

## EFFECT OF TIME ON THE COMPOUND LAYER FORMED DURING SALT BATH NITRIDING OF AISI 4140 STEEL

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### Abstract

This research was carried out to study the effect of time on the compound layer of AISI 4140 steel in salt bath nitriding. The nitriding process were implemented on AISI 4140 steel in salt bath component for different times (from 1 h to 10 h) at 580 °C. Samples of AISI 4140 steel were treated and characterized (at surface and core of samples) through the following technique: optical microscopy, scanning electron microscopy, X-ray diffraction (XRD) and microhardness tester. Shows that thick compound layers are formed during continuous salt bath nitriding. The thickness of the compound layer and surface hardness increases with increasing time.

**Keywords:** Salt bath nitriding, AISI 4140 steel, compound layer, time

### 1 Introduction

Thermochemical nitriding treatments are used heavily in many kinds of steel because frequent use of steels in all ranges of industry [1, 2]. Nitriding is a surface thermochemical treatment technique used to introduce and install nitrogen atoms into metallic materials, surface to improve their surface hardness, mechanical properties, as well as wear [3, 4] and corrosion resistance [5, 6], as well as fatigue life [7, 8, 9]. The most important and old method of thermo chemical surface hardening operation is nitriding operation of steel which changing chemical properties of surface layer [10]. Currently there are several ways to add nitrogen to a surface, for example, you can add nitrogen to surface the metal from contact with another surface with nitrogen content at a proper temperature. Various nitriding operations are developed by this method. For instance, gas nitriding, salt bath nitriding, plasma nitriding, laser accumulating operation, reactive magnetron spraying (injecting), nitrogen implanting and plasma [11]. The primary aim of the process of salt bath nitriding (SBN) [12] is to obtain high surface hardness, improve mechanical properties and increase wear resistance, fatigue life, dimensional stability and corrosion resistance as a result of the presence of compounds formed at the surface, in addition to a zone of diffused nitrogen in solid solution with the base material, subjacent to the compound layer [13, 14, 15].

Nitriding is one type of the most widely used thermo-chemical treatments in the industry, which produces strong and shallow case with high compressive residual stresses on the surface of steel components such as gears, crankshafts, dies and tools [16, 17, 18, 19]. The machine components working under cyclic loads are severely subjected to the threat of fatigue failure [20, 21]. Salt bath nitriding is commonly used as an alternative to high temperature conventional heat

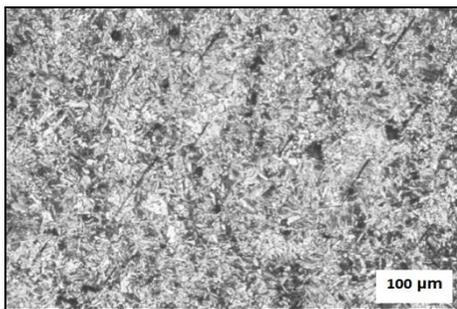
treatments such as carburizing and carbonitriding, nitrogen and carbon diffuses modifies surface and near surface microstructure producing hard layers with altered mechanical properties. During the process, a two parts surface layer is formed, an outer compound layer ( $\epsilon + \gamma'$ ) with a nitrogen diffusion layer ( $\alpha$ ) below it [22]. It is called Compound layer also 'white layer' [23, 24, 25], it inferred that the hardness and the depth depend largely upon the time and the temperature of the salt bath nitriding treatment. [22], initial microscopic situation of the steel [26, 27], the amount presence of nitride elements in the steel [26, 28]. The characteristics of a nitrided steel component after nitriding treatment are defined by both the core and the structural properties of the white layer and the diffusion layer [29]. There's plenty of research that aims to predict the behaviour of steel after nitriding treatments and examine changes in the resulting structures of treatment nitrided components [30, 31, 32, 33]. However, the studies on the effects of salt bath nitriding on AISI 4140 steel and the variables in the morphology evolution of the white layer are very scarce.

In this study, the nitriding process was implemented on AISI 4140 steel in salt bath component at ten different times (1, 2 ... 10) h when the temperature was constant at 580°C. The main objective of the present work is to present the influence of salt bath nitriding time on structural characteristics such as microhardness profile, thickness of the white layer.

