Contributions of Surface Engineering to Modern Manufacturing and Remanufacturing

3rd International Conference on Surface Engineering October 10-13, 2002 Chengdu

By
Editor-in-chief Siwei Zhang
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Southwest Jiaotong University Press Chengdu, P. R. China

图书在版编目(CIP)数据

表面工程对现代制造及再制造业的贡献:第三届表面工程国际会议论文集/张嗣伟主编.一成都:西南交通大学出版社,2002.10

ISBN 7-81057-661-5

I. 表... II. 张... III. 金属表面保护 - 国际学术会议 - 文集 - 英文 IV. TG17-53

中国版本图书馆 CIP 数据核字(2002)第 065574号

第三届表面工程国际会议论文集
3rd International Conference on Surface Engineering表面工程对现代制造及再制造业的贡献
Contributions of Surface Engineering to
Modern Manufacturing and Remanufacturing

张嗣伟 主编

出版人 宋绍南 责任编辑 祁素玲 封面设计 肖 勤

西南交通大学出版社出版发行

(成都市二环路北一段 111 号 邮政编码: 610031 发行科电话: 87600564)

http://press.swjtu.edu.cn E-mail: cbsxx@swjtu.edu.cn 西南冶金地质印刷厂印刷

开本: 880mm×1230mm 1/16 印张: 38.75 字数: 1188 千字 印数: 1—600 册 2002 年 10 月第 1 版 2002 年 10 月第 1 次印刷 ISBN 7-81057-661-5/TG・293

定价: 180.00 元

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Preface

Along with the rapid development of science and technology on a global scale, the Surface Engineering is growing fast and is becoming even more important than it has been in the past. However, it will face the new developing opportunities and severe challenges, such as environment, energy and sustainability. As one of the series of major surface engineering international conferences in China, the 3rd International Conference on Surface Engineering is just held in Chengdu, a famous historic city in China, against this background. The main theme of the conference is a major contribution towards surface engineering to the modern manufacturing and remanufacturing. The major aim of this conference is facilitating the exchange of expert knowledge and experiences from all over the world and attempting to emphasize the bridge between research and application.

This set of conference proceedings contains valuable contributions by many experts in the surface engineering profession. There are altogether 111 selected papers and 17 invited lectures in this volume, of which 59 papers are from about 15 countries in the world and 69 papers from China. They fall essentially into the following topics, according to which the technical sessions will be mainly conducted:

- Thermal Spraying Technology
- Electrodeposition and Electroless Deposition Technology
- Vapour deposition technology
- Diffusion treatment technology
- High energy beam modifications
- Surface engineering in biomaterials
- Nano-surface engineering and nanotribology
- Tribology

These lectures and papers reflect the recent achievements of research and development in the areas of surface engineering, the high quality of which will further promote the development of this science and technology.

Some members of the Program Committee helped greatly to review the technical papers and to offer valuable suggestions. To them, I want to express my heartfelt gratitude.

Wishing every success to the conference and to all participants.

