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Mechanical and metallurgical properties of ion-nitrided austenitic-stainless steel welds

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Ion nitriding is an operation widely used in industry to harden materials surface. Nowadays, friction welding is one of the special welding methods used for welding the same or different kinds of materials. Especially in industry, it can be necessary to use materials after having operated them with different techniques or to use materials obtained by different manufacturing techniques. Investigating the mechanical and metallurgical properties of this kind of materials can be crucial. In this study, austenitic-stainless steel was used as an experimental material. Additionally, the samples of austenitic stainless steel with a diameter of 10 mm were joined by friction welding. The samples were subjected to ion nitriding process at 550 °C for 24 and 60 h. Then, tensile, fatigue, notch-impact and hardness tests were applied to the weldless and welded parts, and metallographic examinations were carried out. It was found that chromium and iron nitrides precipitated along the grain boundaries and in the middle of the grains. Spectrum patterns revealed that the most dominant phases resulted from the formation of CrN, Fe_4N and Fe_3N. However, the tests revealed that high temperature and longer time of ion nitriding caused a decrease in the values of fatigue and tensile strengths as well as in the notch-impact toughness in the ion nitrided joints.

Keywords: *surface treatment; friction welding; scanning electron microscopy*

1. Introduction

Usually, a machine element can be produced using different production methods. Accordingly, the selection of the method is defined by considering such factors as material, expected strength, number of parts and cost. In some cases, obtaining a part by a combination of more than one production methods instead of only one can be preferred. This kind of approach, in some situations, can help in reaching an economical solution. Friction welding is one of the methods with a higher rate of use among other welding methods, and is one of the most economical and highly productive methods of joining heterogeneous metals, widely used in tool industry.

Surfaces of mechanical parts are exposed to higher stress and abrasive forces compared to inner mechanical parts during the operation of mechanical components, carrying out their expected functions. When stresses and forces exceed the surface strength limit of material, cracks may arise on the material surface, leading to abrasion and corrosion. Therefore, surface strength of materials needs to be increased to provide a longer service life. Ion (plasma) nitriding is a possible remedy for surface wear, and an operation extensively used in the automotive industry, metallurgy and tool-manufacturing industry to harden materials surface, to increase materials abrasion resistance and to provide a longer service life.

At the same time, welded parts may easily be exposed to failure under the influence of corrosion and external forces. Hence, the surface hardening processes can be applied to welded parts in order to protect the welding zone from the problems mentioned above and to increase the wear resistance at this zone by means of hardness increase.

There are many factors affecting the change of material properties through a plasma or ion nitriding process. Numerous studies have been conducted in order to reveal the impact of the plasma nitriding parameters on material properties and introducing this method to the industry. Compared to 304-stainless steels, which have sharp interface, low chromium alloy steels have an extended diffusion zone, and also the depth of ion nitriding increases as the chromium

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Table 1. Chemical composition of AISI 304 Steel [20].

Material	% C	% P	% S	% Mn	% Si	% Cr	% Ni
AISI 304	<	<	<	<	<	17	8.5
(X5CrNi1810)	0.07	0.045	0.030	2.0	1.0	19	10.5

content decreases [1–3]. Surfaces of structures are the most susceptible regions exposed to fatigue failure, and fatigue cracks are generally initiated there. Ion nitriding is widely and successfully used in many machine parts to improve their fatigue performance and mechanical properties [4–11]. However, a comparison of fatigue strength of nitrided specimens and base material also shows a decrease of this parameter, when the parts are nitrided. It was observed that the influence is more significant in high cycle fatigue tests [12]. During ion nitriding, different structures may occur at the surface of the steel, known as a compound layer and case depth from surface to core, respectively. In low-alloy AISI 4140 steels, the thickness of the compound layer increases with time at 500 and 550 °C; however, it starts to decrease with time at 600 °C [13]. In stainless steels, nitrogen-enriched layers of sufficient thickness can be produced in an industrial plant at temperatures up to 400 °C [14].

