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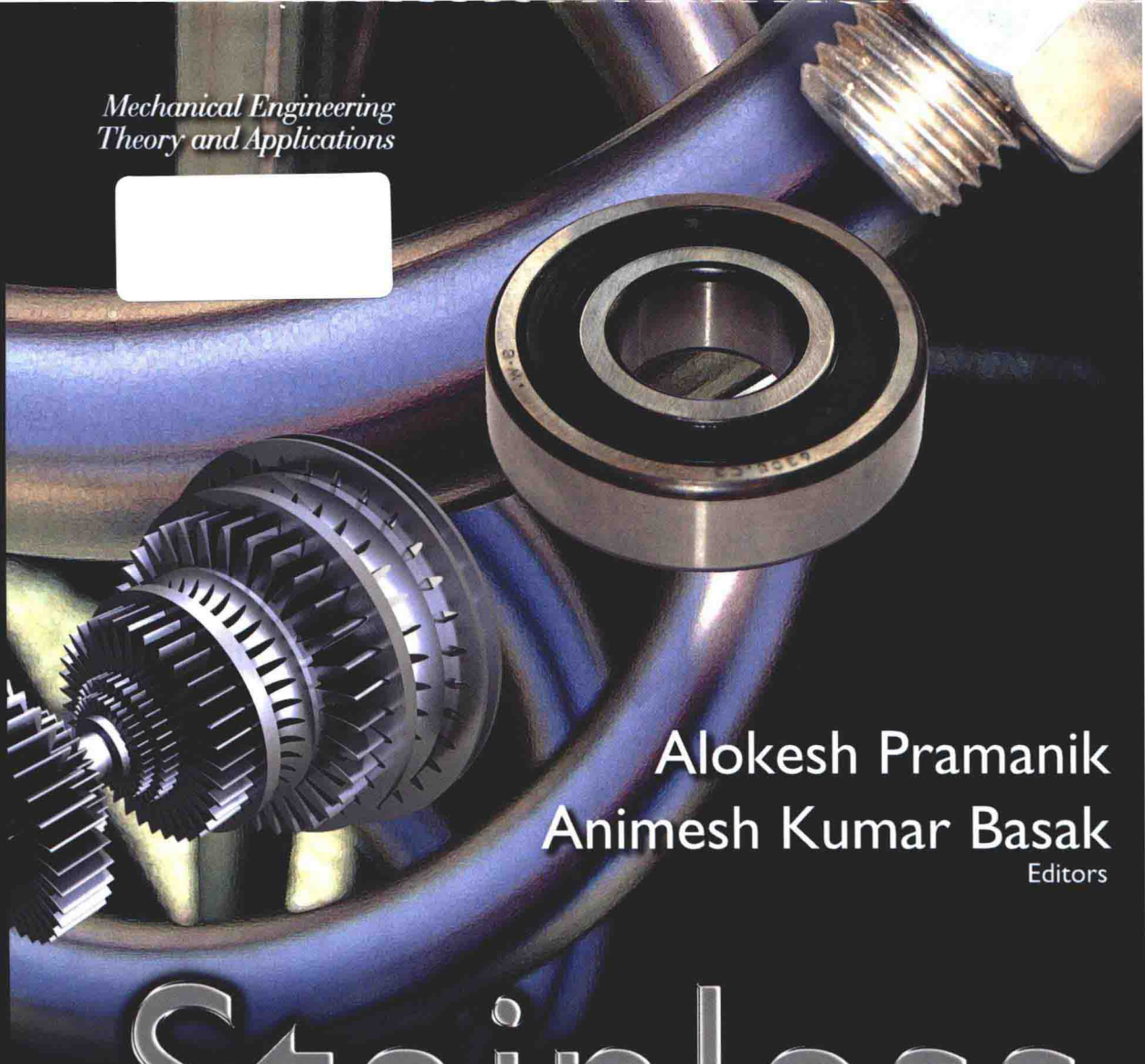

Alokesh Pramanik
Animesh Kumar Basak
Editors

Stainless Steel

Microstructure, Mechanical Properties and Methods of Application

NOVA

MECHANICAL ENGINEERING THEORY AND APPLICATIONS

STAINLESS STEEL

MICROSTRUCTURE, MECHANICAL PROPERTIES AND METHODS OF APPLICATION

ALOKESH PRAMANIK
AND
ANIMESH KUMAR BASAK
EDITORS

nova publishers
New York

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Library of Congress Cataloging-in-Publication Data