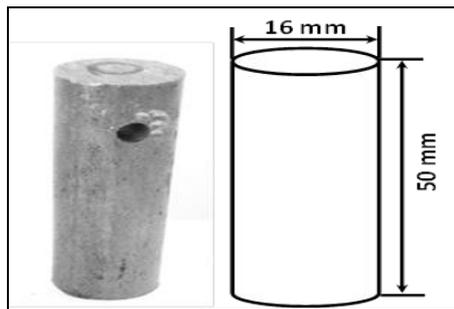
## 2 Experimental procedure

### 2.1 Material and methods

The material used for the investigation was AISI 4140 steel with the following chemical compositions (in mass%): 0.47 C, 0.93 Cr, 0.2 Mo, 0.79 Mn, 0.13 Ni, 0.25 Si, 0.015 P, 0.037 S and balance Fe, and the steel has the microstructure of martensitic (**Fig. 1**) with an average hardness of 348 HV. Samples of dimensions 16 mm diameter and 50 mm height were machined; The dimensions of the specimens are given in **Fig. 2**, ground and polished to a surface roughness (Ra) of 0.9  $\mu\text{m}$ .



**Fig. 1** Microstructure of AISI 4140 steel



**Fig. 2** Shape and dimensions of specimen

The samples was subjected to a heat treatment consisting in: austenitization at 850 °C during 30 min. then oil-quenching and tempering at 600 °C for 120 min. with further slow cooling in air. The samples were placed in the oven at 350 °C, Next, the nitriding processes were carried out in salt bath comprising of cyanates and carbonates. The nascent nitrogen diffuses into the surface of steel. The samples were immersed into salt bath for ten different times (from 1 h to 10 h) at 580°C.

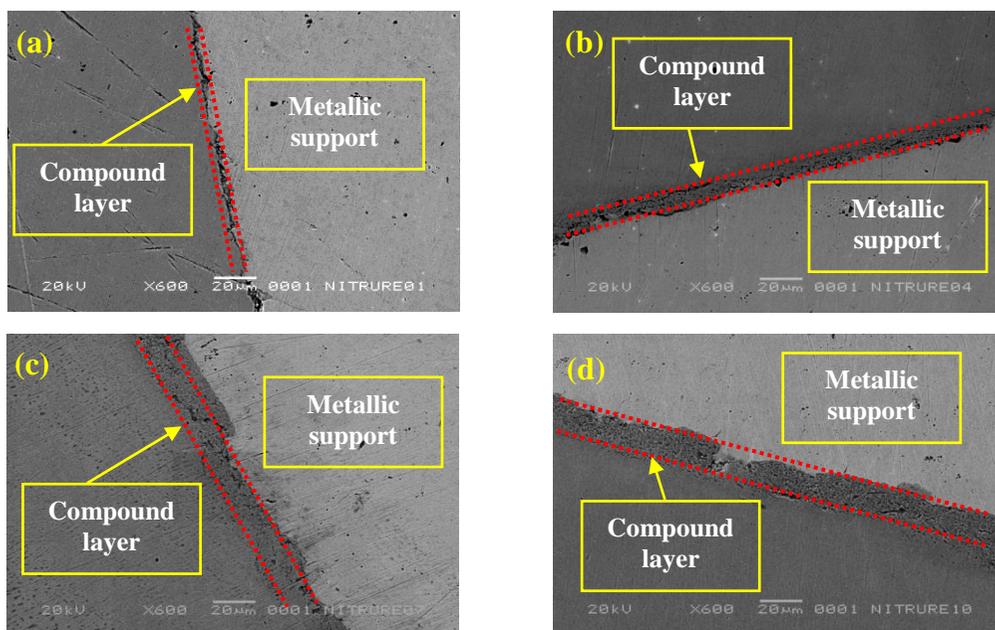
For the scanning electron microscopy (SEM) investigation was used to characterize the microstructures and measure the thickness of the white layer, for this reason, pieces were cut

from the samples and prepared as cross-sections by subsequent embedding, polishing. The sample micrographs were obtained using a scanning electron microscope (JEOL JSM 6060 LA). Phases were determined by X-ray diffraction (XRD) with Cu-K $\alpha$  ( $\lambda = 1.54 \text{ \AA}$ ) radiation. The surface hardness of specimens before and after salt bath nitriding were evaluated using a microhardness tester with a load of 300 gf, by the machine Digital Micro Vickers Hardness Tester (Model: HVS-1000Z), according to the ASTM E384-99 standard [34].

### 3 Results and discussion

The typical cross-sectional microstructure of samples treated at different conditions is shown in Fig.3. The specimens were mechanically fine-ground and polished. It can be clearly seen that a compound layer (also called “white layer”, because it stays white when the steel is etched with Nital) is formed on the sample surface during salt bath nitriding. Through the last figure could be observed the presence of a continuous and with a non-uniform thickness for white layer. Therefore, values smaller of compound layer thickness have been relied upon in this work.