Inspecting the metallurgical texture of the ion nitrided parts has shown that the texture of these parts is composed of white layer, diffusion layer and base metal. While the white layer is composed of Fe and N phases or their combinations, the diffusion layer has relatively low nitrogen rate and low hardness [15]. Studies have shown that the microstructure of the surface layer can be affected by variable process parameters such as temperature, time and gas mixture ratio. In the ion nitriding process of AISI 5140 alloy steel, the compound layer thickness and the case depth increase with increasing the treatment time and temperature. However, with increasing nitrogen content in the gas mixture, the compound layer thickness increases whereas the case depth decreases [16–18]. The nitriding treatment of austenitic-stainless steels produces a hardened surface layer consisting mainly of the so-called S phase. The presence of nitrides and the thickness of the modified layer depend on the applied treatment pressure [19].

Although ion nitriding of austenitic-stainless steel has been carried out many times so far, there are no detailed mechanical and metallurgical examinations of ion-nitrided steel welds. The aim of this study is to apply the ion nitriding process to austenitic-stainless steel parts joined with friction welding and to investigate the mechanical and metallurgical properties of these parts. In this study, austenitic-stainless steel parts, which are widely used in practice, were subjected to ion nitriding process at 550 °C for 24 h and 60 h, and their mechanical and metallurgical properties were examined using EDX (Energy Dispersive X-ray) analysis so as to gain a detailed insight into the phases occurring during nitriding at the surface.

2. Experimental details

2.1. Material

The examined parts were machined from AISI 304 austenitic-stainless steel. Austenitic stainless steels are characterized by excellent forming capability, good mechanical properties, good weldability and corrosion resistance. Austenitic-stainless steels preserve desired mechanical qualities at high temperatures and they are easily workable as they have good ductility and non-magneticity. Austenitic-stainless steels are readily used in production of mechanical sidings, architectural applications, chemical facilities and equipment. Catalogue values of chemical composition are shown in Table 1 [20].

2.2. Friction welding

Friction welding is a welding method utilizing the heat obtained by conversion of mechanical energy produced during friction on the work pieces interfaces. Friction welding easily joins equal or different cross-sections of the same and different material types [21–23]. Usually, friction welding is divided into two different welding methods: con-

tinuous drive friction welding and inertia friction welding [24, 25]. The set-up used in the friction welding experiments was designed and constructed as continuous drive. A drive motor with 4 kW power and 1410 rpm was selected adequately taking into account the friction and the upset pressures required for the torque capacity in friction welding of steel bars within a diameter of 10 mm. Then, the parameters obtained from the pilot welding experiments (friction time = 9 sec, friction pressure = 60 MPa, upset time = 20 sec and upset pressure = 110 MPa) were taken as optimum welding parameters [24, 25].

2.3. Ion nitriding

Ion nitriding, which has recently become industrially important, is a surface hardening process primarily used to increase the fatigue strength, wear and corrosion resistance, and surface hardness of ferrous alloy steels. Owing to the characteristics like fast nitrogen penetration, simplicity in application, economical and easy control of compound and diffusion layer formation, compared with conventional techniques such as gas and liquid nitriding, ion nitriding has recently received considerable industrial interest. The process has been successfully applied to alloy steels, tool steels, and stainless steels.

A 180-mm diameter glass tube with high temperature resistance was used as a vacuum cell in the experimental set-up. An AISI 304 stainless steel sheet was employed as an anode, while the specimen acted as a cathode. The specimen temperature was measured using thermocouples. A single stage rotary vane pump was used to maintain a vacuum pressure of 280 Pa. The voltage applied between the cathode and the anode was 450 V. The gas mixture was composed of 25 % N_2 and 75 % H_2. Ion nitriding was performed at a temperature of 550 °C for 24 and 60 h.

Later, the ion nitriding process was applied to weldless and welded austenitic stainless-steel parts, and tensile, fatigue, notch-impact, microstructure analysis and hardness tests were applied to both austenitic stainless-steel parts and ion nitrided austenitic stainless-steel welds.

3. Experimental results and discussion

3.1. Tensile test

Tensile tests were performed using an Instron 8501 machine with 100 N/sec. The specimen to be tested was clamped at its two ends by two grips. In the current setting, the upper grip was fixed although its vertical position could be adjusted so as to accommodate the specimens of different sizes. The lower grip was driven by a powerful hydraulic actuator. Once the specimen had been attached to the grips, the vertical movement of the lower grip generated the desired loading on the specimen.