Editor-in-Chief

Zhang Si-Wei, Professor

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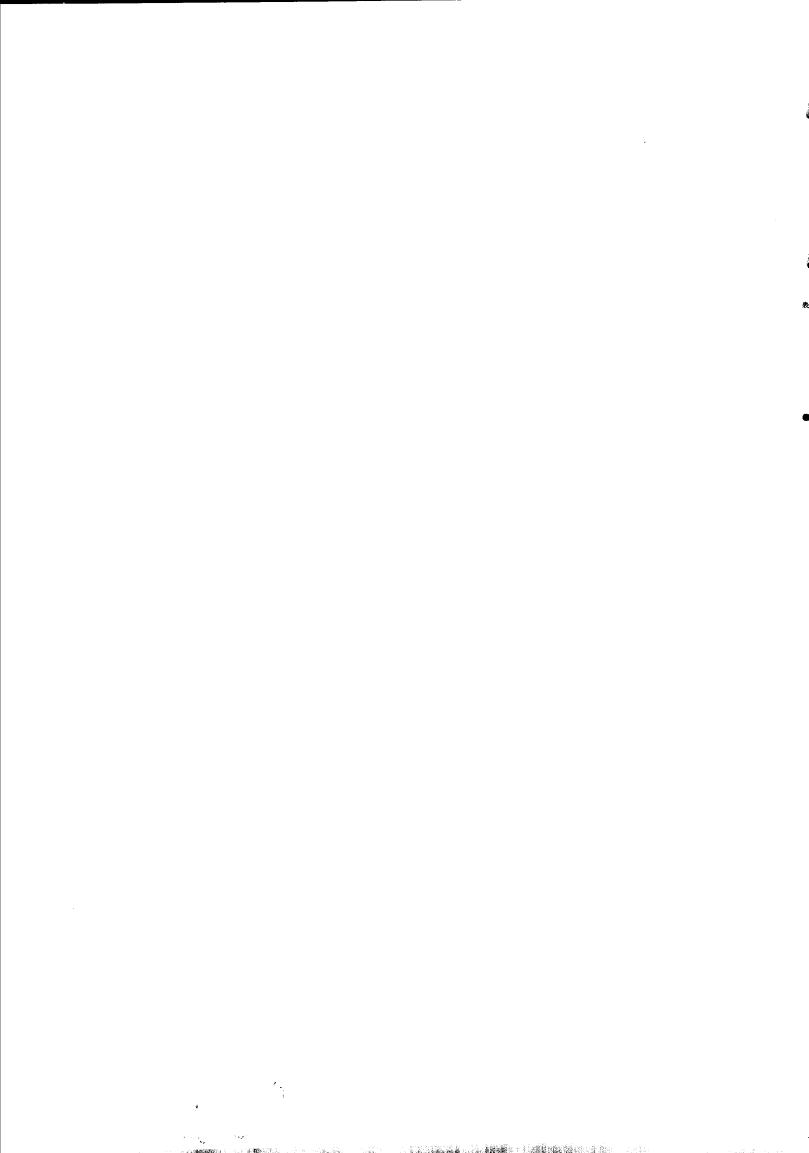
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Invited Papers



Low Temperature Plasma Nitriding and Carburising of Austenitic Stainless Steel for Combined Wear, Corrosion and Fatigue Properties

T Bell, X Li, H Dong

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Abstract

Austenitic stainless steels are attractive materials for various industrial sectors to combat environmental and corrosive attack. However, the inherent poor tribological behaviour (in terms of high friction and low wear resistance) has been the main barrier to their wider applications under corrosion-wear conditions. Therefore, it has long been a dream for surface engineering researchers to develop novel surface engineering technologies to effectively enhance the wear resistance of stainless steel surfaces without loss of their attractive "stainless" characteristics. This has been highlighted by the discovery of low temperature plasma nitriding phenomenon in the mid 1980's, where a new phase with a high hardness and good corrosion resistance could be produced on austenitic stainless steel surfaces. Since then great research and development have been made towards combined improvements in wear, corrosion and fatigue properties. This has been demonstrated by various low temperature surface alloying processes (including plasma, ion beam, gaseous and salt bath methods) and increasing numbers of academic publications, reflecting rapidly expanding niche markets in the food, chemical, nuclear and medical sectors.

In this paper, the historical evolution and development of low temperature thermochemical surface alloying processes are overviewed; the research and development of low temperature surface alloying of austenitic stainless steels towards combined improvements in wear, corrosion and fatigue properties are assessed; finally new directions and key areas for future development are identified and discussed.

Keywords: austenitic stainless steel, carburise, corrosion and fatigue properties

Introduction

Austenitic stainless steels are attractive materials for various industrial sectors to combat environmental and corrosive attack. However, the inherent poor tribological behaviour (in terms of high friction and low wear resistance) has been the main barrier to their wider applications under corrosion-wear conditions. As early as 1971 the poor wear resistance of austenitic stainless steels was recognised and investigated by Jean-Paul Lebrun for his PhD at the University of Nancy [1] Various attempts have been made in the past decades to modify the surfaces of stainless steels so as to improve their surface hardness and tribological properties. Thermochemical surface engineering, such as nitriding and nitrocarburising, have proved to significantly increase the surface hardness and wear resistance of austenitic stainless steels. However, conventional thermochemical surface treatment (T > 550°C) is generally characterised by the precipitation of chromium nitrides, which leads to the depletion of chromium in the austenitic solid solution and thus the material loses its "stainless" features [2, 3]. Therefore, it has thus become imperative to develop novel technologies that can effectively improve the wear resistance of stainless steel surfaces without adversely effecting the corrosion resistance.

