EFFECT OF SALT BATH NITRIDING ON SURFACE ROUGHNESS BEHAVIOUR OF AISI 4140 STEEL

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Abstract
In the present research, AISI 4140 steel was nitrided in salt bath to study and analyze the behaviour of the surface roughness. The structural surface characterization behaviour of the nitrided steel was compared to the behaviour of the same steel which was untreated. The nitriding process was implemented in the salt bath component at ten different times (from 1 h to 10 h) when the temperature was constant at (580ºC). The influence of nitriding treatment on structural properties of the material was studied by scanning electron microscopy (SEM), microhardness tester and surface profilometer. It was found that salt bath nitriding was effective in improving the surface properties behaviour of this steel. Experimental results showed that the nitrides ε-Fe2-3(N,C) and γ'-Fe4(N,C) present in the compound layer increase the microhardness (406–502 HV0.3), the initial surface roughness values of nitrided samples were higher than those of unnitrided specimens, it also observed that the increasing the nitriding time increases the surface roughness parameters (Ra, Rq and Rz).

Keywords: salt bath nitriding, AISI 4140 steel, microhardness, surface roughness

1 Introduction

Surface topography is an important characteristic that determines, among other things, catalytic activity, electrochemical potential, adhesion, friction coefficient, susceptibility to wear and scuffing failure and aesthetic appearance [1-2].
Salt bath nitriding [3,4,5] is one of the most widely used thermo-chemical methods [6,7,8], which produces strong and shallow case with high compressive residual stresses on the surface of steel components such as gears, crankshafts, dies and tools [9,10,11].
Surface roughness of industrial components strongly affects their performance, i.e, many surface properties such as friction [12-13], surface wear [14], fluid flow in rough pipes [15] and the functioning of vacuum seals [16] are strictly dependent on surface roughness. In addition, surface roughness seems to be important in bioengineering for example in the joints of the bones [17]. Quite often small roughness is desired since small roughness reduces wear and energy loss, but when high friction is needed also the roughness should be larger. Hence, the inspection of surface roughness of the work piece is very important to assess the quality of a component.
The quality of surface roughness is an important requirement for coated parts [18].
There are at least five different mechanisms by using the surface modification methods to increase the fretting resistance: (1) to induce a residual compressive stress; (2) to decrease the coefficient of friction; (3) to increase the surface hardness; (4) to alter the surface chemistry; (5) to increase the surface roughness [19].

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There's plenty of research that aims to predict the behaviour of steel after nitriding treatments, AISI 5140 [20-25], AISI 430[21], AISI 1045[22], AISI 4340[23], AISI 410 [24], API X-65 [26], Fe–Cr alloys [27] and examine changes in the resulting structures of treatment nitrided components by plasma nitriding [28,29,32,33], ion nitriding [30,31,34], plasma nitrocarburizing [35] and salt bath nitriding [5]. On the other hand there's many studies interested to study surface roughness [36,37,38,39,40,41], methods of measurement of surface roughness parameters [43,44,45,47] and others [42,46,48].
AISI 4140 is a low-alloy steel widely used in various applications such as automotive driving elements (steering components, crankshafts). There is still an interest in studying the properties of AISI 4140 steel.
The main concern of these studies is improving the mechanical performance of the material using surface treatment [49-50], corrosion resistance [51], wear behaviour [52, 53, 54], microstructure [55], fatigue life [56, 57, 58, 59, 60] and other investigations [61]. However, the studies on the effects of salt bath nitriding on AISI 4140 steel and the variations in surface roughness parameters of the nitrided 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 580ºC). Therefore, the aim of the paper is to make an attempt to investigate the influence of processing time on the microstructure, the microhardness and the surface roughness parameters in the nitrided layer by using scanning electron microscopy (SEM), Digital Micro Vickers Hardness Tester and surface profilometer.

2 Surface roughness parameters

Surface roughness parameters are defined in international standards [62]. It is convenient to divide them into three main groups: (1) Height parameters, comprising height information only, such as roughness average (Ra), RMS roughness (Rq) and average maximum peak to valley height (Rz); (2) Spacing Parameters; (3) Hybrid parameters - which combine height and spacing information such as RMS slope (Rdq). The most common surface roughness parameter used in industry is the average roughness (Ra). It is defined as:

$$R_a = \frac{1}{L}\int_0^L |z(x)|dx \quad (1.)$$

where: Ra [μm] - roughness average, L [μm] - total horizontal measurement path, z [μm] - height coordinate, x [μm] - length coordinate.
When we have digital data the integral is normally approximated by a trapezoidal rule:

$$R_a = \frac{1}{N}\sum_{i=1}^{N} |ri| \quad (2.)$$

where: N - the number of sample points evaluated, ri [μm] - represents the absolute value of the profile deviation from the mean line.
The root mean square (Rq) is the square root of the average of the square of the deviation of the profile from the mean line. This parameter is more sensitive to the peaks and valleys than Ra.

$$R_q = \sqrt{\frac{1}{L}\int_0^L z^2(x)dx} \quad (3.)$$

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Maximum height of the profile (Rz) is the sum of these two, or the vertical distance from the deepest valley to the highest peak, for a digital profile:

$$R_z = R_p + R_v \quad (4.)$$

where: Rp [µm] - The maximum profile peak height, Rv [µm] - The maximum profile valley depth.