Three specimens were tested in each condition in the experiments and the average of three measurements was presented. After the tensile test, it was seen that the welded parts ruptured from the joined zones. Tensile strengths of the specimens not subjected to any processes and the friction welds were measured as 751 MPa and 750 MPa, respectively. Those of the welds ion nitrided for 24 h and 60 h were 736 MPa and 692 MPa, respectively, while tensile strengths of the weldless specimens – ion nitrided for 24 h and 60 h were 753 MPa and 714 MPa, respectively. Tensile strength of the ion nitrided welds decreased similar to that of the ion nitrided weldless specimens. Tensile strengths of the welded samples, ion nitrided for 24 h and 60 h, were lower due to the exposure to high temperature for a longer period of time [1, 3, 8, 13, 17], which resulted in a white layer and chromium carbide precipitation in the nitrided parts.

3.2. Fatigue test

The fatigue tests were performed using an Instron 8501 machine. During the tests, the load application frequency was 20 Hz. The fatigue test machine stopped automatically as soon as a specimen failure occurred.

Fatigue tests were conducted by superimposing fluctuating tensile loads on a constant tensile load. The constant tensile load produced tensile stresses of 300 MPa. Fluctuating tensile stress amplitudes varied between 150 MPa and 250 MPa, and the numbers of cycles preceding the fracture were recorded.

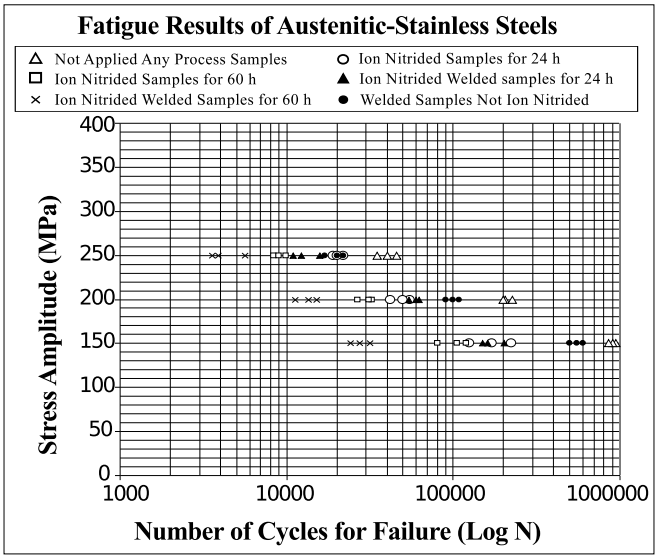


Fig. 1. Fatigue test results.

Three specimens were tested in each condition in the experiments. Fatigue results for the ion nitrided parts were compared with those of the machined base parts. After the fatigue test, it was seen that the welded parts ruptured from the joined zones similar to the tensile test results. Especially, the number of cycles of the ion-nitrided welded parts decreased with prolonged exposure to high temperature, the alteration of surface properties and the case depth, inducing compressive residual stress at the surface (Fig. 1) [5–7, 12, 18].

3.3. Notch-impact test

The ion nitrided specimens were exposed to notch-impact tests using Charpy method. Energy absorbed in Charpy-V notch impact test for each specimen was divided into its net area according to DIN 50115, as the dimensions of each of the specimens were different. First, the values of fracture energy of the specimens were investigated, and then notch-impact tests were applied to the welded parts. The results were compared with those of the base metal.

The calculated notch-impact toughness values are given in Table 2. A compound layer occurred on the surface of steel due to diffusion of nitrogen atoms during ion nitriding. The hard layer improved the values of fracture energy, especially in ion nitrided weldless samples. So, the samples did not break during the notch-impact test. However, the notch-impact toughness of the ion-nitrided welded parts decreased. Then, the parts fractured at the joined zones.

