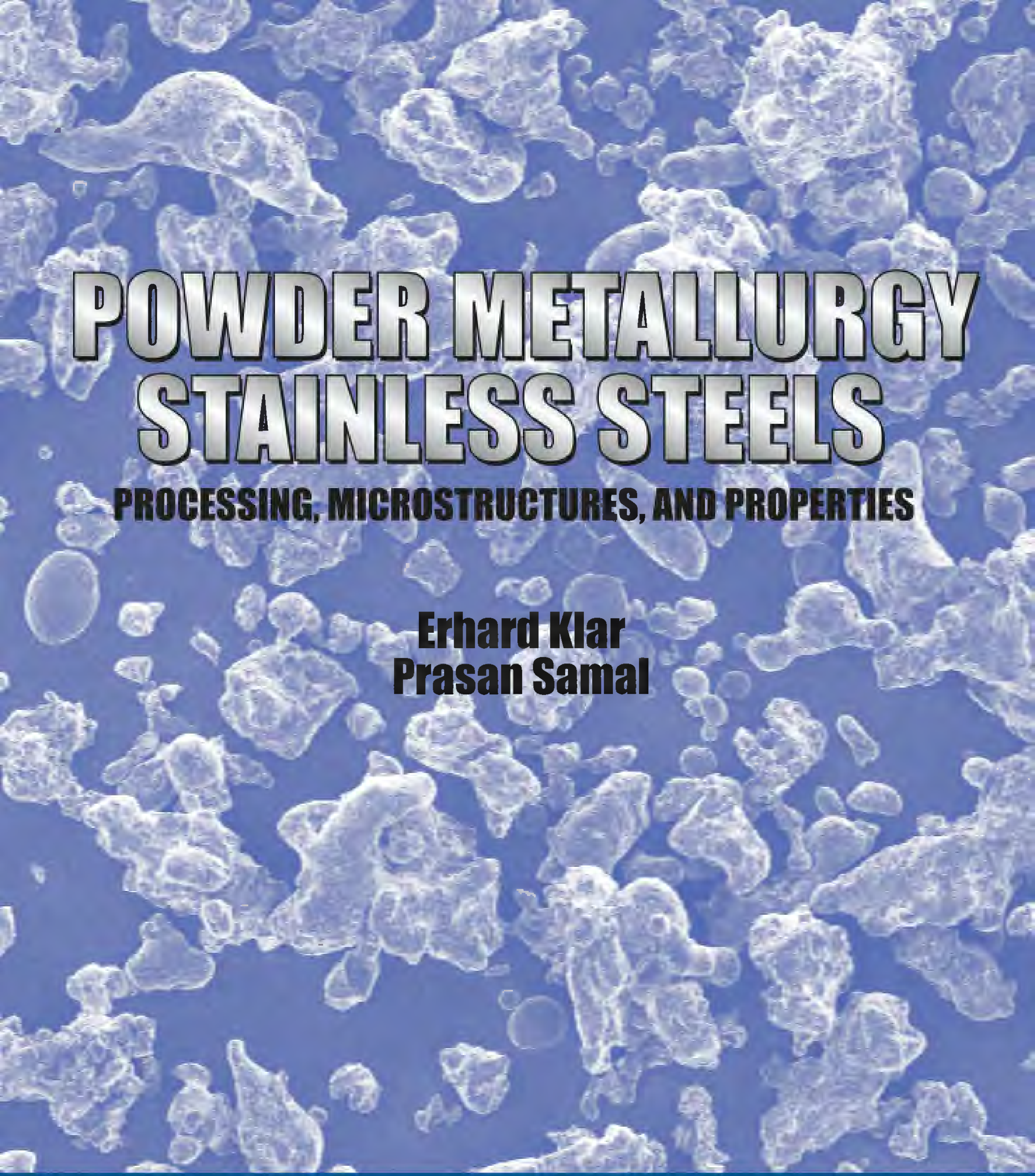

POWDER METALLURGY STAINLESS STEELS

PROCESSING, MICROSTRUCTURES, AND PROPERTIES

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Prasan Samal

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POWDER METALLURGY STAINLESS STEELS

Processing, Microstructures, and Properties

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Preface

The treatment of sintered stainless steels in this book addresses the need to more clearly understand the many factors that affect the corrosion resistance of powder metallurgy (PM) stainless steels. For over a half-century, PM technology has been an effective method of net shape processing to produce structural parts for the automotive and other industries. Conflicting literature on the factors that influence the corrosion resistance of PM stainless steels has led to some widespread misconceptions, which generally attribute poor corrosion resistance to just the presence of pores (whereby the presence of pores increases the effective surface area of a sintered part and thereby increases the corrosion rate in the passive region). A crevice-sensitive density region does exist in which a neutral chloride environment can give rise to crevice corrosion. Nonetheless, many or most cases of underperformance cited can be traced to inappropriate sintering practices that result in poor metallurgical soundness.