Great research and development efforts have been made during the past 15 years in this direction. These have been highlighted by the discovery of a low temperature nitriding phenomenon in the mid 1980s and plasma carburising recently, where a new phase with a high hardness and good corrosion resistance could be produced on austenitic stainless steel surfaces ^[4, 5, 6, 7]. Significant progress has been made by a number of researchers all over the world by using various techniques,

including plasma, ion beam, gaseous, and salt bath methods. On this topic, an International Current Status Seminar was successfully held in 2001 in Japan, and as the fruit of the seminar, a newly published book reflects the enormous strides which have been made in the last few years in the development of low temperature thermochemical processing of austenitic stainless steels [8].

The present paper discusses various aspects of these two novel plasma processes for austenitic stainless steels, in terms of processing characteristics, structural characterisation, mechanical and chemical property evaluation. The industrial applications of this class of surface engineering technology in diverse sectors are also discussed.

The material used for the investigation was AISI 316 austenitic stainless steel. The chemical composition of the material is 0.06 wt% C, 19.23 wt% Cr, 11.26 wt% Ni, 2.67 wt% Mo, 1.86 wt% Mn, balance Fe. Cylindrical discs, 6~8 mm in thickness, were cut from hot rolled bars of 25mm diameter. The specimens were then manually ground using SiC grinding papers down to 1 200 grade to achieve a fine finish. Plasma carburising was carried out using a 60 kW Klöchner DC plasma treatment unit at about 570 V with a current density of about 30A/m². Various treatment parameters were practised in order to optimise the innovative process. After completing the process, the samples were cooled in the carburising atmosphere until the temperature was below 60°C.

The chemical composition of samples were analysed using a LECO GDS-750 QDP glow discharge spectroscopy (GDS). The optical metallographic microstructures of plasma nitrided specimens were revealed using a 25%HNO₃, 50%HCl and 25%HO₂ etchant. X-ray diffraction (XRD) was carried out on all plasma treated samples for phase identification using a Phillips

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X-ray X'Pert diffractometer with Cu- $K\alpha$ radiation. Microstructure and chemical composition were examined using a JEOL 4000FX TEM. The hardness and depth of the modified layer were measured in the sectioned samples using a Leitz microhardness tester. The electrochemical behaviour of the carburised specimens was investigated employing a DC anodic potentiodynamic polarisation technique. The electrolyte used in the test was a 3.5%NaCl solution in deionised water.

Low Temperature Plasma Nitriding

Low temperature plasma nitriding was first investigated by Zhang and Bell ^[9] in the mid of 1980s and independently studied by Ichii et al ^[5] in the same period. They discovered that when plasma nitriding of AISI 304 or 316 stainless steels at temperatures around 400°C, a nitrided layer up to 20 µm thick could be produced on the sample surface, which had not only a high hardness, but also good corrosion resistance. Since then, great efforts have been made to further investigate the low temperature nitriding phenomenon of austenitic stainless steels, aiming at a better understanding of the nitrided structures and the optimisation of the process for high quality layer production ^[10-12].

Plasma Nitriding Process

Basically, the low temperature plasma nitriding is carried out in a similar way as the conventional nitriding except that the processing temperature is lower and the time sufficiently short. However, there are considerable differences with regard to the microstructures and thus the properties of a low temperature and a high temperature nitrided layers.