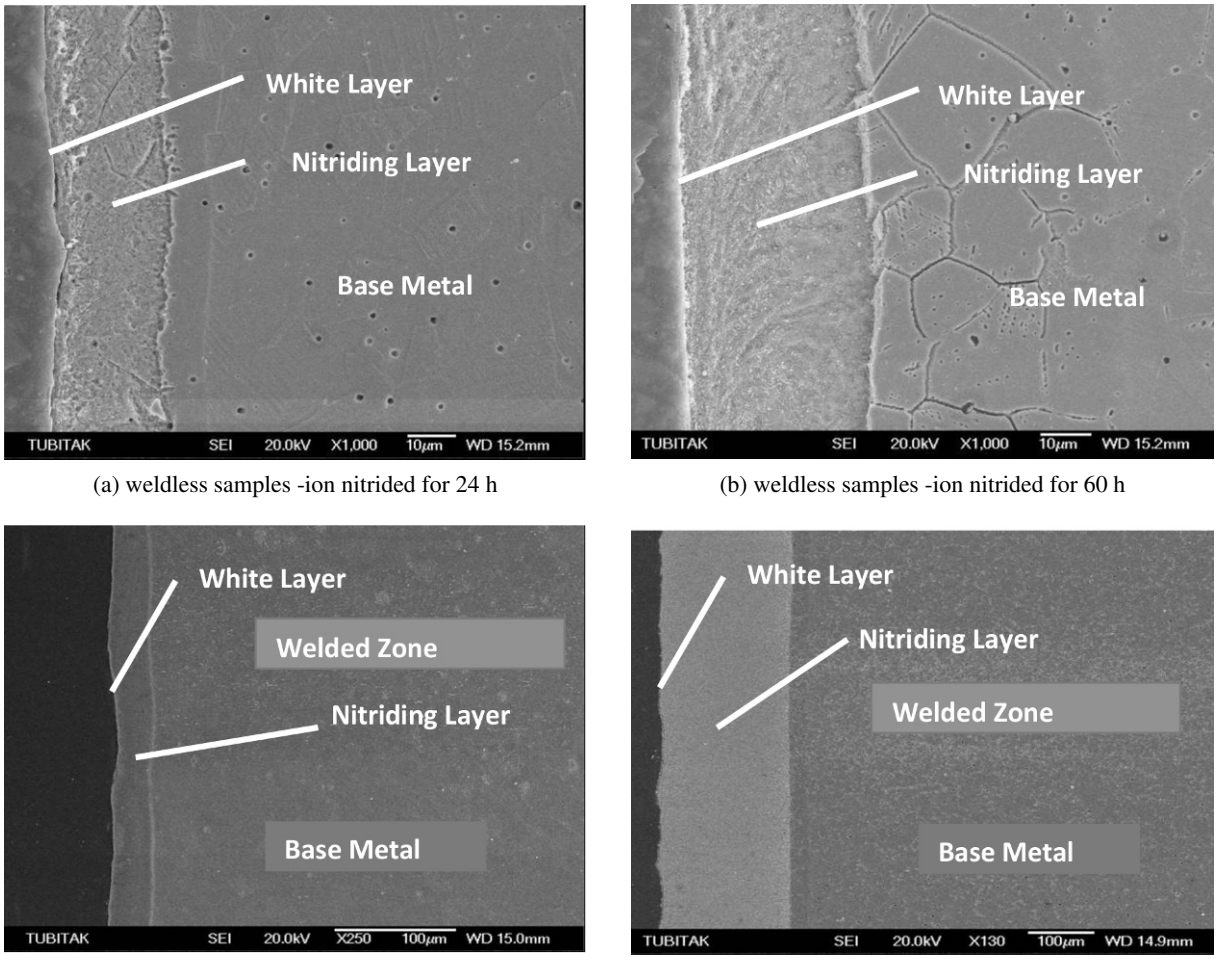
3.4. SEM-EDS analysis

The ion nitrided specimens were subjected to microstructural analysis. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analyses were performed in order to investigate the phases that occur on the surface of the ion nitrided parts. The observations were carried out with a 200 kV field effect scanning electron microscope (SEM- JEOL JSM 5410 LV microscopy) coupled to EDS (energy dispersive X-ray spectroscopy). The software allowed piloting the beam, scanning along a surface or a line to obtain X-ray cartography or concentration profiles of the elements. The EDS analysis was carried out within the defined zone on the SEM images.

SEM analysis shows high relief at the grain boundaries. Some grain boundaries are observed to lean forward on the adjacent (Fig. 2). Then, a growth of a nitrided layer, mainly due to nitrogen diffusion, can be observed as ion nitriding time increases. SEM images of the microstructures and spectrum analysis of the nitrided layer and the base

Table 2. The notch – impact test results.