3 Experimental procedure

3.1 Studied Material

The test material used in this investigation is the low alloyed steel 42CrMo4 (AISI 4140). The chemical composition (mass percentage) of this quenched and tempered steel is shown in **Table 1**. Chemical composition of AISI 4140 using by SOLARIS CCD Plus spectrometer (is an advanced CCD based optical emission spectrometer for Metal Analysis).

Table 1 Chemical composition of AISI 4140 [wt. %]

C	Si	Mn	Cr	Ni	P	S	Mo	Fe
0.47	0.25	0.79	0.93	0.13	0.015	0.037	0. 2	bal

3.2 Nitriding processes

In order to improve hardness and toughness of AISI 4140 steel, the specimens were subjected to a heat treatment consisting in: austenitization at 850 °C during 30 min. Then fast cooling in oil, then 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 in salt bath for ten different times (1h, 2h, 3h, 4h, 5h, 6h, 7h, 8h, 9h, 10h) at 580 °C, salt bath nitriding treatments applied to 42CrMo4 steel are given in **Fig. 1**.

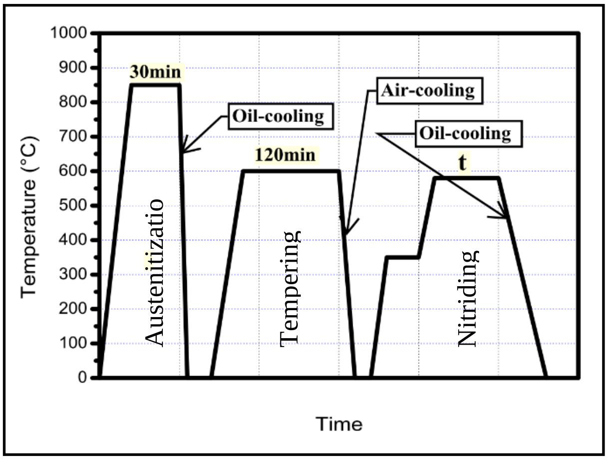

Fig. 1 Salt bath nitriding treatment

3.3 Microstructure and microhardness and surface roughness measurements

Scanning electron microscopy (SEM) type of JEOL JSM 6060 LA was used to characterize the microstructures of AISI 4140 after salt bath nitriding. Cross sections from nitrided specimens were ground, polished.

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Microhardness measurements were used to determine the diffusion layer thickness as well as to aid in characterizing the physical properties of the diffusion layer as a function of depth. The surface hardness and the microhardness profile of salt bath nitrided specimens were evaluated using a microhardness tester with a load of 300 g, according to the ASTM E384-99 standard by the machine Digital Micro Vickers Hardness Tester (Model: HVS-1000Z).
Surface profilometer was used to determine of the surface roughness before and after salt bath nitriding (see **Fig. 2**). Surface roughness was characterized by measuring the surface roughness parameters (Ra, Rq and Rz), according to the ISO 4287:1997 standard [62]. During the surface roughness measurements, the measurement length was chosen as 20 mm. For each specimen, surface roughness was determined by five measurements and the average was plotted.

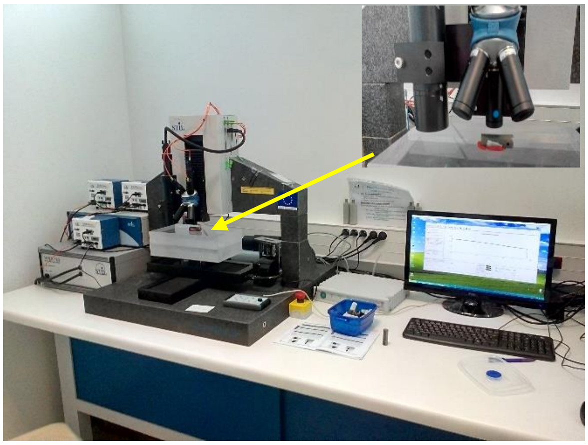

Fig. 2 Surface profilometer

4 Results and discussion

4.1 Microstructure

Fig. 3 shows cross-section morphology of the sample of AISI 4140 steel under the salt bath nitriding at a temperature of 580 °C for 3h by Scanning electron microscope. During salt bath nitriding process, a compound layer and diffusion layer have been formed from surface to core of