Fig. 1 compares the microstructures of plasma nitrided AISI 316 steel produced at 400°C, 550°C for 20 hours in a 500 Pa gas

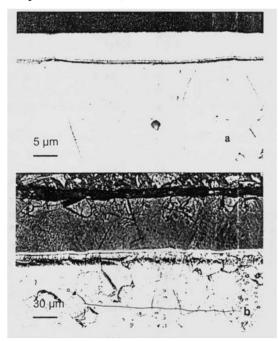


Fig. 1 Optical micrographs showing nitrided layers produced on AISI 316 steel at (a) 400°C and (b) 550°C for 20 hours

mixture of 25%N₂+75%H₂. It can be seen that the low temperature nitrided layer (400°C) is resistant to the etching reagent, such that it appears "bright" under an optical microscope,

Fig. 1a. On the other hand, the higher temperature nitrided layer (550°C) is heavily attacked and appears "dark", Fig. 1b. Comprehensive investigations with regard to the nature, morphology and properties of the nitrided layers, have shown that the "bright" layer produced at lower temperature is precipitation free; whilst the "dark" phases are associated with the precipitation of chromium nitrides. It has been established that once the precipitation of chromium nitrides occurred and the "dark" phase appeared, the corrosion resistance of the steel would become deteriorated.

It is thus important in nitriding practice to select optimised nitriding conditions to avoid the formation of the "dark" phases, thus maintaining corrosion resistance, and at the same time, to produce a relatively thick nitrided layer, thus ensuring the improvement of the tribological properties. Experimental results have shown that the transition from precipitation-free to precipitation occurrence relies very much on the processing temperature. For a specific processing temperature, there exists a critical time. Fig. 2 shows the experimentally established threshold T — t curves for AISI 316 austenitic stainless steel. When the processing temperature and time are so selected that they lie just below the curve, a precipitation-free layer with the highest thickness will be produced [6, 13]. The threshold T — t curve therefore has practical importance in the process control of nitriding austenitic stainless steel to achieve combined wear and corrosion resistance.

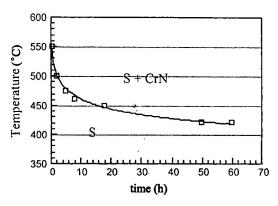


Fig. 2 Threshold Temperature (T) — time (t) curves for the three austenitic stainless steels

Structure of Low Temperature Plasma Nitrided Layer

A variety of analytical techniques have been employed to characterise the structures of the low temperature nitrided layer. It is now generally accepted that the "bright" layer produced at low temperature, as shown in Fig. 1 a, is a single phase which is free from nitride precipitation. However, the nature of this single phase layer has been subject to controversy ever since it was first discovered.

Initially, the 'bright' layer was regarded as a γ -Fe₄N compound layer, since some of its X-ray diffraction peaks appeared at reflection angles close to those from γ nitride [15]. Later, Ichii et al [14] carried out XRD analysis on a series of low temperature plasma nitrided layers. They concluded that the crystal structure of the nitrided layers could not be identified by the X-ray diffraction peaks according to the ASTM X-ray Diffraction Index, and gave the name of S-phase to this unidentified structure. The term "S phase" is now frequently

used to refer to the low temperature nitrided, and recently the low temperature carburised layer formed on the austenitic stainless steel surface.

Fig. 3 shows the typical X-ray diffraction patterns of low temperature plasma nitrided and untreated austenitic stainless steel AISI 316. Two well-defined peaks of the nitrided layer, indicated as S1 and S2, appear at lower angles than the substrate face-centred cubic austenite (111) and (200) peaks, suggesting that the single phase (S-phase) has a larger lattice parameter than the substrate austenite. The S phase was thus referred to as an expanded austenite, and the expansion has been caused by the supersaturation of nitrogen in austenite [15, 16]. The S phase was also thought to be a metastable and nitrogen supersaturated solid solution with a disordered face centred cubic (fcc) structure. This was based on the fact that the positions of the Xray diffraction peaks of S phase deviate from the ideal positions for a face centred cubic lattice [17]. Some investigators also suggested that the low temperature nitrided layer was corresponding to a body-centred tetragonal (bct) structure [18].