Material	Notch-impact toughness (J/cm^2)
Samples not subjected to processing	181
Welded samples	400
Welded samples ion nitrided for 24 h	126
Welded samples ion nitrided for 60 h	125
Weldless samples -ion nitrided for 24 h	Not broken
Weldless samples -ion nitrided for 60 h	Not broken



(a) weldless samples -ion nitrided for 24 h

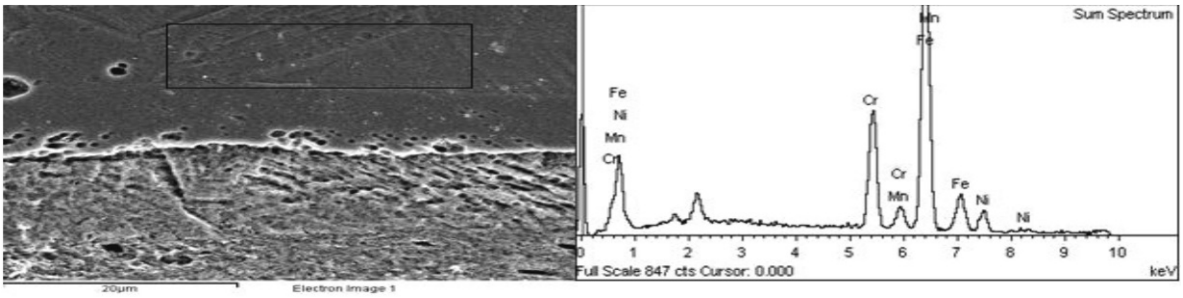
(b) weldless samples -ion nitrided for 60 h

(c) welded samples -ion nitrided for 24 h

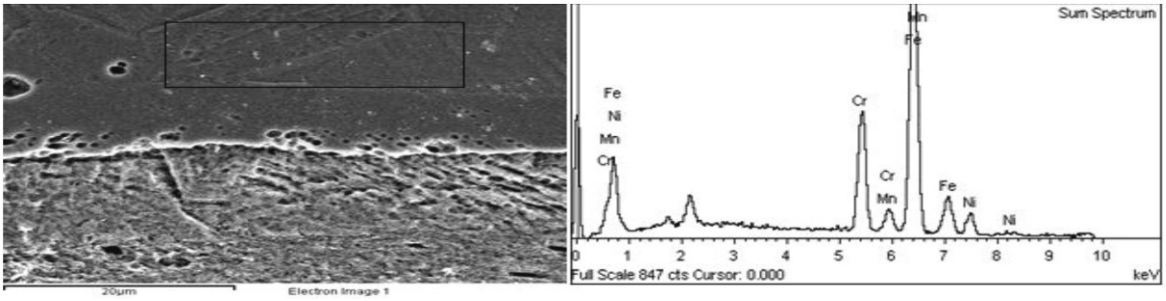
(d) welded samples -ion nitrided for 60 h

Fig. 2. SEM microstructure of ion nitrided sample.

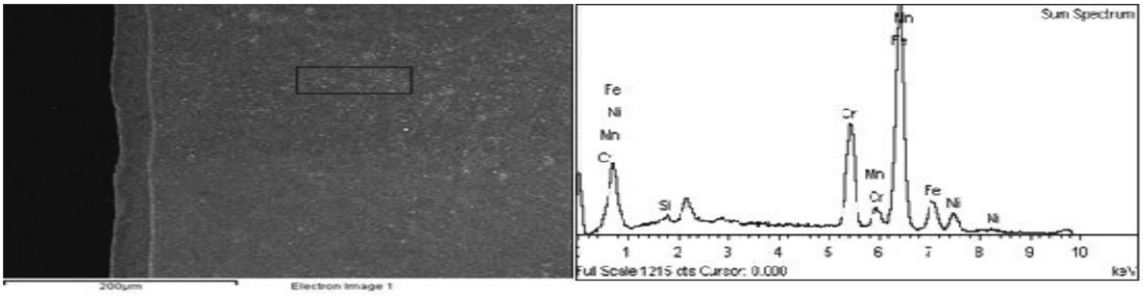
metal in the samples ion nitrided for 24 h, are given in Fig. 3. EDS analysis results corresponding to the SEM microstructures are shown in Table 3. The EDS analysis indicates that the constituents in the defined zone of an ion nitrided layer after 24 h nitriding are as follows: 70.91 % Fe, 18.48 % Cr, 7.52 % Ni, 2.0 % Mn and 1.08 % N (Table 3). In contrast, the EDS analysis shows that the defined zone of the base metal in the ion nitrided sample after 24 h nitriding does not contain nitrogen (Table 3). It can be seen from Table 3 that nitrogen, which provides surface hardness, cannot reach the welded zone and the



