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Towards commercialisation of fast gaseous nitrocarburising of stainless steel

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Abstract

A novel method for fast and versatile low temperature nitrocarburising of stainless steel has recently been invented by the present authors. Selected results obtained with this new surface hardening process are presented. It is shown that it is possible to obtain a case thickness of 20 µm on austenitic AISI 316 within a process cycle time of 90 minutes, and a case thickness of 35 µm on martensitic AISI 420 within a process cycle time of 75 minutes.

1. Introduction

Low temperature surface hardening of stainless steel has hitherto been associated with various inherent problems. The native oxide layer on stainless steel, which imparts favourable corrosion properties, is impenetrable for nitrogen and carbon atoms. Furthermore, in order to avoid impairing the corrosion properties of the stainless steel, incorporation of nitrogen and/or carbon has to be carried out at temperatures where development of chromium nitrides or carbides does not occur, i.e. formation of a N/C solid solution called expanite [1]. Typically this temperature is below approximately 500°C for carbon and 450°C for nitrogen. The impenetrable oxide layer on the stainless steel necessitates an activation step, i.e. removal of the oxide before incorporation of nitrogen and/or carbon is possible. Plasma and ion-based methods have been used extensively for low temperature hardening of stainless steel, as an inherent part of the process involves sputtering and hence removal of the oxide layer. Methods involving salt-bath techniques, plating of metallic surface layers and activation with gaseous halogen containing compounds have also been applied.

A new *approach* to gaseous surface hardening has very recently been developed by the present authors, which enables nitriding, carburizing and nitrocarburising of stainless steel. The present paper deals with a process variant which enables fast low temperature nitrocarburising of stainless steel. Details about the process are not disclosed herein, but emphasis lies on the capabilities of this new process. In the following selected examples of fast nitrocarburising of stainless steel are presented.

2. Nitrocarburising procedure

All nitrocarburising experiments were carried out in a horizontal laboratory tube furnace equipped with 3 heating zones with the possibility of relatively fast air cooling by removal of the furnace. In the following only the cycle time is given, all other process parameters are omitted for obvious reasons.

3. Fast nitrocarburising of stainless steel – selected results

Micrographs of low temperature nitrocarburised stainless steel are given in Fig. 1. The treated austenitic stainless steel AISI 316 has an approximately 20 µm thick zone of expanite. The expanite surface zone consists of both nitrogen and carbon, where nitrogen is located at the outermost surface (this is somewhat difficult to see in the micrograph, but is indicated by the white arrows). The hardness measured at the surface (not in a cross section) is higher than 1300 HV.