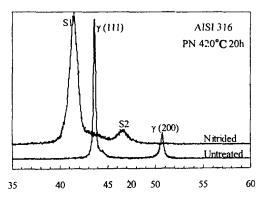


Fig. 3 XRD patterns of untreated and nitrided AISI 316 steel, showing the two broad peaks, S1 and S2, generated from low temperature nitrided layer

A more detailed study has been conducted recently to characterise the low temperature nitrided layers by XRD and TEM analysis [13, 19]. Several observations have been made regarding the nature of the S phase. Firstly, the S phase indeed has a face centred structure like the original austenite, but its lattice is distorted to such an extent that it no longer exhibits a cubic, but rather a tetragonal structure. The S phase therefore has a face centred tetragonal (fct) crystal structure, which is metastable [20, 21]. Secondly, a very high compressive residual stress is built up in the nitrided layer, which accounts in part for the observed lattice distortion of the S phase [13]. Thirdly, the metastable nature of the S phase determines that the nitriding and the service temperatures must be sufficiently low to ensure S-phase formation and to avoid its decomposition to the more stable and equilibrium phase, i.e. austenite and nitrides [20]. Fourthly, the lattice parameter of the S phase lies between 0.365 nm and 0.390 nm, which is up to 8% larger than that of austenite and the cell rise varies with nitrogen content [19]. These structural characteristics of S-phase determine that the low temperature nitrided stainless steels have some superior properties which cannot be compared to either by untreated or conventionally higher temperature nitrided steels, as discussed below.

Properties of Low Temperature Plasma Nitrided Layers

The formation of nitrogen S-phase produced a significant

hardening effect on the austenitic stainless steel surface. Typically, the micro-hardness on the treated surface can be as higher than 1000 HV, while it is no more than 250 HV on the untreated surface. As a result, the wear resistance of the stainless steel can be significantly improved by the low temperature plasma nitriding process. Fig. 4 shows that, under dry sliding conditions, the wear volume of the nitrided specimens is more than two orders of magnitude less than that of the untreated specimen. Regardless of the difference in treatment temperature and thus in layer thickness, low temperature nitriding provides improved wear resistance which is comparable to or even higher than the higher temperature nitriding [22].

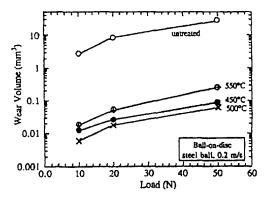


Fig. 4 Wear volume as a function of applied load for AISI 316 specimens sliding against a steel slider for a distance of 1 200 m

More importantly, the low temperature nitrided layer has a corrosion resistance as good as or better than the austenitic stainless steel substrate [4, 6, 10, 11]. As already shown, the low temperature alloyed layer is resistant to etching by the reagent normally used to reveal the microstructural features of austenitic stainless steel, so that it appears "bright" under a microscope, Fig. 1 a. Electro-chemical corrosion tests in NaCl solutions indicated that low temperature nitriding produced a layer that did not suffer from pitting corrosion even at a very high potential; whilst the untreated steel suffered from pitting corrosion as indicated by a sudden increase in corrosion current density at potentials above a critical value. In contrast, nitriding at higher temperature (above 500°C) resulted in a significant increase in corrosion current density, suggesting a deteriorated corrosion property in the testing solutions [3, 10, 23]. The crosssection micrographs after electro-chemical test are given in Fig.5. It can be seen that some deep pits were evident for the untreated sample. No pits were presented in the surface layer of the 450°C plasma nitrided sample. However, large and deep pits were present in the 550°C nitrided sample, which penetrated throughout the whole nitrided layer and were stopped at the case/substrate interface.

Due to the combined enhancement in hardness and corrosion resistance, the low temperature nitrided layer exhibits much improved wear resistance not only under dry and other non-corrosive environments, but also under severe corrosive environments. Fig. 6 compares the wear curves for various AISI 316 specimens by pin-on-disc tests in a corrosive solution of 10% HCl + 5% HNO₃ + 85% H₂O. It can be seen that in the tested solution, the specimens nitrided at 500°C and 550°C did not improve the corrosive wear resistance. Low temperature nitriding, on the other hand, significantly improved the corrosive wear resistance of the steel. Surface and subsurface

examination of the worn specimens revealed that material lost from the high temperature nitrided specimens was mainly due to corrosion, whilst that from the untreated specimen was due to metallic wear [24].