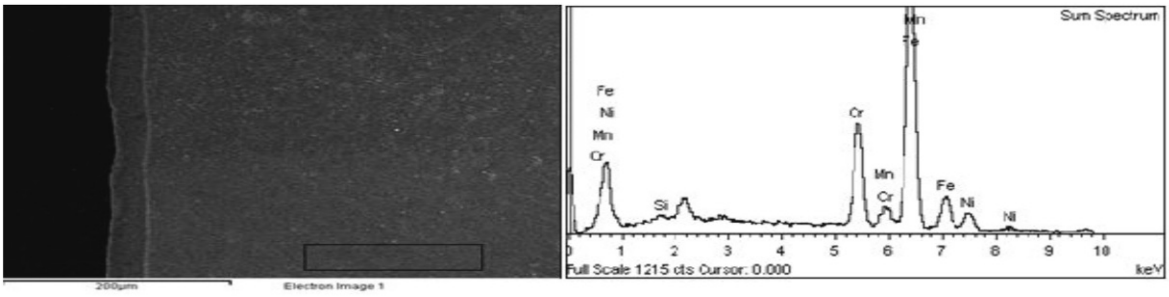
(a) Nitriding layer in a weldless sample -ion nitrided for 24 h



(b)) Base metal in a weldless sample-ion nitrided for 24 h



(c) Welded zone in a welded sample - ion nitrided for 24 h



(d) Base metal in a welded sample -ion nitrided for 24 h

Fig. 3. SEM microstructure and spectrum analysis of parts nitrided for 24 h.

base metal in front of the nitriding zone. The EDS analysis shows a thin iron nitride layer consisting of FeN and CrN intermetallic phases in the specimen ion nitrided for 24 h. Chromium and iron nitrides are found to precipitate along the grain boundaries and in the middle of the grains.

The SEM microstructures and spectrum analysis of the nitriding layer and the base metal, in the

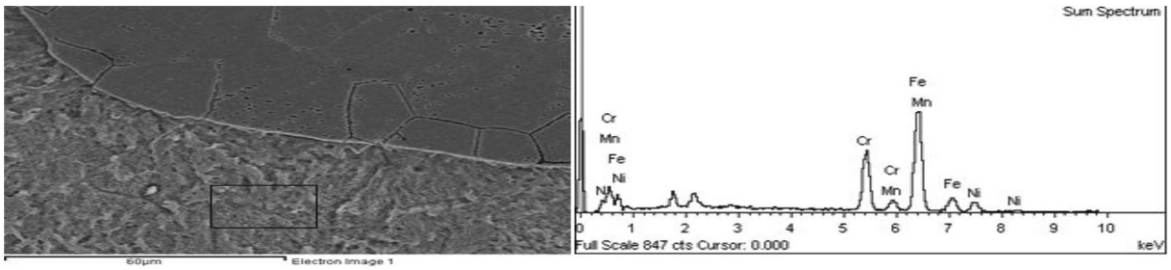
Table 3. EDS analysis results corresponding to SEM microstructure.

	24 h			60 h		
	Element	Weight %	Atomic %	Element	Weight %	Atomic %
Nitriding layer in a weldless-ion nitrided sample	N	1.08	4.15	N	6.30	20.91
	Cr	18.48	19.04	Cr	21.83	19.53
	Mn	2.00	1.95	Mn	2.61	2.21
	Fe	70.91	68.00	Fe	61.06	50.86
	Ni	7.52	6.86	Ni	8.20	6.50
	Total	100.00		Total	100.00	
Base metal in a weldless-ion nitrided sample	Cr	19.09	20.29	Cr	19.43	20.64
	Mn	1.75	1.76	Mn	1.45	1.45
	Fe	70.98	70.25	Fe	71.96	71.17
	Ni	8.18	7.70	Ni	7.16	6.74
	Total	100.00		Total	100.00	
Welded zone in a welded-ion nitrided sample	Si	0.89	1.73	N	0.78	1.53
	Cr	19.28	20.31	Cr	19.062	0.10
	Mn	1.56	1.56	Mn	1.69	1.69
	Fe	70.84	69.47	Fe	70.65	69.38
	Ni	7.42	6.93	Ni	7.82	7.30
	Total	100.00		Total	100.00	
Base metal in a welded-ion nitrided sample	Si	0.54	1.05	N	0.52	1.03
	Cr	19.18	20.28	Cr	18.55	19.62
	Mn	1.44	1.45	Mn	1.87	1.87
	Fe	71.04	69.92	Fe	71.49	70.40
	Ni	7.80	7.30	Ni	7.56	7.08
	Total	100.00		Total	100.00	