The surface microstructure on nitriding steels has been widely investigated and discussed in several papers [35, 36, 37]. The nitriding process develops a zone which can be subdivided into a compound layer, with carbo-nitrides, and an underlying diffusion layer. The compound layer is formed by  $\gamma'$ -Fe<sub>4</sub>(N,C) and  $\epsilon$ -Fe<sub>2-3</sub>(N,C), while interstitial nitrogen and alloy forming elements  $\alpha$ -Fe nitrides are present in the diffusion layer.

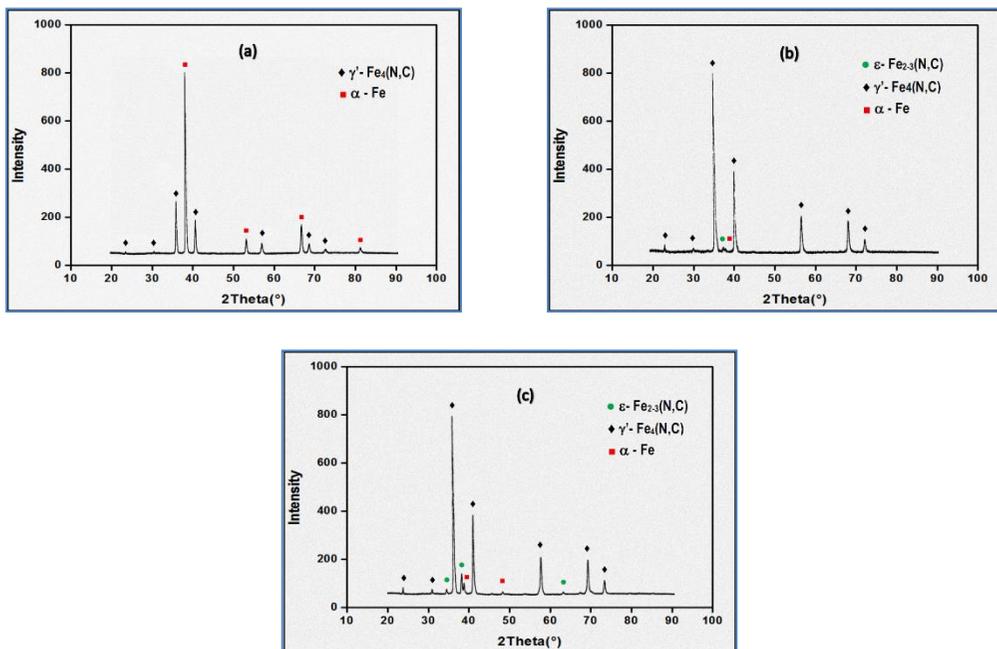


**Fig. 3** Cross-sectional microstructure of 42CrMo4 steel after salt bath nitriding at 580°C for different times

Treatment of nitriding by salt bath at 580 °C at various times (2, 6 and 10 h) of treated time produced different nitrided layers in terms of morphology, thickness and phase structure:  $\alpha$  phase, the  $\gamma'$ -Fe<sub>4</sub>(N,C) phase and the  $\epsilon$ -Fe<sub>2-3</sub>(N,C) phase [38, 39].

The microstructure of the treated samples was examined by X-ray diffraction analysis. This analysis was performed to demonstrate the effect of the salt bath nitriding on AISI 4140 steel for identify the surface phases, the diffraction pattern obtained from the treated material is displayed in the **Fig. 4**.

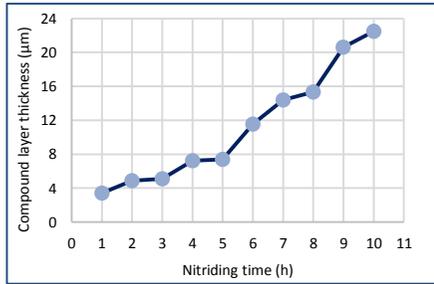
When the XRD patterns were examined after 2h of treatment, it has been seen that both alpha-iron and  $\gamma'$ -Fe<sub>4</sub>(N,C) phases formed on the surface of the steels, but  $\alpha$ -Fe phase appeared before the  $\gamma'$ -Fe<sub>4</sub>(N,C) phase as shown in **Fig. 4(a)**. While after several hours of treatment the peaks for  $\gamma'$ -Fe<sub>4</sub>(N,C) phase increased, on the other hand, the  $\alpha$ -Fe phase has decreased while the peaks for  $\epsilon$ -Fe<sub>2-3</sub>(N,C) phase began to appear as shown in **Fig. 4(b)** and **Fig. 4(c)**. It was concluded that as nitriding time increased, the intensity of peaks for  $\epsilon$ -Fe<sub>2-3</sub>(N,C),  $\gamma'$ -Fe<sub>4</sub>(N,C) phases increased in the compound layer and  $\alpha$ -Fe phase decreased.