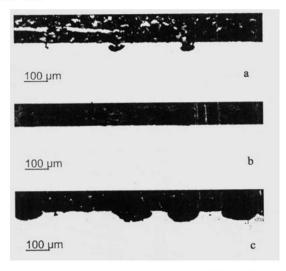


Fig. 5 Cross-section view of (a) untreated, (b) 450°C/20h nitrided and (c) 550°C/20h nitrided surface after electro-chemical tests.

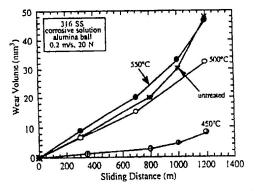


Fig. 6 Wear curves generated during pin-on-disc sliding testing in a 10%HCl + 5%HNO₃ + 85%H₂O solution

Apart from the improvement in wear and corrosion resistance, the fatigue properties of the austenitic stainless steels can be substantially improved after low temperature nitriding. This is mainly due to the formation of a hardened layer which delays the fatigue crack initiation, and the introduction of compressive residual stress which reduces the fatigue crack propagation rate [25, 26]. As an example, Fig. 7 shows the plain fatigue and fretting fatigue S-N curves of AISI 316 steel before and after plasma nitriding at 400°C for 15 hours. Fatigue tests were carried out under rotating bending conditions, and fretting was achieved by a specially designed assembly which allows two contact pads (316 steel) to be clamped symmetrically on the sample surface during fatigue tests. It can be seen that plasma nitriding at a temperature as low as 400°C improves the plain fatigue strength (at 10⁷ cycles) of the steel by 25%, but its effect on fretting fatigue is even more significant. At the high cycle fatigue (HCF) region, the fretting fatigue strength of the nitrided steel is 2 to 3 times greater than that for the untreated material. Whilst at low cycle fatigue (LCF), there is little to distinguish between the fretting fatigue curve of the nitrided samples and that of the as-received samples under plain fatigue conditions, indicating the deleterious effect of fretting at LCF region has been eliminated by the low temperature plasma nitriding employed. These results regarding the effectiveness of low temperature plasma nitriding in improving both plain fatigue and fretting behaviour of austenitic stainless steel have important implications for industrial applications where fatigue loading and/or fretting are involved.

Low Temperature Plasma Carburising

The low temperature plasma nitrided austenitic stainless steels perform satisfactorily in many industrial applications. However, there are several technical problems associated with the plasma nitriding technique, a major one being a very thin asnitrided S-phase layer ^[27]. Great efforts have been made in the past with the aim to increase the layer thickness. However, due to the metastable nature of the nitrogen S phase and the constrains imposed on processing temperature and time, it is rather difficult to produce a precipitation-free layer thicker than 20 µm. The thin, hard nitrided layer with an abrupt interface is expected to have a relatively low load bearing capacity and limited resistance to impact.

Recently, it has been found that some of the problems with nitriding can be eliminated if carbon, rather than nitrogen, is used as a major alloying species during the plasma surface engineering process ^[17]. This has led to the development of a low temperature plasma carburising process, which is carried out at temperatures between 300°C and 550°C to facilitate the diffusion of carbon into the stainless steel surface, forming a carbon S-phase layer ^[28]. The process characteristics, the structure of the carbon S-phase layer, and the properties of the plasma carburised austenitic stainless steel are discussed below.

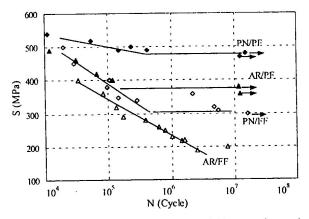


Fig. 7 Plain fatigue (PF) and fretting fatigue (FF) S-N curves of as-received (AR) and plasma nitrided (PN) 316 steel. PN at 400°C for 15 hours

Low Temperature Carburising Process

The processing cycle in low temperature plasma carburising is similar to that in plasma nitriding, but a carbon bearing gas such as a mixture of methane and hydrogen, is used in carburising. Under the glow discharge of a plasma, activated carbon species are generated and then transferred to the austenitic stainless steel surface and diffused into the material, forming a carbon-enriched layer. When the processing temperature is high, chromium carbides will form in the carburised layer, as in the situation with conventional carburising of high alloy steels [29], and the corrosion characteristics of stainless steels will be damaged. However, when the processing temperature is sufficiently low, carbide