Stainless steel : microstructure, mechanical properties and methods of application / editors, Alokesh Pramanik and Animesh Kumar Basak (Department of Mechanical Engineering, Curtin University, Western Australia, and others).
pages cm. -- (Mechanical engineering theory and applications)
Includes index.
ISBN 978-1-63482-080-6 (hardcover)
1. Stainless steel. I. Pramanik, Alokesh. II. Basak, Animesh Kumar.
TA479.S7S676 2015
669'.142--dc23

2015002242

Published by Nova Science Publishers, Inc. † New York

MECHANICAL ENGINEERING THEORY AND APPLICATIONS

STAINLESS STEEL

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PREFACE

Stainless steel, termed as 'miracle metal' is all around us in numerous applications ranging from everyday household items to sophisticated biomedical applications. In earlier days, stainless steel was used to produce the finest, the most durable products that money can buy at that time and was only accessible by the upper class of the society. With time, both the production and the application of stainless steel increase as well as its availability to wider people. The unique combination of corrosion resistance and retention of strength in a range of temperatures, from cryogenic to high temperature, expand the dramatic increase in the use of stainless steel. The discoveries of the stainless steel date back in early 1900's and still in high demand around the industries. Subsequent research on composition, structure properties and role of alloying elements leads to the discovery of various categories of stainless steel suitable for specific applications. Upon its discovery, the next significant development of stainless steel occurs at mid 1900's by the development of argon-oxygen decarburization process which leads to the nitrogen alloyed and duplex grade stainless tell. As a result of such significant development and worldwide demand, stainless steel is one of fastest growing segments in metal industries. Taking those facts into accounts, the present initiative 'Stainless Steel: Microstructure, Mechanical Properties and Methods of Application' is an attempt to compile the recent developments in the properties, applications and further processing of stainless steel by various surface treatment processes and recent research trends. The present book include the high-tech characterization techniques of stainless steel to address the decomposition behaviour; decomposition –induced transformation to correlated the property-microstructure; powder metallurgy to produce difficult to cast stainless steel components, depassivation/repassivation behaviour, surface treatment as well as wide range of machining techniques to address the need the of machinibility of stainless steel.

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September 2014

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In: Stainless Steel
Editors: A. Pramanik and A. K. Basak

ISBN: 978-1-63482-080-6
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Chapter 1

AUSTENITE INSTABILITY AND PRECIPITATION BEHAVIOR OF HIGH NITROGEN STAINLESS STEELS

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ABSTRACT

Nitrogen is a strong austenite stabilizer and improves the mechanical properties and the corrosion resistance of austenitic stainless steels. For these reasons, nitrogen addition to steel has been extensively studied in the last 40 years. This chapter presents a literature review since 1926, when a pioneer study on the effects of nitrogen in iron-based alloys was reported. The maximum solubility of nitrogen in stainless steels is high, although it decreases considerably at temperatures below 1000°C. Therefore, depending on the composition, high nitrogen austenitic stainless steels can undergo different phase transformations during exposure to temperatures ranging between 500°C and 1000°C. Continuous and discontinuous precipitations of chromium nitride, as well as ferrite and sigma phase formation, have been observed and, as a result of these phase transformations, a loss of toughness and lower corrosion resistance are frequently detected. The aim of this chapter is to present and discuss the austenite stability and

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illustrate the precipitation behavior of high nitrogen austenitic stainless steels. Three different high nitrogen stainless steels are used in order to highlight stabilization/ destabilization effects and microstructural features associated with the presence/depletion of nitrogen. The nucleation of discontinuous precipitation of chromium nitride and its growth kinetics are discussed and transformation models are presented.

