys	338
Appendix 2 Registered trademarks and associated companies	342
Appendix 3 Abbreviations used in the text	343

Part I

Introduction

Introduction

Nickel–chromium, nickel–iron–chromium and, to a lesser extent, cobalt–chromium alloys are the major class of materials used for the high-temperature components of aircraft, marine and land-based power systems, and they are also utilized in applications such as hot-working tools and dies. The successful application of these alloys, commonly termed ‘superalloys’, is due to their high long-time creep strength and stability at elevated temperatures combined with their outstanding resistance to corrosion (often with protective coatings) in the aggressive environments encountered during service.

Nickel-base superalloys owe their high-temperature mechanical properties to the presence of γ' precipitates, which are coherent and remain stable to relatively high temperatures¹. Carbides are the major strengtheners in cobalt-base superalloys², though some progress has been made towards producing a stable γ' precipitate in these alloys³. The physical metallurgy principles underlying superalloy development have been described in great detail in numerous excellent reviews^{4–9}, which cover the subject more comprehensively than is possible here.

1.1. Structure and chemistry of superalloys

Since the onset of the development of modern superalloys more than 40 years ago the chemistry of these alloys has become more and more complex. The development was spurred by the need of aircraft and industrial gas turbine designers to increase the thermal efficiency of their engines by continuously raising the gas inlet temperatures and thereby also increasing the metal temperatures. The results of this development have led to alloy chemistry and processing developments with the goal of utilizing all the strengthening mechanisms known today. Superalloys are high-strength heat-resistant alloys based on Group VIIIA elements, and can generally be divided into three main groups according to the main element which forms the matrix component:

- nickel base;
- nickel–iron base and iron base;
- cobalt base.

1.1.1. Structure of nickel-base superalloys

Nickel-base superalloys with their present chemical composition are the most complex alloys; thus their development often paralleled the development of new physical metallurgical principles. Theoretical models such as Phacom¹⁰ have been used to predict the occurrence of various phases and to prevent the development of deleterious phases such as σ .

TABLE 1.1 Alloying elements in nickel-base superalloys

	<i>Element</i>	<i>Function</i>
Matrix-class elements	Co, Fe, Cr, Mo, W, V, Ti, Al	Solid-solution strengthening (Al and Cr, corrosion resistance)
γ' -phase-forming elements	Al, Ti, Nb, Ta	Precipitation strengthening
Carbide-forming elements	Cr, Mo, W, V, Nb, Ta, Ti, Hf	Reduction of grain-boundary sliding
Grain-boundary-active elements	Zr, B	Enhancement of creep strength and rupture ductility

The elements which are added to nickel can be divided into different groups depending on their contribution to strengthening and corrosion resistance (*Table 1.1*).

1.1.1.1. Matrix-class elements

The principal advantage of nickel-base alloys is their high tolerance to alloying elements and preservation of the face-centred cubic (f.c.c.) structure up to $0.8T_M$ (where T_M is the melting point) and for times up to 10^5 h. The elements contributing to solid-solution strengthening in the γ matrix are cobalt, iron, chromium, molybdenum, tungsten, vanadium, titanium and aluminium. Aluminium, mainly known as a precipitation-strengthening element, also rates as a potent solid-solution strengthener. Tungsten, molybdenum and chromium also contribute strongly, while iron, titanium, cobalt and vanadium act as weak solid-solution strengtheners. In absolute terms, solid-solution strengthening is nearly temperature independent; since the precipitation-hardening effects tend first to increase but then to decrease with higher temperatures, the relative contribution to high-temperature strength from solid-solution strengthening even increases with higher temperatures. Chromium, although a minor solid-solution-strengthening element, can still make a substantial contribution to strength if added in larger quantities. The main purpose of chromium, however, is to improve corrosion properties.

1.1.1.2. γ' -phase-forming elements

Although, as just mentioned, aluminium and titanium serve as potent solid-solution strengtheners in superalloys, their main function is to combine with nickel and in conjunction with niobium and tantalum to form the γ' precipitate, which provides the main strength to precipitation-hardened superalloys.

