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Recovery of the microstructural changes of different duplex stainless steel alloys

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Abstract

Purpose – To find out the optimum heat treatments to recover the microstructural changes of stainless steel alloys.

Design/methodology/approach – A total of four alloys were used in this study: two duplex stainless steel (DSS) alloys type 2304 and 2205, super DSS (SDSS) type 2507 and austenitic stainless steel alloy type 316 L. The alloys were heated to different temperatures, 750, 850, 950 and 1,050°C, for three different times, 10 min, 1 and 4 h.

Findings – The microstructural investigations showed that 2205 and 2507 behaved similarly in recovering their microstructures, especially in terms of the ferrite:austenite ratio within specific heat treatments and changing the hardness values. The results indicated that the microstructure of both alloys started to change above 750°C, the largest changes were shown at 850 and 950°C as the lowest ferrite content (FC%) was recorded at 850°C for both alloys. However, the microstructures of both alloys started to recover at 1,050°C. The reduction in the hardness values was attributed to the formation of new ferrite grains, free of residual stresses. On the other hand, the microstructure of the alloy type 2304 was stable and did not show large changes due to the applied heat treatments, similarly for austenitic alloy except showing chromium (Cr) carbide precipitation.

Originality/value – Finding the exact heat treatments, temperature and time to recover the microstructural changes of DSS alloys.

Keywords Super duplex stainless steel, Microstructure, Ferrite content, Recovery, Intermetallic phase

Paper type Research paper

Introduction

The reason behind the globally rapid growth of the increase in demand and consumption of duplex stainless steel (DSS) in petrochemicals, marine and power stations and other engineering applications is mainly due to the chemical composition, consequently the formation of a microstructure of two phases, ferrite and austenite, which provides high strength and corrosion resistance in harsh environments (Boillot and Peultier, 2014). However, heating stainless steel alloys – during welding and heat treatments – remains challenging while they pass through a range of temperatures, specifically from 500 to 1,100°C (Pramanik *et al.*, 2015; Chaudhari *et al.*, 2019). The changes, besides the formation of embrittlement phases such as sigma and chi phases, and carbide and nitride precipitations also include an imbalance phase ratio of austenite/ferrite which leads to the change of the mechanical properties and corrosion resistance (Guimarães and Mei, 2004; Argandoña *et al.*, 2017; Gholami *et al.*, 2015).

Many researchers have worked on investigating the microstructural changes that are associated with welding and heat treatments. Arabi *et al.* (2019) studied the effect of

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spot welding on DSS type 2304 through controlling downsloping current and *in situ* post-weld short annealing of the weld. Both ways enabled enhancing the austenite volume fraction in the fusion zone. Tan *et al.* (2011) studied pitting corrosion resistance of 2304 after autogenous plasma-arc welding and subsequent short-term post-weld heat treatment at different temperatures. The results indicated that the most favorable annealing temperature for the analyzed welded joints was found to be 1,080°C, at which the joint restored the pitting corrosion resistance lost during welding entirely. Alinejad *et al.* (2016) studied thermomechanical characteristics of type 2304 at a temperature range from 850 to 1,150°C. Microstructural observations showed that dynamic recovery in ferrite is the controlling mechanism at all deformation conditions (Dehghan-Manshadi *et al.*, 2008).

Khoshnaw and Rahmatallah (2010) studied the effect of welding heat input by using tungsten inert gas (TIG) on stress corrosion cracking (SCC) of austenitic stainless steel type 304L and DDS type 2205. The results indicated that increasing the heat input causes brittleness for both alloys. However, the duplex alloy showed higher resistance to SCC than the austenitic alloy due to higher chromium (Cr) content and the dual phases. In another study, Khoshnaw *et al.* (2019) worked on the same alloys, focused on the metallurgical aspects, such as intermetallic compound precipitation, that associate with heating from 500 to 1,100°C using thermal cycle simulator. The results indicated that less than one minute is enough to allow the carbides precipitate in both alloys as no intermetallic phases were appeared due to the short time.