Keywords: High nitrogen steels, austenite stability, chromium nitrides precipitation, ferrite and sigma phase formation

INTRODUCTION

Austenitic stainless steels present an excellent combination of corrosion resistance, ductility, toughness and weldability, and account for about 70% of the world's stainless steel production. The almost centenary AISI 304 (German V2A) and AISI 316 are still the most consumed types of stainless steel [1, 2]. However, the strength level of these common types, particularly the yield strength (YS) in the annealed condition, is relatively low, around 200 MPa [1, 3]. Another important type of stainless steels is the duplex (ferritic-austenitic microstructure) stainless steel, which is employed where higher yield strength (YS), better pitting corrosion and, intergranular corrosion resistance are necessary [4]. However, duplex stainless steels are extremely susceptible to alpha prime (α') formation at temperatures ranging from 300°C to 500°C and to sigma phase formation between 550°C and 950°C. These phases are highly deleterious to the toughness and corrosion resistance of the duplex steels and their formation must be avoided during heat treatment, welding or application [4, 5].

High levels of nitrogen addition stabilize austenite and can increase significantly its YS as well as enhance other properties [6-8]. According to Speidel, 0.4 wt.% is a high nitrogen content for austenitic stainless steels [9], although the levels for high nitrogen content remain debatable [8]. A pioneer investigation on the effect of nitrogen addition to iron-chromium alloys was reported in 1926 by Adcock [10]. A few years later, it was verified that nitrogen was a very strong austenite stabilizer and its effect was about 20 times stronger than that caused by nickel, as showed in Tofaute's work [11]. As a result, the effect of nitrogen was included in a modified Schaeffler diagram for welding processes [12] by DeLong [13] and Espy [14]. Nitrogen addition to duplex (ferritic-austenitic) steels also causes an enlargement of the austenitic field, with possibility of their full austenizatization [15-22]. Additionally, nitrogen improves fatigue and creep resistance [23-30] and inhibits strain-induced martensite formation [25, 31, 32, 33].

The study of the effects of nitrogen in stainless steels showed a significant enhancement during the decade of 1980 and was followed by the development of new or modified processes [8, 15, 21, 34-37]. Nitrogen addition can be carried out both in liquid and solid state and overviews of nitrogen addition technologies have been presented by Simmons [34] and, more recently, by Lo [8]. Therefore, nickel replacement by nitrogen, and in some instances by manganese, became an interesting alternative to retain or improve the mechanical properties and corrosion resistance of stainless steels [6, 22, 32, 33, 38-43].

The benefits of nitrogen in stainless steels occur mainly when it is in interstitial sites in solid solution [6, 8, 25, 34, 44-53] as first observed by Krivobok [44]. The precipitation behavior of chromium nitride in high nitrogen stainless steels presents peculiarities that make

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its evaluation important from the scientific and technological point of views [15, 22, 34, 52, 54-56]. In addition, other phase transformations can occur after chromium nitride precipitation, such as ferrite and sigma phase formation in the nitrogen-depleted regions [16, 17, 20, 21, 39, 53]. The aim of this chapter is to address austenite stability and the precipitation of chromium nitride in high nitrogen stainless steels. For the sake of clarity the discussion about nitrogen effects will be illustrated for three different stainless steels, and will focus on the different phase transformation mechanisms, namely on the nucleation and growth of chromium nitride, on the precipitation kinetics, and on the consequences of the precipitation for the matrix stability.

HIGH NITROGEN STEELS DESCRIPTION AND CHARACTERIZATION

The austenite stability and the precipitation behavior presented in this chapter will be discussed for three different high nitrogen stainless steels, namely, (i) a completely austenitic steel, (ii) a functionally graded steel with an austenitic nitrogen-rich outer region enclosing a duplex (ferritic-austenitic) core and (iii) a nickel-free high-manganese duplex (ferritic-austenitic) stainless steel with a casting microstructure, as described hereafter:

(i) Standard type duplex stainless steel DIN W.-Nr.1.4460 in which nitrogen was added by pressurized electroslag remelting [15]. The result was fully austenitic steel in the form of a forged bar with the following composition (wt.%): 25.1Cr, 1.9Mo, 5.5Ni, 1.5Si, 1.20Mn, 0.03C and 0.87N (designated as AN steel).