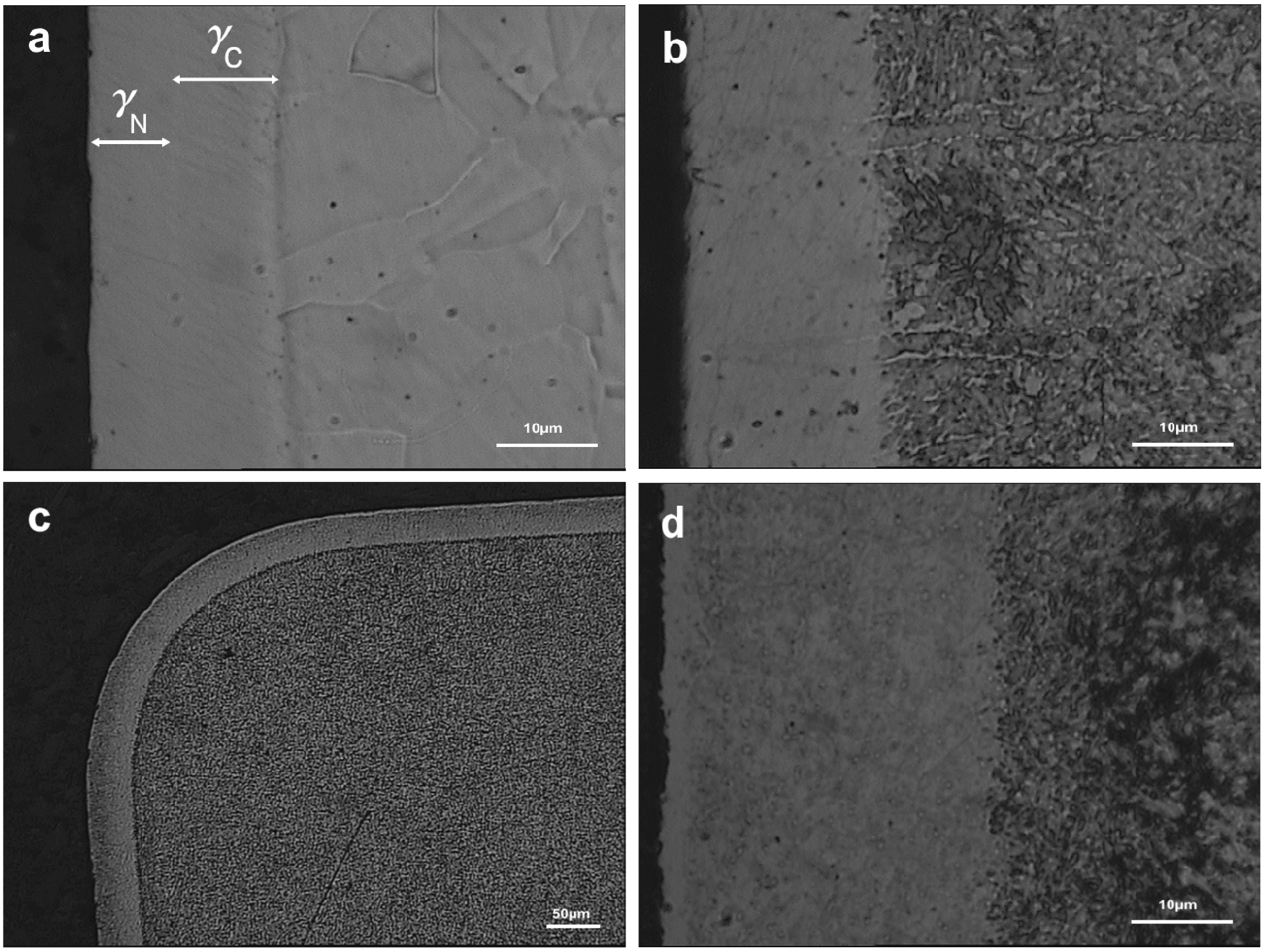

Figure 1. Micrographs of low temperature nitrocarburised stainless steel. a) Austenitic AISI 316; total process time of 90 minutes. b) Martensitic AISI 431; total process time of 90 minutes. c) and d) Martensitic AISI 420 total process time 75 minutes.

Nitrocarburising of martensitic stainless grades is also possible, which is shown in Fig.1b-d. The grade AISI 431 (Fig.1b) treated in 90 minutes yielded a case approximately 20 µm thick and a surface hardness higher than 1800 HV. Depending on the details of the other process parameters the hardened zone can be tailored, i.e. it is possible to obtain expanite, nitrogen/carbon "expanded martensite" or controlled precipitation of nitrides (e.g. epsilon nitride). As a consequence of the very high surface hardness, the surface is totally scratch-free. The martensitic stainless steel grade AISI 420 was treated in 75 minutes, which resulted in the formation of a case with a thickness of approximately 35 µm (fig.1c,d). The surface hardness is higher than 1800 HV.