Recent progress in the understanding of corrosion and corrosion-resistance properties of sintered stainless steels has led to renewed interest in their application in the automotive sector for the benefit of net-shape processing and more efficient materials utilization. To obtain good corrosion-resistance properties, sintered stainless steels require careful processing, starting with powder selection, avoidance of contamination, efficient delubrication, and through to controlled sintering and cooling. There are several distinct, process-related corrosion issues with sintered stainless steels that the PM industry had to cope with over the years:

- Contamination with less noble constituents, causing galvanic corrosion
- Crevice-corrosion-prone density range in neutral saline environments
- Excessive carbon content (from various sources), causing sensitization and intergranular corrosion
- Excessive nitrogen content, due to sintering in H_2–N_2 mixtures containing large amounts of nitrogen (i.e., dissociated ammonia), in combination with slow cooling rates produces sensitization and intergranular corrosion
- Inadequate cooling after sintering, which, in the presence of excessive carbon, can cause sensitization and intergranular corrosion
- Excessive dewpoints and/or inadequate cooling causes reoxidation during cooling and susceptibility to pitting
- Surface chromium losses due to sintering in a vacuum furnace can impair the corrosion resistance of sintered stainless steels
- Pitting corrosion due to incomplete reduction of original residual oxides.

Solutions to all of these problems are at various stages of implementation in the industry. Because of this, and the fact that corrosion resistance is usually the prime property when stainless steel is selected as a material, the subject of PM stainless steel processing, and, more specifically, of optimal processing, from powder to final part pervades the entire book.

For stainless steel parts manufacturers, this book serves as a guide to making parts that possess improved corrosion-resistance properties, thereby opening new market opportunities. Although some of the aforementioned problems can also be present in wrought and cast stainless steels, some are specific to PM, and all have special PM processing-related characteristics. The general approach is first to present the phenomenological aspects of a subject, including problem areas. This is followed by a description of its underlying principles and then a discussion and illustration of available solutions. The perspectives taken are often those of a powder producer, reflecting the authors' affiliation. Significant portions of the data are from Professor Maahn and coworkers of the Technical University of Denmark, who, in a three-year effort (1990 to 1993) in cooperation with industry, made important contributions to this subject.

The structure of the book more or less follows the sequence of the production process. After a brief historical background, the chapters include metallurgical background and alloy compositions, powder manufacture and properties of powders, compaction and shaping, sintering and corrosion, optimal sintering and surface modification, with concluding chapters on mechanical and magnetic properties, corrosion-resistance testing and properties, secondary operations, and applications. Emphasis is concentrated on the press-and-sinter technology of PM, although some consideration is given to metal injection molding, powder extrusion, and hot isostatic pressing. The discussion of optimal sintering in Chapter 6, "Alloying Elements, Optimal Sintering, and Surface Modification in PM Stainless Steels," although based largely on press-and-sinter technology, is relevant, with appropriate restrictions, to other modes of PM shaping and consolidating.

Introductory books on PM and corrosion science provide a useful basis for this text, because the reader is assumed to possess a basic knowledge of metallurgy, powder metallurgy, and corrosion science. Suggested references are *Powder Metallurgy Science* by R.M. German (Ref 1); *Powder Metal Technologies and Applications*, Volume 7, *ASM Handbook*, 1998 (Ref 2); *Corrosion Engineering* by M.G. Fontana (Ref 3); *Corrosion and Corrosion Control* by H.H. Uhlig and R.W. Revie (Ref 4); and *Corrosion: Fundamentals, Testing, and Protection,* Volume 13A, *ASM Handbook*, 2003 (Ref 5). Standards on metal corrosion are found in Volume 3.02 of the *Annual Book of ASTM Standards*. Nevertheless, the authors have attempted to keep the text simple and to facilitate understanding through the use of numerous pictures, illustrations, and references. A brief glossary of definitions of powder metallurgy and corrosion terms is shown in an Appendix, with more complete versions available in ASTM standard B-243 (Ref 6) and in the aforementioned *ASM Handbook* on corrosion.