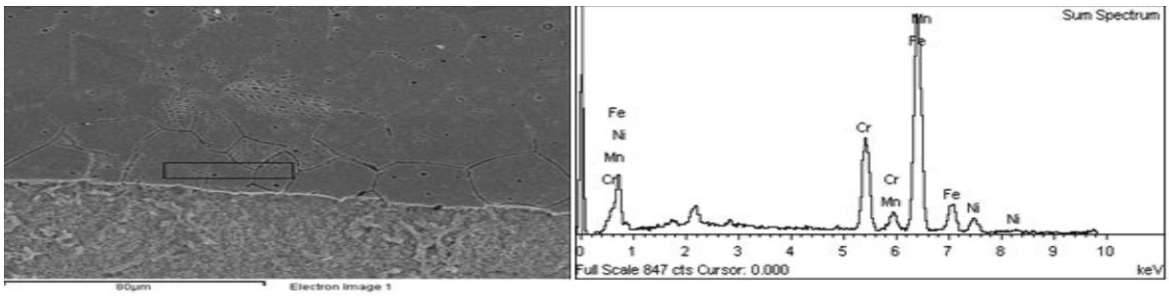
samples ion nitrided for 60 h, are shown in Fig. 4. The results of EDS analysis corresponding to SEM microstructures are shown in Table 3. The EDS analysis shows that the constituents in the defined zone of nitriding layer in the sample ion nitrided for 60 h are as follows: 61.06 % Fe, 21.83 % Cr, 8.20 % Ni, 2.61 % Mn and 6.30 % N. The EDS analysis indicates that nitrogen does not occur in the defined zone of base metal even when the nitriding time has been extended to 60 h (Table 3). The EDS analysis shows that a thick iron nitride layer consisting of FeN and CrN intermetallic phases has been formed due to the long exposure to high temperature during the 60 h nitriding process. The thickness of the layer increased or decreased depending on the diffusion ratio of nitrogen and the processing time. Dark alloy nitrides have been shown inside the layer in the SEM photos. Austenitic stainless steels are susceptible to carbide formation and precipitation within a temperature range of 450 °C–850 °C, which is known as sensitization. In this case, a longer time, in which austenitic stainless steel has been processed at 550 °C, induced carbides precipitation mainly on the grain boundaries (chromium carbide precipitation). In fact, the precipitation on grain borders can be seen in Figs. 2b, 4a and 4b.

3.5. Hardness test

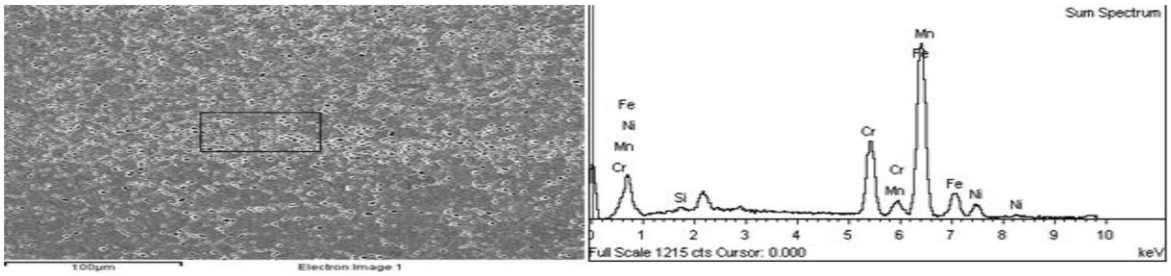
Hardness profiles of austenitic-stainless steel specimens ion nitrided at 550 °C for 24 and 60 h, were obtained under 200 g loads by micro hardness (Vickers) testing. The hardness decreased parabolically from the surface to the core, with a decrease in the concentration of metal nitrides towards the core (Fig. 5). Surface hardness of the specimens ion nitrided for 60 h was found to be higher than that of the specimens nitrided for 24 h. The hardness