(ii) Standard type duplex stainless steel DIN W.-Nr.1.4462 to which nitrogen was added by solid solution nitriding [18, 21]. The result was an austenitic stainless steel case near the surface and a duplex steel in the inner region. The sample was a plate with a thickness of 2.5 mm and the following composition (wt.%): 22.04Cr, 2.9Mo, 5.5Ni, 0.47Si, 1.70Mn, 0.025C, with the nitrogen varying in austenite from about 0.7 on the surface of the sample to 0.4 wt.% in the inner part [18, 21] (designated as ADN steel).

(iii) Nickel-free duplex stainless steel (ferritic-austenitic microstructure) to which 0.54 wt.% nitrogen was added [38, 39]. The resulting steel was received in as cast condition with the following composition (wt.%): 25.8Cr, 0.11Ni, 1.12Si, 17.2 Mn, 0.035C and, 0.54N (designated as DN steel).

The microstructural characterization was carried out by several complementary techniques, such as optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy-dispersive spectroscopy (EDS), wavelength-dispersive spectroscopy (WDS) and X-ray diffraction (XRD), as well as Vickers hardness and magnetic measurements. The metallographic sample preparation was described in previous works [20, 21, 39], however, it is worth reporting the composition of V2A-Beize, a valuable chemical etchant for stainless steels, which was used in the metallographic preparation of the investigated steels prior to optical and scanning electron microscopy observations: 100 ml of HCl, 100 ml of distilled water, 100 ml of HNO_3 and 0.3 ml of Vogel's Sparbeize. Crystal structures and lattice parameters of the predominant phases have been determined by XRD

using CuKα1 radiation. The parameters employed in the hardness measurements can be found elsewhere [20, 21, 39]. The amount of ferromagnetic phases was determined using the magnetic induction method (Fisher Permascope® M11D with a 0.1% ferrite detection limit).

SOLUTION ANNEALING AND AGING HEAT TREATMENTS

Austenite or ferrite-austenite (duplex) solid solutions (free of precipitates) were obtained for each steel using specific heat treatments. Solution annealing of the AN and DN steels was carried out, respectively, at 1250°C for 1 hour and at 1050°C for 2 hours, whereas a solid solution nitriding process was used for the ADN steel. This treatment involved heating for 5 hours at 1200°C in N_2 partial pressures of 2 bar in order to obtain a nitrogen-rich layer at the surface. The samples were water quenched after annealing for nitrogen to remain in solid solution. The subsequent aging treatments were carried out at temperatures ranging from 600°C to 1100°C, for times between 1.5 and 6000 minutes, and were followed by water quenching. Throughout the text austenite will be designated by A or γ, ferrite by F or α, sigma phase by S or σ and duplex regions by D or α + γ.

NITROGEN SOLUBILITY

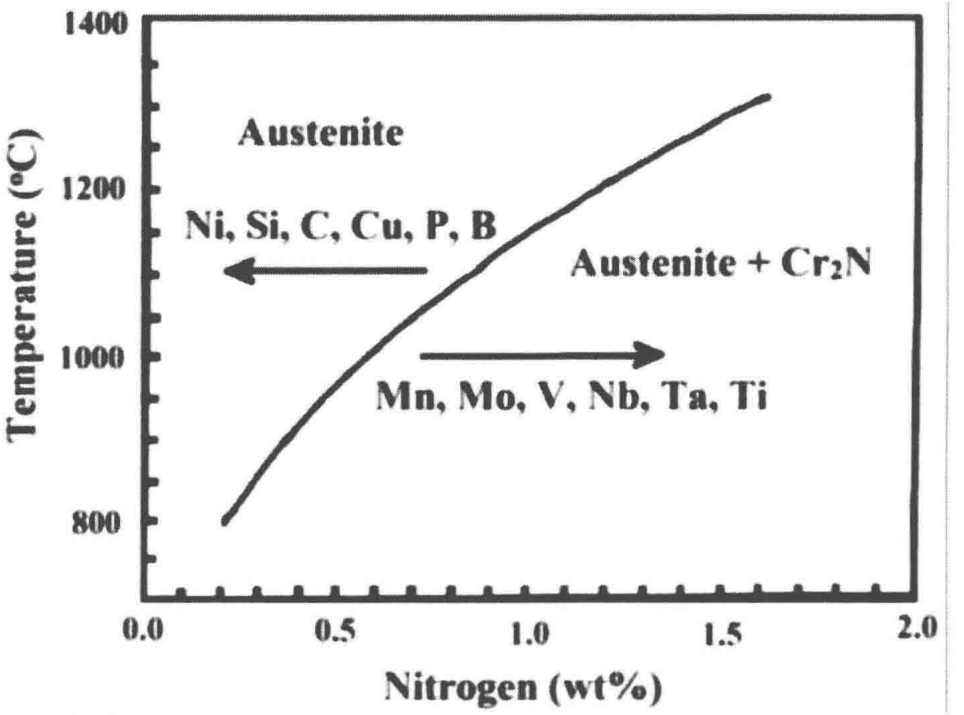
Nitrogen solubility in iron and steels is strongly dependent on the chemical composition and on the crystal structure(s) of the alloy [57], in addition to temperature and pressure [54, 58]. As a starting point we will summarize the known solubility behavior for the Fe-Cr-Mn and Fe-Cr-Ni systems, which to a first approximation can be considered to include the AN, ADN and DN steels. The effect of alloying elements on the nitrogen solubility for the Fe-Cr-Mn system is shown in Figure 1, whereas Figure 2 shows the influence of crystal structure for the Fe-Cr-Ni system.