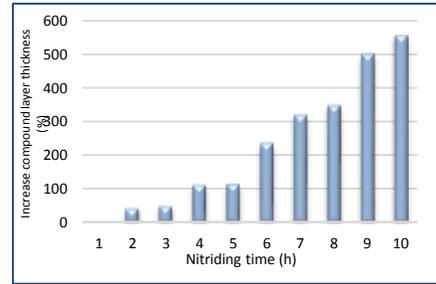
**Fig. 4** X-ray diffraction patterns of AISI 4140 nitrided at 580°C h for (a) 2h, (b) 6h, (c) 10h

**Fig. 5** shows the variation of compound layer thickness as a function of treatment time. The compound layer thickness of the surface of the nitrided sample at 580°C was observed in the range of 3.43–22.5  $\mu\text{m}$ . Can be concluded that the compound layer thickness increased with increasing treatment time [40, 41, 42]. This can be associated with the increase of the diffuse nitrogen and carbon into the surface of AISI 4140 steel with increasing treatment time [43, 44, 45].

The variation in the increase compound layer thickness as a function of the nitriding time is shown in **Fig. 6** for 580°C. The thickness values for compound layer increased a significant in percentage compound layer thickness after salt bath nitriding in the range of 42.27 % to 555 % for both times 1h and 10h, respectively. This increase can be explained by increasing the distribution of carbon and nitrogen over time in the surface layers of samples.

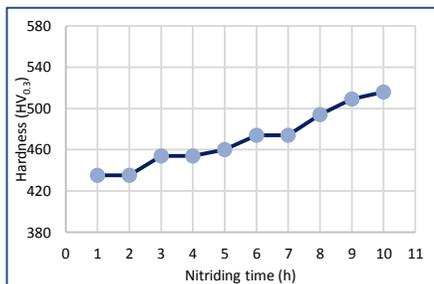


**Fig. 5** The relationship between compound layer thickness and treatment time

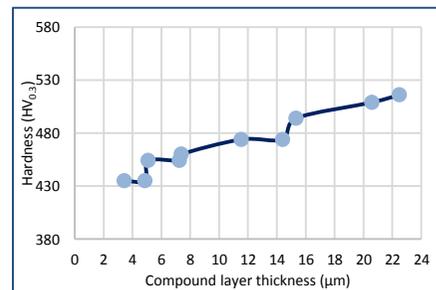


**Fig. 6** Increase compound layer thickness as a function of the nitriding time

The surface hardness values measured after nitriding are reported by microhardness tester with a load of 300 g. The values are the mean of 5 measurements randomly taken on the surface. In **Fig. 7**, the effect of treatment time on the surface hardness behavior of salt bath nitrided samples from 580°C are given. The hardness are increased with the increase of process time [46]. The surface hardness of specimens is in the range of 410–516 HV<sub>0,3</sub>, since the concentration of metal nitrides increases with increasing time. The effect of thickness of the white layer on the hardness behavior of nitrided samples of AISI 4140 is shown in **Fig. 8**. Surface hardness show an increasing trend with increasing thickness of the white layer.



**Fig. 7** The relationship between surface hardness and treatment time



**Fig. 8** The relationship between surface hardness and thickness of the white layer

#### 4 Conclusions

The principal aim of this paper is to understand and interpret the effect of time on the compound layer formed during salt bath nitriding of AISI 4140 steel. Based on the results obtained, the most important relationships derived are as follows: The presence of the compound layer after salt bath nitriding on Samples. The principal phases that form during the salt bath nitriding of AISI 4041 steel are first a solid solution of  $\alpha$ -Fe, the next phase that forms is gamma-prime ( $\gamma'$ ), and is usually represented by the chemical formula  $\text{Fe}_4(\text{N,C})$ . The third phase for consideration is  $\varepsilon$ - $\text{Fe}_{2-3}(\text{N,C})$ . The compound layer thickness increase with increasing time. The surface hardness increased with increasing time of salt bath nitriding treatment.

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