In this context, it is hoped that this work provides a contribution to the more effective processing of sintered stainless steels to achieve improved corrosion resistance and successful applications in more demanding environments. Evidence for this is presented, and the authors believe that it will be only a matter of time until the versatility of the PM process closes any gaps that still exist with wrought or cast forms. As the industry implements the solutions to the previously mentioned problems, knowledge from wrought and cast stainless steel technology can be used and applied more effectively to PM stainless steels. This then should develop into a more comprehensive use and representation of PM stainless steels within the overall field of metals technology.

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CHAPTER 1

Introduction

POWDER METALLURGY (PM) COMPONENTS produced from corrosion-resistant alloys are a growing area of PM application, of which stainless steel PM alloys span a variety of industries, including aerospace, automotive, chemical processing, medical, and recreational. Recent progress has also led to the understanding that proper processing and sintering of PM stainless steel are critically important factors in achieving corrosion resistance for increasingly demanding applications. In fact, many (if not most) cases of underperforming PM stainless steel parts in terms of corrosion resistance can be traced to metallurgical defects due to improper processing. Therefore, improved understanding of the PM stainless steel processing factors can have important results in terms of corrosion resistance and the extended use of PM technology for its well-known economic value in terms of net shape processing and more efficient material utilization in a large number of applications for the automotive and other industries.

1.1 Historical Background

In North America, laboratory and small-scale exploration of PM stainless steels in the 1930s and 1940s led to their commercial production in the late 1940s. Initially, stainless steel compositions were simply copied from known wrought stainless steels, and mixtures of elemental powders of iron, chromium, and nickel were pressed and sintered in dry hydrogen (Ref 1, 2). High sintering temperatures and uneconomically long sintering times were required to achieve full homogenization of the microstructure. The so-called sensitization method made use of sensitized stainless steel sheet, that is, stainless steel with grain boundaries depleted of chromium. When leached in acid, such sheet disintegrated into a fine powder consisting essentially of the individual grains of the stainless steel sheet (Ref 3–5). This powder actually had several promising properties and was commercially produced in the 1940s. Its compacting properties, however, were marginal.

In the late 1940s, Vanadium Alloys Steel Company began to use water atomization for making alloyed stainless steel powder. In spite of the initially high oxygen contents of these powders, they had adequate green strength and could be sintered in reasonable times. Subtle but critical modifications to wrought stainless steel compositions, as well as improvements in the atomization process itself, led to much improved powders. Even though the corrosion resistance of sintered parts was still low, it was sufficient for applications requiring only moderate corrosion resistance. The first such large-volume application was the rear-view mirror bracket in passenger cars. One of the reasons for using stainless steel in this application is the requirement that the material must match the coefficient of thermal expansion of the windshield glass.

In the early years of commercial use of sintered stainless steels, emphasis was placed on improving the compacting properties, compressibility, and green strength of stainless steel powders. Compressibility was even more important for stainless steel powders than it was for iron powders, because of the higher hardness of the former and therefore the high compacting pressures required to obtain useful green densities.

Stainless steel powder shipments (Fig. 1.1) illustrate the evolution and growth of sintered stainless steels in North America.

A low growth rate of approximately 5% in the 1970s and 1980s was due in large part to the relatively low corrosion properties of sintered