The γ' phase has an f.c.c. structure and a lattice constant which shows only small deviations from that of the matrix (range 0–1.5%; most alloys have a difference in lattice parameter of much less than 1.5%). Coherence between the two phases is maintained by tetragonal distortion. A consequence of this coherency is the easy homogeneous nucleation of the precipitate and, because of the low values of interfacial energy, a long-term stability at elevated temperatures.

At very high temperatures part of the precipitate is dissolved and the strengthening effect gradually decreases.

1.1.1.3. Carbide-forming elements

Carbides in nickel-base superalloys form mostly at the grain boundaries. The carbide-formers are chromium, molybdenum, tungsten, vanadium, niobium, tantalum, titanium and hafnium. The role of carbides in superalloys is far more complex and less well understood from a theoretical point of view than that of γ' precipitates. There are two competing effects on high-temperature mechanical properties: carbides dispersed at the grain boundaries have a beneficial effect on rupture strength by reducing grain-boundary sliding, while carbides of certain morphologies can have adverse effects on ductility. There is also a chemical effect due to the removal of carbide-forming elements from the matrix surrounding the grain boundary. It is the aim of alloy development to utilize carbides to improve the elevated-temperature mechanical properties and to reduce their detrimental effect on ductility by selecting suitable morphologies.

There are four basic types of carbides.

The *MC carbide* which forms first after solidification is coarse and forms both at the grain boundaries and within the matrix. Thermodynamically, HfC is the most stable compound, followed by the carbides of titanium, tantalum, niobium and vanadium. The actually observed sequence of precipitation, however, may differ from the one predicted^{11,12}. Hafnium, added in amounts of 1.5%, has a specially beneficial effect¹³. In cast alloys such as IN 713 LC the Chinese-script MC carbides are modified to a discrete particulate morphology; hafnium also partitions to the γ' phase, which exhibits a dendrite-shape morphology instead of the usual cube-like arrangement. As a consequence the grain boundaries are modified from planar interfaces to a convoluted configuration. The low-temperature ductility is improved as a result of the modified carbide morphology and the serrated grain boundary improves the creep resistance at elevated temperature. Hafnium is also added in order to improve the ease with which superalloys undergo directional solidification.

MC carbides can transform at lower temperatures to the more stable $M_{23}C_6$ carbide. $M_{23}C_6$ carbides have a significant effect on the mechanical properties of superalloys. They form discontinuous blocky grain-boundary precipitates, and they are mainly beneficial in this form because they prevent grain-boundary sliding. They can have very detrimental effects on ductility in the form of continuous brittle grain-boundary films. It should be recalled, however, that even as the blocky grain-boundary carbide that they limit ductility by either fracturing or decohesion of the carbide–matrix interface.

Cr_7C_3 usually forms as a blocky grain-boundary precipitate in chromium-lean superalloys. In more complex superalloys these carbides are unstable and transform into $M_{23}C_6$.

M_6C carbides have effects on mechanical properties similar to those produced by $M_{23}C_6$, except that they are stable at higher temperatures.

The type of carbides formed in superalloys depends upon composition, temperature and time. The MC carbides which form directly after solidification can be transformed into the lower carbides by the following metallurgical reactions:



These reactions are considered to be beneficial, and they are made use of during heat treatment to form discontinuous grain-boundary precipitates. The γ' precipitate which is another reaction product forms an envelope around the carbides, making the grain-boundary layer more ductile.

1.1.1.4. Grain-boundary-active elements

Small additions of zirconium and boron significantly enhance creep properties and rupture ductility. Although the reasons for this improvement are not entirely clear, it is believed¹⁴ that these elements, because their atomic size deviates markedly from that of the matrix, segregate to grain boundaries, filling vacancies and reducing grain-boundary diffusion.