The AN and the ADN steels had their nitrogen solubility increased since the nitriding treatments were carried under pressure. Indeed some technological processes are carried out at high pressures to promote the introduction of higher quantities of nitrogen into the steel [15, 18, 58]. The mass percentage of nitrogen in the alloy (C_0) and its relation with the N_2 pressure (P_{N2}) follows Sievert Law [15]:

$$C_0 \propto \sqrt{P_{N2}} \tag{1}$$

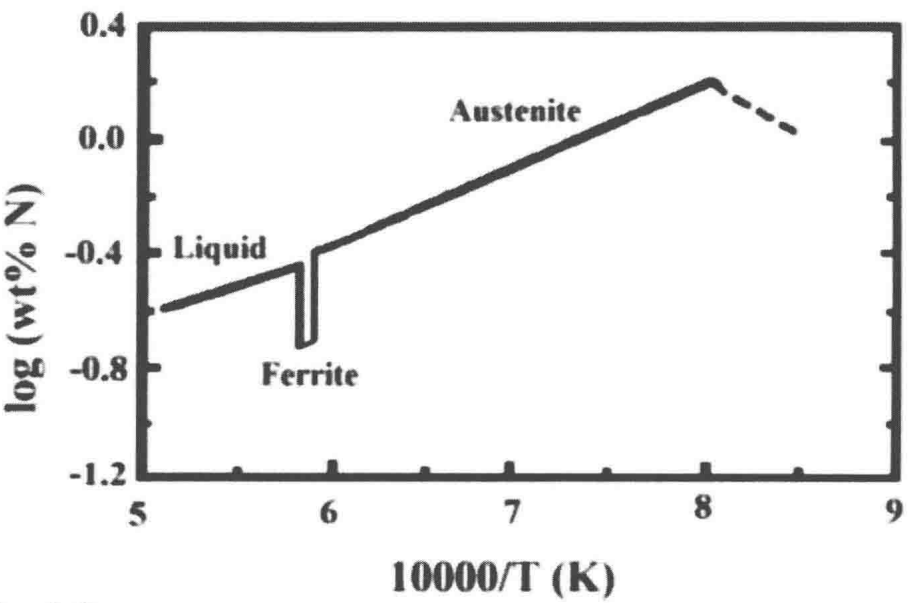
which is typically used at pressures of about 1 atm. However, there has been evidence that this law can also hold for higher pressures, i.e., $C_0(P \neq 1) = C_0(P_{N2})$ [54, 59-61] and equation (1) can be considered more general [15]. As a result of the high nitrogen content in the AN and ADN steels, complete dissolution of nitrogen was achieved only at high temperatures.

In the AN steel, the chromium nitride precipitation occurs up to 1050°C, therefore solution annealing treatments have to be carried out at higher temperatures. On the other hand, the stability domain of ferrite starts at temperatures as low as 1300°C, and annealing at higher temperatures results in duplex microstructures.



Adapted from [54].

Figure 1. Influence of alloying elements and temperature on Cr_2N precipitation in the Fe-Cr-Mn-N system.



Adapted from [54].