Kashiwar *et al.* (2012) investigated the effect of the solution annealing of alloy type 2205 at 1,050 and 1,100°C, also isothermally aged at 700°C for 15 min to 6 h. The results indicated that a significant amount of carbides was observed in the ferrite grains after 1 h of aging at 700°C. Cronemberger *et al.* (2015) found that the intermetallic compound formation in DDS type 2205 increases the brittleness and decreases its corrosion resistance. Badji *et al.* (2008) studied the effect of the subsequent annealing treatment on the phase transformations and mechanical behavior of welded DSS 2205. The results indicated that the temperatures from 800 to 1,000°C resulted in precipitation of sigma phase and Cr carbides. Optimal mechanical properties and an acceptable ferrite:austenite ratio throughout the weld regions correspond to annealing at 1,050°C. Zhang *et al.* (2017) studied the same alloy in terms of the effect of nitrogen content on the pitting corrosion resistance. They showed that nitrogen facilitates primary austenite formation in the weld metal and suppressed Cr_2N precipitation. Deng *et al.* (2009) also studied the microstructure and properties of the same alloy following isothermal aging from 450 to 1,000°C for 10 min. The results indicated that temperatures from 600 to 950°C resulted in precipitation secondary phases, which had a significant reduction in pitting corrosion resistance.

Cojocaru *et al.* (2017) investigated the microstructural changes induced by solution treatment of super duplex stainless steel (SDSS) alloy type F53, 45% ferrite and 55% austenite, to emphasize the correlation between the component phases and the heat treatments. Samples were heated at 1,100°C, with variable holding times: 10, 60 and 180 min. Ciuffini *et al.* (2017) also attempted to show this correlation, the changes of the ferrite:austenite ratio with the heating temperature of two SDSS alloys: F53–S32750 and F55–S32760. They found that heating at 1,080°C gives the best ratio. Pettersson *et al.* (2015) evaluated the changes in mechanical properties of SDSS type 2507 during aging at 300°C up to 12,000 h. Both transmission electron microscopy (TEM) and atom probe tomography were used to follow Cr concentration fluctuations, which was proportional to the hardness of the ferrite phase and the decrease of impact toughness (Taban, 2008). Argandoña *et al.* (2017) studied the alloy UNS S32760, which showed that the resulting hardness values were increased as a function of a longer heat treatment duration which was associated with the formation of a higher percentage of the sigma phase. However, nanoindentation hardness measurements of this phase showed lower values than expected, being a combination of two main factors, namely, the complexity of the phase structure as well as the surface roughness. Calliari *et al.* (2011) studied the effect of heating from 750 to 1,000°C on four types of alloys; 2205, 2507, 2304 and 2101 to study the formation of secondary phases of isothermally aged samples. The results indicated the precipitation of intermetallic phases for 2205 and 2507 grades. On the contrary, the secondary phases are rarely observed after both isothermal aging, up to 750 h for 2304 and 2101.

The above review has shown that many researchers, in the last two decades, focused on finding new metallurgical changes which associate with heating stainless steel alloys and investigating the effects on mechanical and corrosion behavior (Khoshnaw and Gardi, 2007) (Pramanik *et al.*, 2015). However, much lesser research studies can be found which has focused on studying the methods to recover the changes which are associated with welding and heat treatments. Based on that, to fill a part of this gap, this research aims to find the optimum heat treatments which can recover the microstructural changes of a few DSS alloys back to the original manufacture setup microstructure.

Materials and experimental works

In the previous study (Khoshnaw *et al.*, 2019), a thermal cycle investigator was used to investigate the heating treatments of DSS type 2205 and austenitic type 304L within a sensitization range from 500 to 1,100°C. In this study, almost the same temperatures, but for longer time intervals, were applied on four types of stainless steel alloys, which are two DSS type 2205 and 2304, super duplex type 2507 and austenitic type 316L. Table 1 shows the chemical composition of the alloys. Plates with thicknesses from 8 to 12 mm were cut to samples 50 × 10 mm. Different heat treatments – heating to different temperatures and keeping the sample at specific times – were applied, starting from 750 to 1,050°C – the interval increase was 100 °C – for different times, that is, 10 min, 1 and 4 h.