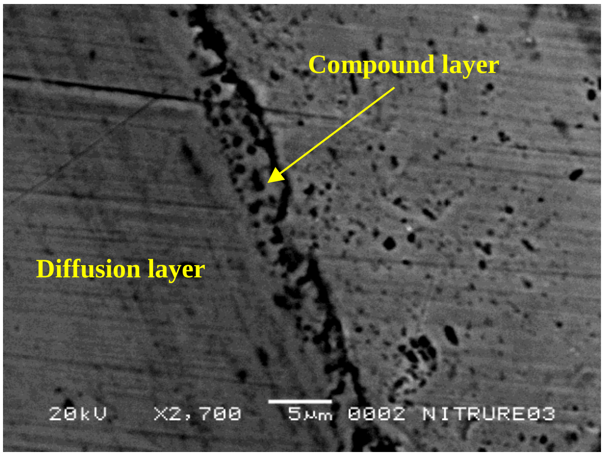

Fig. 3 Cross-sectional microstructure of sample salt bath nitriding at 580 °C for 3h

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the material, As expected in [63, 64, 65, 66, 67]. A white layer of a few microns thickness and a relatively thick diffusion layer were formed at the surface. We have also observed that we cannot distinguish between the two phases (ε-Fe2-3(N, C) and γ'-Fe4(N,C)) in the compound layer.

4.2 Microhardness

Fig. 4 shows the microhardness profiles after the salt bath nitriding treatment. The surface hardness values are in the range of 406–502 HV0.3 for the a temperature 580°C at different times (from 1 to 10) hr.

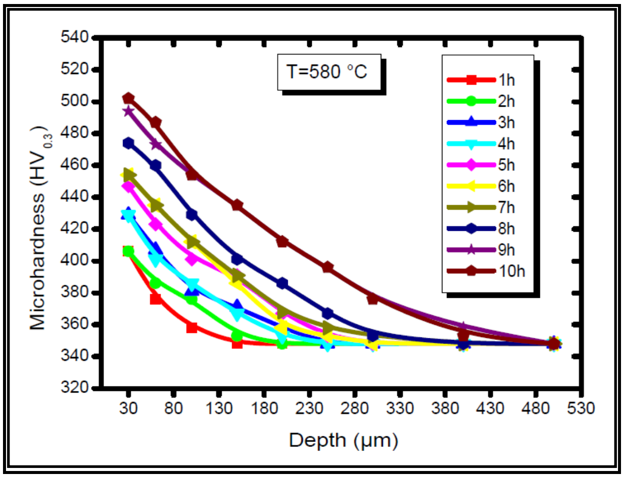

Fig. 4 Microhardness profile of AISI 4140 sample as a function of a depth

The surface hardness of 42CrMo4 steel is increased up to 60% by the salt bath nitriding process. The core hardnesses of specimens were measured as 348 HV0.3. It should be noted that the core hardness of all nitrided specimens was recorded at the same level as in the specimen nitrided after heat treatment and before salt bath nitriding (348 HV0.3). For longer times the maximum microhardness increase for values higher than 500 HV0.3 as a consequence of the high (nitrogen and carbon) pick-up by diffusion through the matrix. This leads to the hardening and trapping of the ε-phase (Fe3N, C) and γ'-phase (Fe4N, C) in the nitrided layer and the concentration of the latter decreases towards the core [68-69].

A significant increase in the values of hardness at a small depths, it was found, for example, that the increasing values for 8h were 36.2% and 32.16% for both depths 30 μm and 60 μm, respectively. The hardness of salt bath nitrided specimens is in the range of 376–487 HV0.3 for 580 °C when the depth was 60 μm. This range is gradually decreasing at different depths from the surface of the sample to case core. The percentage increase is obvious at small depths of diffusion layer. This is a result of more interaction time, which allows for diffusion of more nitrogen and carbon into the middle layer of salt bath nitriding process [70-71].

4.3 Surface roughness measurements

The roughness of plates was determined using profilometer. The length of the measured section was 20 mm. The accuracy of surface roughness measurement was 0.01 μm. The 2D surface profiles of the hard turned surfaces along the feed direction are shown in **Fig. 5**.