Figure 2. Influence of crystal structure on the nitrogen solubility for alloys of the Fe-Cr-Ni system at 0.9579 atm of N_2.

Annealing at 1250°C represents the best compromise to achieve complete dissolution of nitrides and simultaneously guarantee the absence of ferrite and minimize grain coarsening [16, 17, 20]. Figure 3 shows the X-ray diffraction pattern of the AN steel after solution annealing.

Figures 4 and 5 show, respectively, a nitrogen profile across an ADN sample and the transition microstructure between the austenitic and the duplex regions formed after the solid solution nitriding process.

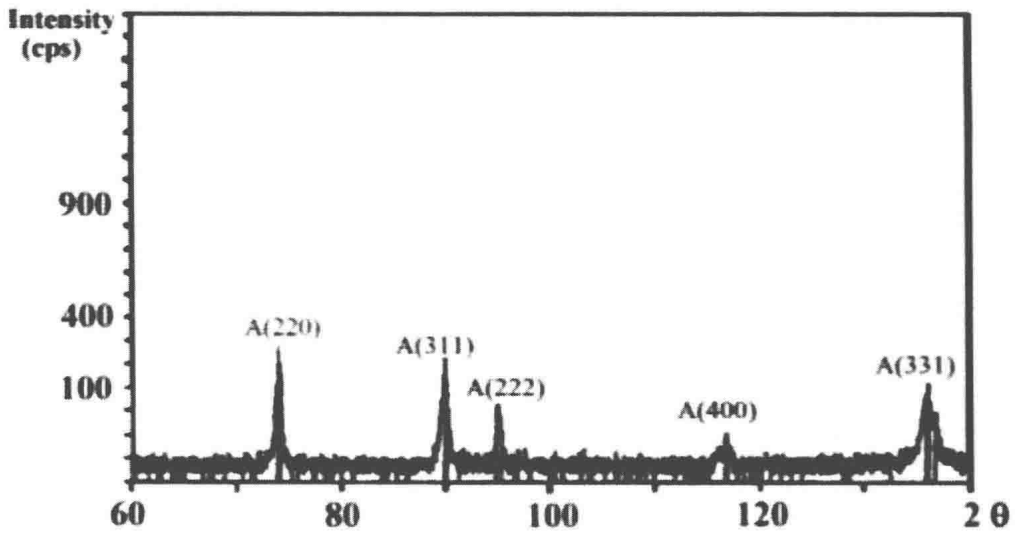
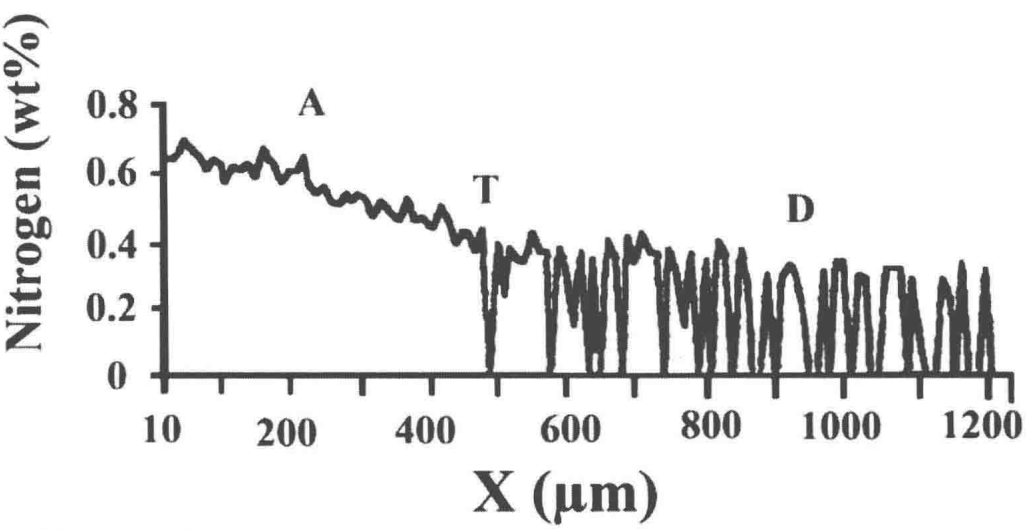


Figure 3. X-ray diffraction pattern (CuKα1 radiation) of AN steel after the solution annealing at 1250°C for 1 hour followed by water quenched.