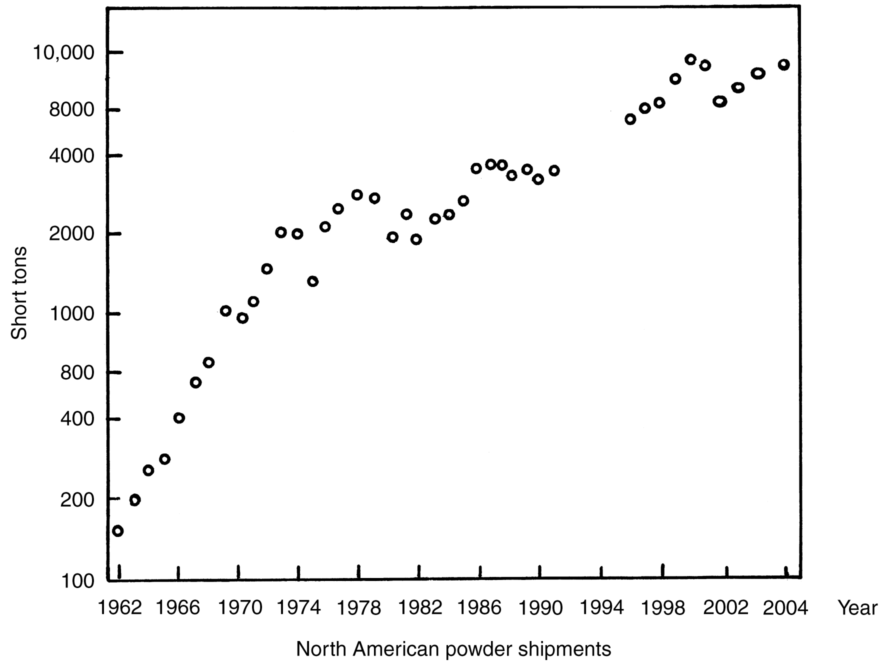

Fig. 1.1 Stainless steel powder shipments for North America. Source: Metal Powder Industries Federation. Reprinted with permission from MPIF, Metal Powder Industries Federation, Princeton, NJ

stainless steels in those years. Contributing to this lower growth were generalized conclusions and statements in the literature about the corrosion resistance of sintered stainless steels that were half-truths at best. In technical seminars presented to powder metallurgists, one of the authors summarized such statements (Table 1.1) to illustrate the problem areas.

These statements and their contradictions capture the insecure and sometimes chaotic state of the industry in those years regarding the corrosion resistance of sintered stainless steels. All of the statements in Table 1.1 reflect problem areas that, at least in part and to a varying degree, are still with us. They are dealt with in detail, including solutions, particularly in the chapters on sintering and corrosion-resistance testing and evaluation.

Table 1.1 Half-truths about the corrosion resistance of sintered stainless steels

Sintering only in hydrogen gives good corrosion resistance.
Vacuum sintering gives the best corrosion resistance.
Vacuum sintering gives poor corrosion resistance because of chromium losses.
Sintering in dissociated ammonia gives poor corrosion resistance because of the formation of chromium nitrides.
Good corrosion resistance cannot be obtained at low (1150 °C, or 2100 °F) sintering temperatures because of the lack of reduction of chromium oxides.
It is possible to obtain good corrosion resistance of parts sintered in laboratory furnaces but not in industrial furnaces.
Sintered stainless steel will always have inferior corrosion resistance because of the presence of pores that give rise to crevice corrosion.

By the mid-to-late 1980s, both stainless steel powder manufacture and the sintering processes for stainless steel parts had been improved sufficiently to qualify for the second large-volume application: antilock brake system sensor rings in cars. This application made increased demands on both corrosion resistance and magnetic properties of (ferritic) stainless steels. As is shown in the chapter on sintering, sintering conditions conducive to good magnetic characteristics are also conducive to good corrosion-resistance characteristics.

With continuing progress, and attendant with an increase in the compound growth rate of stainless steel powders to over 15%, the third automotive application followed in the 1990s: coupling flanges and sensor bosses in car exhaust systems. Requirements, in addition to general and hot exhaust gas corrosion resistance, included elevated-temperature oxidation resistance, resistance to thermal cycling, leak resistance, and weldability, as well as improved room- and elevated-temperature mechanical properties. In some respects, the PM parts were even superior to their wrought counterparts (Chapter 11, "Applications").

Even though sintered stainless steels benefit from the same economic advantages as carbon steel structural parts, namely energy efficient, environmentally acceptable, and nearly scrap-free mass production, it is clear from the aforementioned that the gradual improvement of corrosion properties over a period of 20 to 30 years had been the dominant growth characteristic for this industry. This also emerges from state-of-the-art reviews on the powder metallurgy of stainless steels by Ambs et al. (1977) (Ref 6), Dyke et al. (1983) (Ref 7), and Klar (1987) (Ref 8).

Industrial growth of metal injection molding (MIM) commenced in the 1980s with some aerospace applications. The technology is presently still in its rapid growth phase. Over half of all injection-molded metal parts are stainless steel parts; of these, 316L is the most widely used, followed by ferritic and precipitation-hardened stainless steels. In this regard, injection molding parallels the early history of conventional compacted and sintered stainless steels.

The market for MIM parts was estimated to be $150 million for North America and $360 million globally in 2005 (Ref 9). The MIM stainless steel tonnage was estimated worldwide to be 1450 metric tons in 2005 (Ref 10). Relatively low capital investment cost, in comparison to conventional press-and-sinter technology, as well as further reductions of the cost of MIM-grade powders due to economies of scale and improvements in atomizing technologies are likely to further drive the impressive growth of this technology.