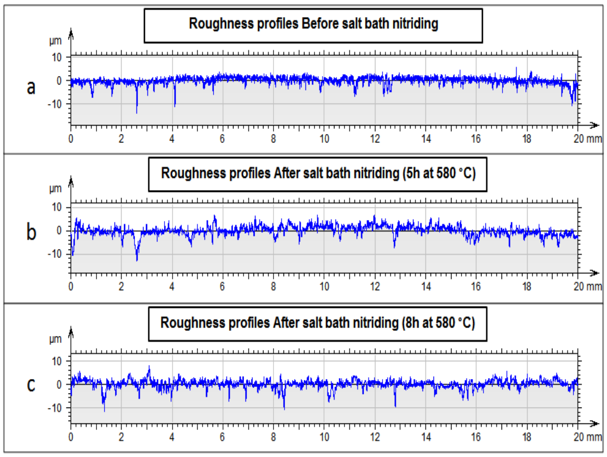

Fig. 5 Roughness profiles of AISI 4140 samples before and after treatment

I must be noted that all the 2D profiles have represented pure roughness values, i.e. the waviness components have been filtered out. **Fig. 5a** shows surface profiles of the AISI 4140 steel before salt bath nitriding, While **Fig. 5b** and **Fig. 5c** they represent surface profiles profiles of the AISI 4140 steel after nitriding treatment at 580 °C for 5 h and 8h, respectively. It can be explained the difference in three surface height variation of the AISI 4140 steel by forming the nitrided layer ((Fe3N, C) and (Fe4N, C)) after nitriding treatment.
Fig. 6 illustrates the changes in the values of surface roughness parameters (Height parameters: Ra, Rq and Rz) at various nitriding times, it can be observed an important increase in the surface roughness values with increasing time of nitriding treatment [72, 73, 74, 75]. The average roughness (Ra) values are in the range of 0,89 –1,48 µm, while the values of the root mean square (Rq) are in the range of 1.25 –1.92 µm, on the other hand the values of Maximum height of the profile (Rz) are between 7.41 and 10.3 µm. The increased surface roughness in higher nitriding time can be attributed to the high chemical sputtering rate of N and C. The increase in surface roughness also depends on the processing salt bath compositions in addition to the treatment time where the latter one factors are practically constant in this experiment.

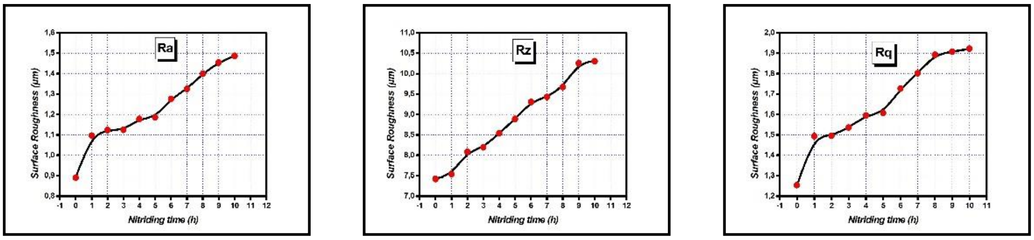

Fig. 6 The effect of salt bath nitriding nitriding on surface roughness

Fig. 7 shows the variation of increase surface roughness parameters (Ra, Rq and Rz) in the time of nitriding for temperature 580°C. The parameters values for 42CrMo4 steel increased a significant in percentage surface roughness after salt bath nitriding in the range of 23.26 % to 66.95 %, 19.13 % to 53.28 %, 1.56 % to 38.91 % for both parameters Ra, Rq and Rz, respectively.

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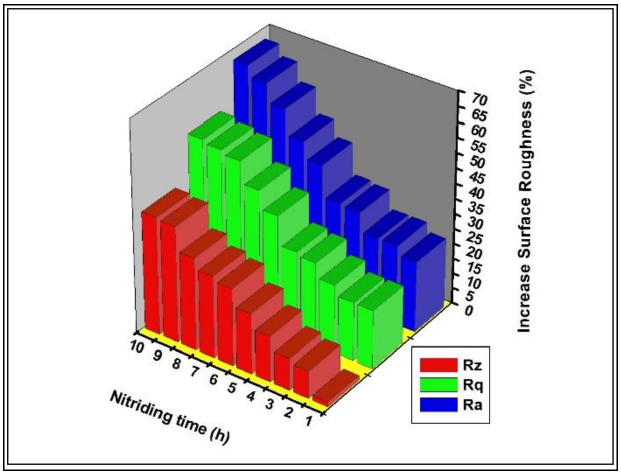

Fig. 7 Increase surface roughness parameters (Ra, Rq and Rz) of AISI 4140 steel

Conclusions

Based on the results obtained, the most important effects of a previous salt bath nitriding on properties and structure of nitrided surface layer in a AISI 4140 steel can be summarized as follows: the presence of compound layer and diffusion layer after salt bath nitriding on samples. The microhardness increased with increasing time of salt bath nitriding treatment when the temperature is constant. Surface roughness parameters (Height parameters: Ra, Rq and Rz) increases with increasing treatment time. The surface roughness is increased by salt bath nitriding and the degree of the increase depends on the nitriding time.

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