Adapted from [17, 18, 21].

Figure 4. Nitrogen concentration profile (WDS measurements) of the ADN steel case after the solid solution nitriding process at 1200°C for 5 hours and at 2 bar N_2 pressure. T is the transition region between the austenitic region and the duplex (ferritic-austenitic) region. X is the distance from the surface.

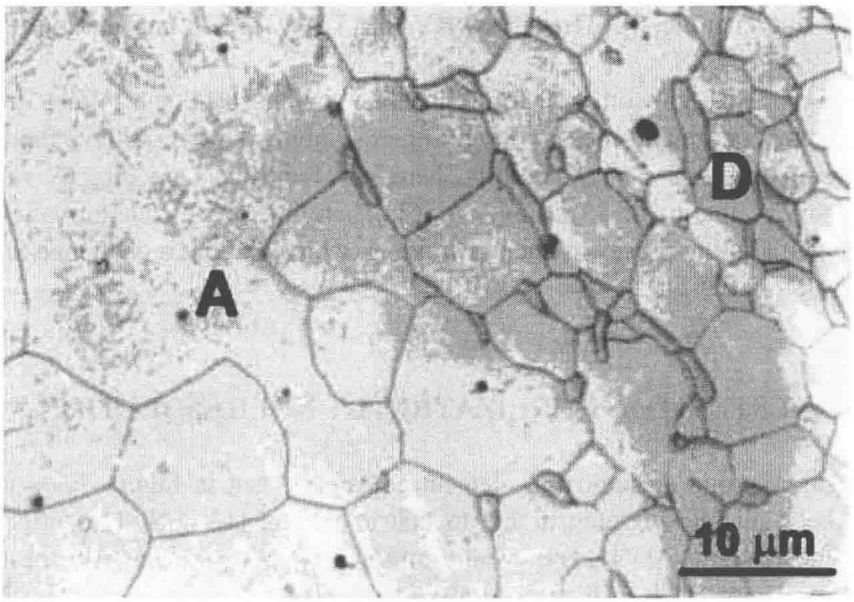


Figure 5. Microstructure (Optical microscopy) of the transition between the austenitic region (nitrogen-rich) and the duplex region of the ADN steel after the solid solution nitriding process at 1200°C and 2 bar of N_2 for 5 hours.