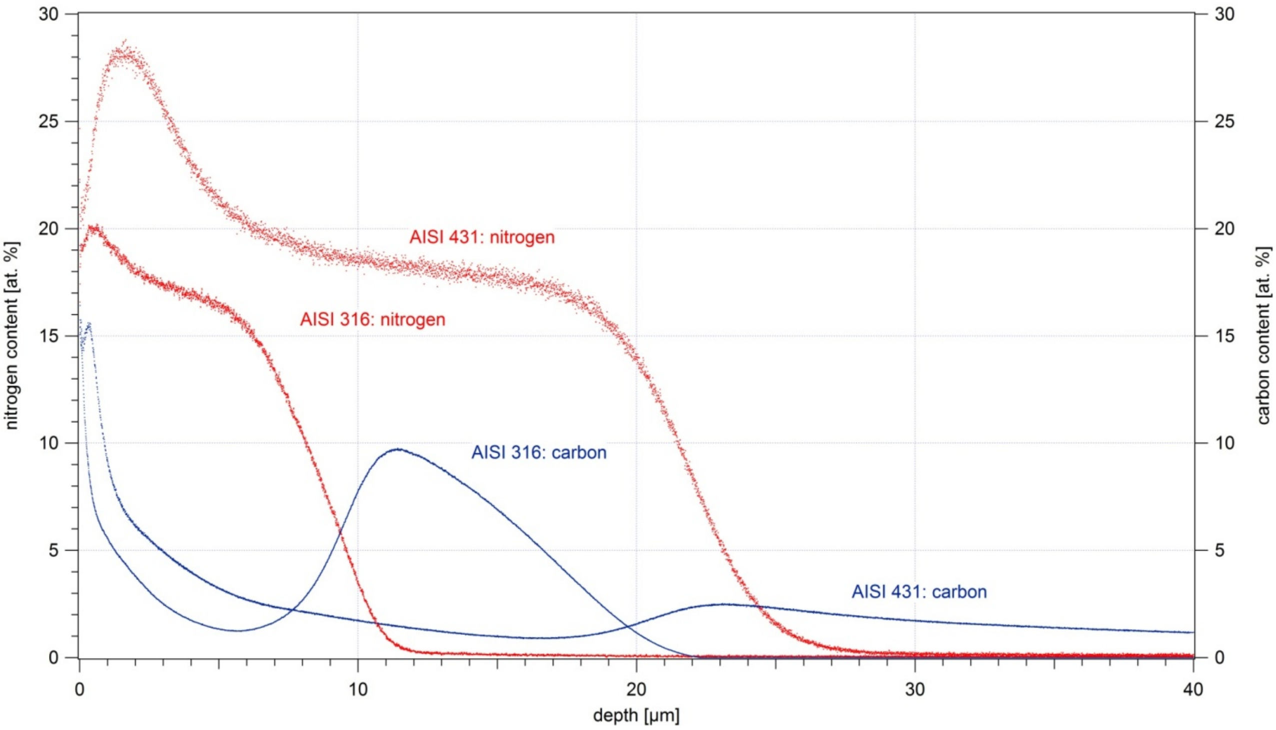

Fig. 2. Glow discharge optical emission spectroscopy (GDOES) concentration-depth profiles of low temperature nitrocarburised stainless steel. The profiles are obtained on the samples shown in Fig. 1 (AISI 316 & AISI 431).

Concentration-depth profiles of the nitrocarburised AISI 316 and AISI 431 (micrographs shown in Fig. 1a,b) are depicted in Fig.2. For the austenitic AISI 316 it is obvious that the expanite "layer" consists of a nitrogen and carbon rich part. The near-surface zone consists of nitrogen expanite whereas the carbon expanite is located underneath. The sum of the two interstitial profiles yields a very smooth and gradual transition from the surface into the substrate in terms of total interstitial content. Such a profile is favourable in terms of load bearing capacity and fatigue properties to prevent the "egg shell" effect. For the martensitic steel grade the nitrogen content is higher than for AISI 316 and extents deeper into the material. A more shallow carbon "peak" is observed at the interface between the nitrogen rich zone and the substrate. Most likely this carbon does not originate from the nitrocarburising gas atmosphere, but is a consequence of "build-up" of carbon inherently present in the martensitic alloy and pushed ahead of the advancing nitrogen front. The total affected zone of the martensitic alloy is hence more than 30 µm.