1.1.2. Structure of nickel-iron-base superalloys^{15,16}

Precipitation-strengthened alloys containing substantial quantities of nickel and iron form a distinct class of superalloys. They include those alloys with an austenitic matrix containing 25–60% nickel and 15–60% iron, and which are hardened by the precipitates γ' , $Ni_3(Al,Ti)$, and/or γ'' , Ni_3Nb . In contrast to nickel-base superalloys, aluminium is only a minor γ' -forming element. The γ'' precipitate is unique in iron-nickel-base alloys. One important characteristic of γ'' is its slow precipitation kinetics, which explains the reduced tendency to post-weld strainage cracking.

Solid-solution strengthening elements which have been used in this class of alloys include chromium, molybdenum, tungsten, titanium, aluminium and niobium.

Types of carbides form which are similar to those in nickel-base superalloys. MC carbides can precipitate both as coarse irregular particles and as globular particles, which is associated with good ductility. $M_{23}C_6$ carbides are formed after appropriate heat treatments as globular or blocky precipitates.

1.1.3. Structure of cobalt-base superalloys

Cobalt alloys have played a very important role in the early stages of superalloy development, but have failed to match the good high-temperature mechanical properties of advanced nickel-base superalloys. Cobalt-base alloys do show advantages compared with nickel-base superalloys in hot-corrosion resistance, and they also offer good structural stability at elevated temperatures but low stresses, which makes them attractive for non-rotating applications.

The inferiority of mechanical properties can be explained by the fact that the cobalt matrix is not as stable as that of nickel. All attempts in alloy development to introduce a coherent γ' precipitate which would remain stable at elevated temperatures have failed. The coherent γ' -Co₃Ti precipitate, which can be formed when titanium is added to cobalt alloys, exhibits poor thermal stability since transformation of the matrix occurs^{3,17}.

Alloying elements used in cobalt-base alloys are nickel, chromium, tungsten, titanium, zirconium, niobium, tantalum and carbon. The principal function of nickel is to stabilize the f.c.c. matrix structure. Tungsten is the most important element added to solid-solution strengthening. The major contribution to strengthening in cobalt alloys comes from carbides. For this reason, the carbon content is quite high (0.25–1.0% compared with 0.05–0.20% in nickel-base superalloys). M₂₃C₆ carbides are the most commonly found carbides in cobalt alloys; the mentioned elements, with the exception of nickel, contribute to carbide formation.

The development of cobalt-base alloys has suffered a severe set-back because of the escalation of cobalt prices¹⁸ and the subsequent drive to find substitute elements for cobalt even in nickel-base superalloys.

1.2 Historical development of superalloys

To understand why present-day powder metallurgy (P/M) techniques have been developed for the fabrication of superalloys, a brief historical survey of the development of superalloys and, more specifically P/M superalloys, is essential.

Although nickel–chromium alloys have been known since the beginning of the century (alloy Nichrome V containing 80 wt.% nickel and 20 wt.% chromium¹⁹) serious alloy development started in the 1930s, leading to the first useful heat-resistant alloys around 1940. The major driving force behind this development was the need for improved high-temperature alloys in the first aircraft gas turbine – the British Whittle engine²⁰. The first alloys used for turbine blades were austenitic stainless steels, which soon proved to be inadequate. The first nickel-base superalloy, Nimonic 75, was derived from Nichrome V by adding 0.3% titanium and 0.1% carbon.

Alloy development continued rapidly (*Figure 1.1*) by adding increased amounts of alloying elements to utilize gradually all known strengthening mechanisms. In Britain the Nimonic series²¹ of alloys was developed in rapid succession: increased titanium contents led to the alloy Nimonic 80, while addition of aluminium gave the first γ' -hardened alloy, Nimonic 80a; in order to raise temperature capability further cobalt (Nimonic 90) and molybdenum (Nimonic 100) were added.

The first nickel-base superalloy developed in the United States was Inconel X (ref.22), which was a derivative of Inconel (15%Cr–7%Fe–78%Ni). This modification of Inconel was made by adding aluminium, titanium, niobium and carbon. This system was further developed until eventually Waspaloy evolved.

The Soviet alloy development programmes²³ differ from the US programmes in that tungsten is added because of the limited supply of molybdenum, cobalt is