1.2 Present State and Scope

As pointed out in the preface, the PM industry is in the midst of applying and implementing the fundamental requirements for optimizing the corrosion-resistance properties of sintered stainless steels for new applications. At present, several types of corrosion (i.e., stress corrosion, corrosion fatigue, erosion and cavitation corrosion, elevated-temperature oxidation) have been investigated only sporadically for sintered stainless steel PM parts, and the corrosion properties of existing commercial parts are sometimes still not as uniform and consistent as those of wrought stainless steels. Then, there also are cases where sintered, that is, porous stainless steels, exhibit corrosion-resistance properties superior to those of their wrought counterparts. Furthermore, many uses of stainless steels do not require the "full" corrosion resistance of an alloy, although the more severe corrosive applications require that the stainless steel be in the condition of its best corrosion resistance. For instance, 316L, sintered for 30 min at 1120 ºC (2048 ºF) in a 90%H_2-10%N_2 atmosphere, has been found to possess critical potentials equal to those of wrought 304 stainless steel (Ref 11). In many applications, the pitting and crevice corrosion behavior, as defined by the critical potentials of a material, is believed to describe the corrosion performance of that material.

In spite of certain limitations of sintered stainless steels on account of their porosity, optimal sintering, as described in the following chapters, will go a long way to produce sintered stainless steel parts that can satisfy many applications. Optimally sintered 317L and SS-100 (20Cr18Ni5Mo), for instance, have shown corrosion resistances in long-term immersion tests in 5% NaCl approaching or equaling those of wrought 316L (Chapter 6, "Alloying Elements, Optimal Sintering, and Surface Modification in PM Stainless Steels"). Exploiting possibilities unique to PM, such as certain kinds of surface modification (Chapter 6) or liquid-phase sintering (Chapter 5, "Sintering and Corrosion Resistance"), further extends the uses of sintered stainless steels. In neutral chloride exposure testing, optimally sintered tin-copper surface-modified stainless steels exhibit corrosion-resistance improvements of an order of magnitude over their unmodified equivalents, in addition to machinability improvements that equal or exceed (the free machinability grade) 303L. Boron-assisted liquid-phase sintering of 316L and of a higher-alloyed austenitic stainless steel (23Cr18Ni3.5Mo0.25B) has demonstrated that corrosion characteristics similar to wrought 316L are possible.

With the implementation of recent insights regarding the control of corrosion-resistance properties, that is, with "optimal" sintering, the authors believe that both sintered, that is, porous stainless steels, as well as nearly fully dense stainless steels will find many new uses. Optimal sintering (Chapter 6) is a recurring theme of this book. For practical reasons, it is defined here as control of processing and sintering that eliminates and avoids all metallurgical defects—with the exception of some residual oxides (i.e., oxides originating in the water atomization process and that, under conditions of commercial sintering, remain partially unreduced)—as well as a crevice-sensitive density region for certain alloys (Chapter 5) exposed to a neutral saline environment. Without such control, progress will be slow and the full potential of sintered stainless steels will be realized later rather than sooner. The role of residual oxygen or oxides in sintered stainless steels was one of the first observed but last addressed. It is still not entirely clear how much and/or in which circumstances residual oxides can be tolerated in regard to corrosion resistance.

The opportunities for surface modification of the porous surfaces of sintered stainless steels, quite amenable to PM processing, have not yet been fully exploited and should, possibly together with the improved control of residual oxides, lead to the elimination of crevice corrosion in neutral saline solutions for the crevice-sensitive density range.

Promising results with transient and persistent liquid-phase sintering of stainless steels will open up the entire density range to applications demanding excellent corrosion resistances, thus more broadly justifying the primary purpose of the use of stainless steels.

More PM-specific corrosion standards and more corrosion data will support these opportunities. In comparison to five families of stainless steels and 158 standard and nonstandard wrought stainless steels listed in the ASM *Metals Handbook Desk Edition*, 2nd ed., 1998, the 2007 MPIF standard 35 lists only 14 sintered stainless steel compositions comprising three families of stainless steels, and three compositions for metal injection molding.

Chapter 1: Introduction

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Chapter 3: Manufacture and Characteristics of Stainless Steel Powders

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Chapter 4: Compacting and Shaping

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Chapter 5: Sintering and Corrosion Resistance

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Chapter 6: Alloying Elements, Optimal Sintering, and Surface Modification in PM Stainless Steels

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Chapter 8: Magnetic and Physical Properties

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Chapter 9: Corrosion Testing and Performance

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Chapter 10: Secondary Operations

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Chapter 11: Applications

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