3.1. Technological examples

The fast nitrocarburising process is considered to be of relevance for a plethora of applications where wear and corrosion resistance are of importance. Hitherto, long processing times have prevented a wide application of low temperature surface hardening of stainless steel. The present breakthrough brings this technology within reach for many new applications. The examples shown here are meant to illustrate the applicability of the process and should not be considered as present commercial applications.

The photograph and micrograph shown in Fig. 3 illustrates the possibility to surface harden a surgical knife made of martensitic stainless steel. The hardened case is clearly observed on the knife. Please notice that the cutting edge of the knife is inclined at a low angle with respect to the plane of section in the micrograph, which makes the case look much thicker than it actually is, nevertheless the case depth is about 20 µm. The hardness of the knife is greatly improved.

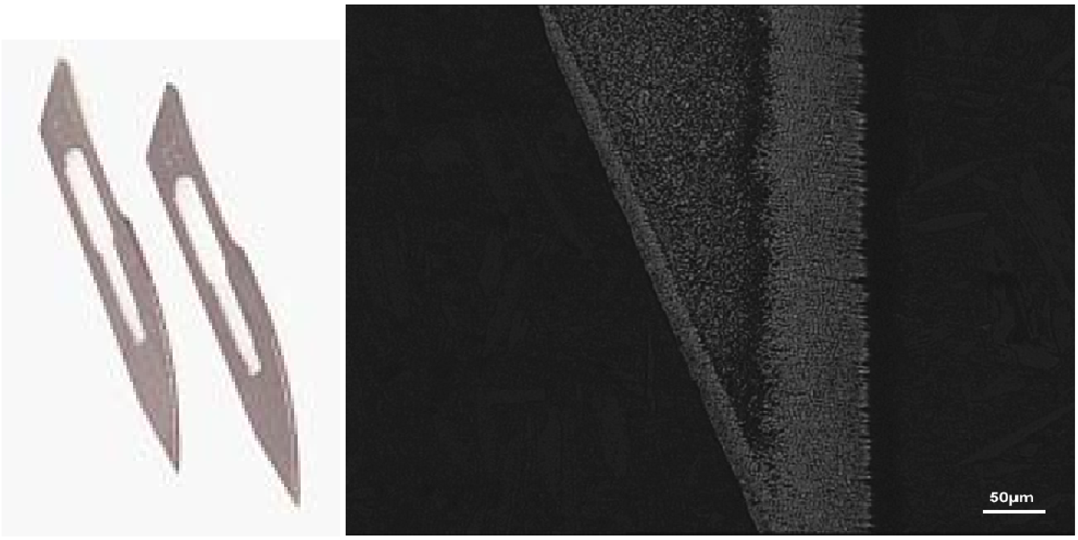

Fig. 3. Micrograph of nitrocarburised martensitic surgical knife. Treated with a total process time of 1 hour.

The photograph and micrographs shown in Fig. 4 illustrate how it is possible to make a self-tapping screw out of austenitic AISI 316 stainless steel by application of a low temperature nitrocarburising process. The surface is transformed into a “duplex layer” of nitrogen and carbon expanite with a total thickness of approximately 15 µm. Please notice the flaw in the material depicted in Fig.4b; as the process is carried out in a gaseous atmosphere nitrocarburising is also observed in this internal “crack”. After nitrocarburizing, the treated screws were successfully applied as self-tapping in aluminium plates without any surface wear.