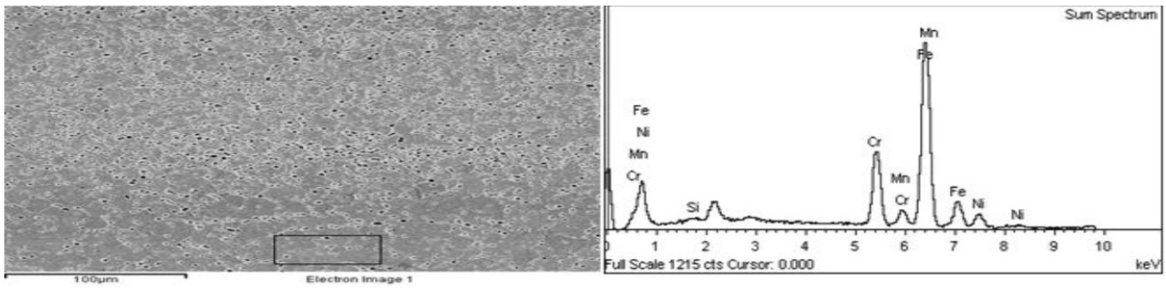
(a) Nitriding layer in a weldless sample -ion nitrided for 60 h



(b) Base metal in a weldless sample -ion nitrided for 60 h



(c) Welded zone in a welded sample -ion nitrided for 60 h



(d) Base metal in a welded sample -ion nitrided for 60 h

Fig. 4. SEM microstructure and spectrum analysis of parts nitrided for 60 h.

results also point out the importance of the effect of the ion nitriding time and diffusion ratio of nitrogen. There was no difference between the hardness of the base metals in the nitrided samples in terms of the hardness ratio. The core hardness remained the same at 241 $HV_{0.2}$.

4. Conclusions

In this study, effects of ion nitriding for 24 and 60 h in welded austenitic-stainless steels were investigated. Ion nitrided parts were subjected to tensile, fatigue and notch-impact test as well as metallo-

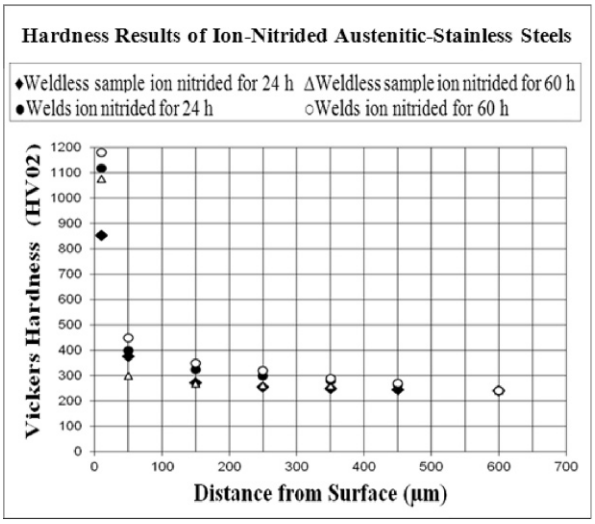


Fig. 5. The hardness profiles of ion nitrided austenitic-stainless steels.

graphic and hardness examinations. The following conclusions can be derived from the results and discussions mentioned above:

- It seems that nitrogen ratio is one of the most important factors governing the ion nitriding process.
- Tensile strengths of the welds ion nitrided for 60 h were lower than that of the welds ion nitrided for 24 h due to exposure to high temperature for a longer period of time.
- Ion nitriding surface treatment improved fatigue strength, but overexposure to high temperature, due to a long process time, caused the fatigue strength to decrease.
- The compound layer did not have dominating effect on the fatigue behaviour of ion-nitrided specimens. When the temperature and time increase, this layer becomes thicker.
- While the ion nitrided weldless parts were not fractured in Charpy test, the ion nitrided welded parts were fractured. This was due to the fact that notch-impact toughness in the ion nitrided welded parts decreased.
- SEM analysis showed high relief at the grain boundaries. Furthermore, formation of nitrides was observed on the surfaces of the parts by SEM and EDS analysis. Spectrum patterns revealed that the most dominant phases resulted from the formation of CrN, Fe_4N and Fe_3N. In addition, the ion nitrided parts included carbide precipitates mainly on the grain boundaries (chromium carbide precipitation).
- Surface hardness increased with an increase of process time due to diffusion of nitrogen during ion nitriding.

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