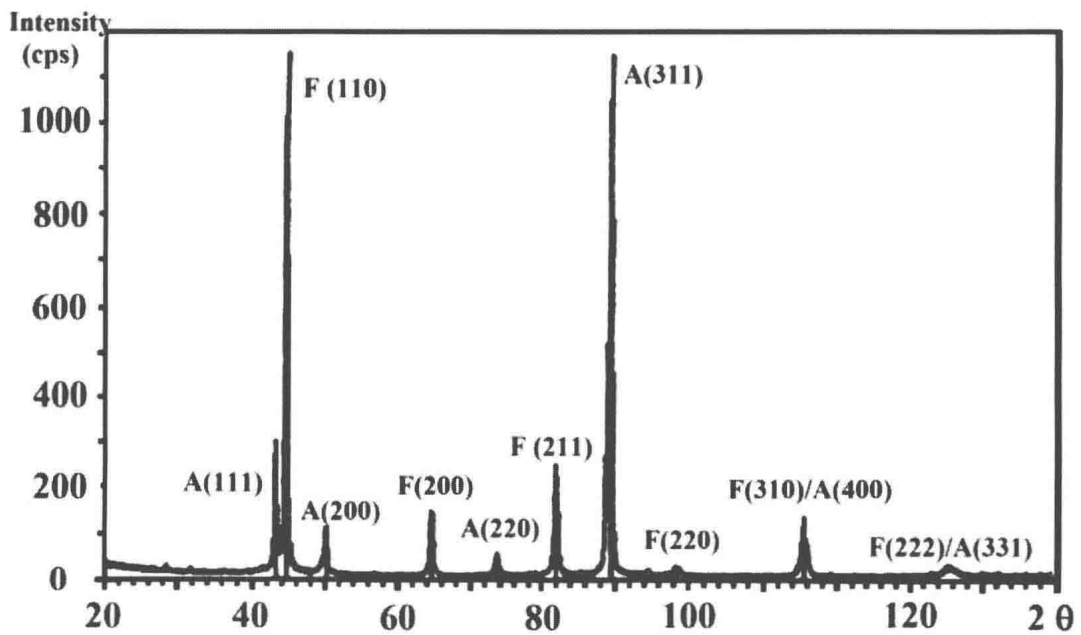


Figure 6. X-ray diffraction pattern (CuKα1 radiation) of the DN steel after solution annealing at 1050°C for 2 hours followed by water quenched.

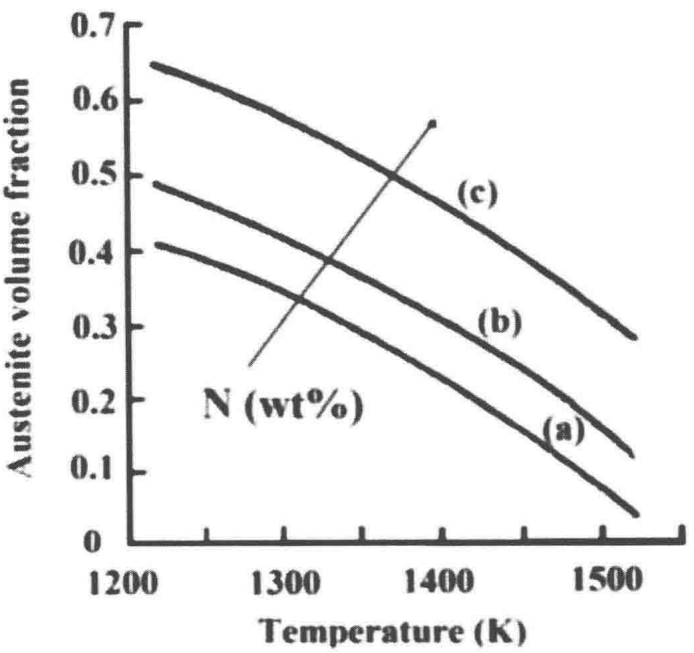
The high levels of chromium and manganese of the DN steel increased the nitrogen solubility up to 0.5 wt.% [38, 39] and the standard solid solution annealing (1050 °C for 2 hours) resulted in a duplex ferritic-austenitic microstructure free of precipitates as shown by the X-ray diffraction pattern presented in Figure 6. The volume fraction of ferrite measured by magnetic induction was 47% (the balance was austenite). After the solid solution

treatment, the microhardness of ferrite was HV (0.2 kg) 274 ± 20 and that of austenite was HV (0.2 kg) 290 ± 16 [39].

The above discussion on solution annealing emphasized the effect of chemical composition, temperature and pressure on nitrogen solubility. In fact, this is the cornerstone of high nitrogen stainless steels, since the benefits of nitrogen are directly related to its presence in solid solution [6, 8, 25, 34, 52-56]. Therefore phase transformations must be avoided in manufacturing processes such as welding, which remains a challenge that demands further research [6, 50, 52, 53].

AUSTENITE STABILIZATION BY SOLID SOLUTION

Nitrogen is a strong austenitizing element as can be seen in Figure 7. Its influence in terms of nickel equivalent is comparable to that of carbon [13, 14]. This effect is clearly observed in the AN and ADN steels, where nitrogen leads to a change in the equilibrium phase diagram. These steels, which present an initial ferritic-austenitic microstructure, are fully transformed into austenite after nitrogen addition.



Adapted from [63].

Figure 7. Volume fraction of austenite in the Fe-Cr-Ni system as a function of temperature and nitrogen content. (a) Corresponds to 0.05, (b) to 0.10 and (c) to 0.20 wt.% nitrogen.

It was demonstrated that the addition of 0.7 wt.% of nitrogen is enough to produce a fully austenitic microstructure in both cases [17, 21, 62]. This result is in agreement with the Espy diagram [14] which can be used on a first approximation to evaluate the volume fraction of the phases present at room temperature. In the case of DN steel the high chromium content and the replacement of nickel by manganese does not stabilize austenite completely and a duplex microstructure is present.

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