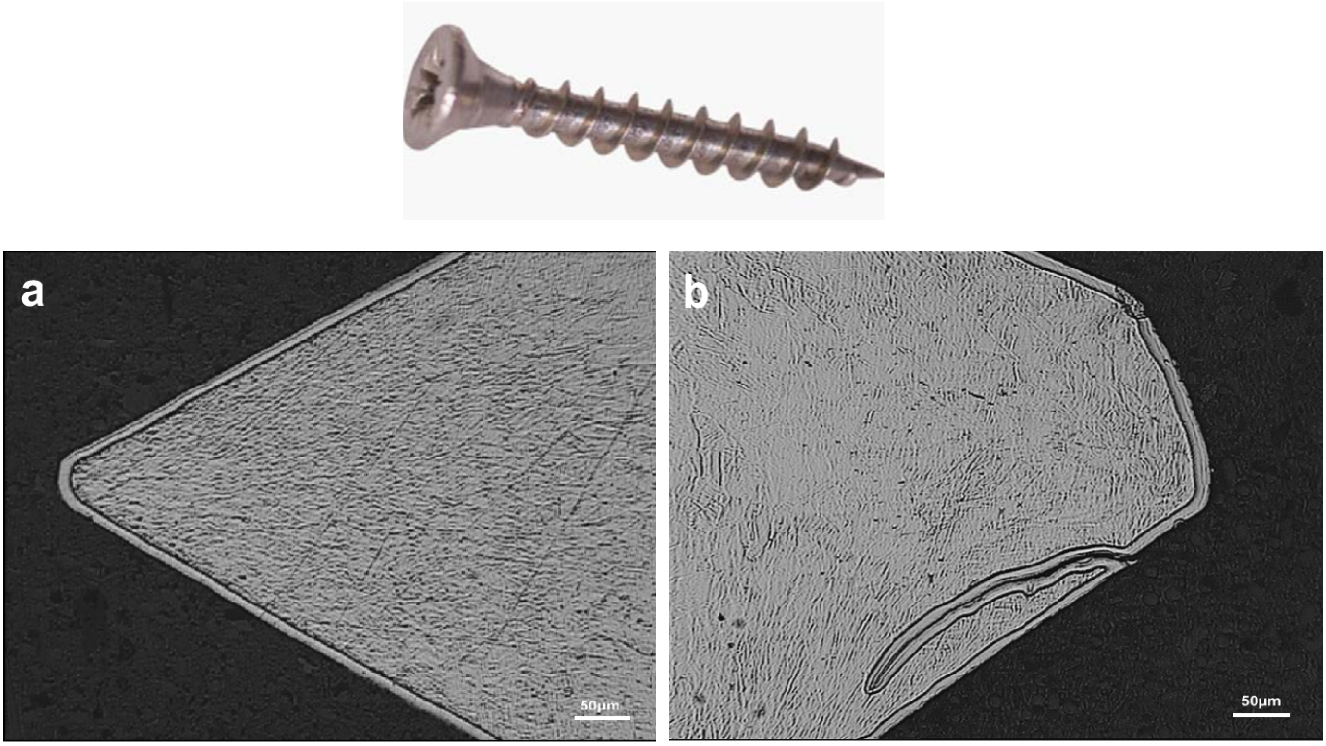

Fig. 4. Micrographs of nitrocarburised austenitic stainless steel AISI 316 screw, treated with a total process time of 1 hour. Please notice the flaw in the material in (b); the internal surfaces are surface hardened, which shows the good penetration ability of the applied gas.

4. Summary and perspectives

A method for fast nitrocarburising of stainless steel has been developed and the applicability of the method has been shown. The treatment times are unprecedented for low temperature surface hardening of stainless steel; it is possible to obtain a case thickness of 15 µm in less than 1 hour for an austenitic stainless steel.

The developed gaseous process can easily be applied in traditional equipment for gaseous surface hardening of steels. It is even envisaged that the process can be fitted to belt/conveyer furnaces for continuous surface hardening of (stainless steel) components. The process has successfully been tested on several other metals/alloys in addition to the ones presented in the present article, including Titanium (alloys), duplex stainless steel, PH stainless steel, tool steel, and ARMCO iron.

References:

[1] T.L. Christiansen, T.S. Hummelshøj, M.A.J. Somers: *Low temperature thermochemical treatment of stainless steel; bridging from science to technology*, DMS 2010.