To proceed with microstructural investigations, samples were prepared following grinding, polishing and etching. Also, two etching solutions were used: for DSS alloys, the electrochemical etching device type Struers LecroPol-5 was used in 45 gm potassium hydroxide (KOH) in 60 ml distilled water, applying 2.5 volt, for 4–5 s at room temperature. For austenitic stainless steel, immersion in a solution containing 10 ml hydrochloric acid (HCL), 5 ml nitric acid (HNO_3) and distilled water for 4 min was applied. Microstructural images were prepared for the as-received and heat-treated alloys at different temperatures and times under consideration in this study. Scanning electron microscope type JEOL-SEM 6400 is used to observe the microstructural changes.

A ferrite measurement device, type Ferritgehalt-messer, was used to detect the changes in the ferrite content (FC%) of the heat-treated alloys, compared with the as-received samples (Eghlimi *et al.*, 2013). The device shows the ferrite number (FN) based on an arbitrarily defined relationship between the magnetic susceptibility of the samples and the FC%. The FN is converted to FC% – as recommended by the device supplier – based on the following equation:

Alloys	Cr	Ni	C	Mn	Mo	Si	Cu	N
2304	23.15	3.40	0.03	0.60	0.3	0.26	0.26	0.09
2205	22.70	5.15	0.02	1.95	3.2	0.75	0.24	0.21
2507	24.70	7.15	0.02	1.05	3.8	0.35	0.20	0.32
316L	17.50	10.1	0.03	1.77	2.7	0.32	0.33	–

Table 1. Chemical composition in %wt of the used alloys

$$\text{Ferrite Content } \% = 7.30583 \times e^{\frac{FN}{8.88399}} - 7.162$$

Results

Table 2 shows the FC% values of the as-received and heat-treated alloys. The Hardness Rockwell C (HRC) machine type Emco was used to measure the hardness of the samples after they were ground and polished. Figure 1 shows the HRC hardness values of all the heat-treated samples; the first bar chart in each figure shows the hardness of the as-received condition of the alloy under consideration.

Duplex stainless steel alloy type 2304

Figure 2a shows the as-received microstructure, which consisted of both ferrite and austenite phases. Figures 2b, 2c, and 2d show microstructural images of samples that cover heat treatments from the lowest to highest temperatures for different times, which are 750°C for 10 min, 950°C for 1 h and 1,050°C for 4 h. The images show that no noticeable changes were observed in this alloy due to application of the heat treatments. Similarly, Table 2 shows that there are no large fluctuations associated with FC% due to the applied heat treatments. The as-received and heat-treated alloys at 1,050°C for 4 h had the highest FC% equal to 66.2%, while the minimum amount, equal to 54.8%, was observed at 850°C for all times. In terms of the hardness test, Figure 1a shows no large changes in hardness values by applying different heat treatments compared with the as-received alloy. The highest and lowest hardness values were 21 and 14 HRC, belonging to the heat-treated alloys at 750 and 1,050°C, respectively (both for 4 h). The hardness values for samples heated at 850 and 950°C for different times were either equal or slightly lower than the as-received alloy.

Duplex stainless steel alloy type 2205

Table 2 shows that FC% of the as-received 2205 alloy was 54.8%. This number was decreased by increasing the temperature and time, from 750 to 950°C; however, the lowest FC % was observed at 850°C for all times. At 1,050°C – for 10 min – the FC% started to increase and reached the highest amount 58.4% in 4 h. In return, the investigations for the microstructural images approved the occurrence of such changes. Figure 3 shows

Alloys	Temp. (°C)	Ferrite % Time 10 min	1 h	4 h
2304	750	66.2	54.8	54.8
as received = 66.2 % ferrite	850	54.8	54.8	54.8
	950	62.2	62.2	58.4
	1,050	62.2	62.2	66.2
2205	750	42.3	24.2	10.2
as received = 54.8 % ferrite	850	15.3	14.3	9.7
	950	28.1	28.1	15.1
	1,050	*54.8*	*43.4*	*58.4*
2507	750	45.2	37.0	10.2
as received = 66.2 % ferrite	850	18.0	16.0	14.0
	950	17.2	15.6	15.6
	1,050	*48.2*	*54.8*	*64.8*
316L	750	0.6	0.6	0.6
as received = 0.5 % ferrite	850	0.4	0.3	0.3
	950	0.4	0.2	0.2
	1,050	0.2	0.2	0.2

Table 2. Ferrite % of the as-received and heat-treated alloys

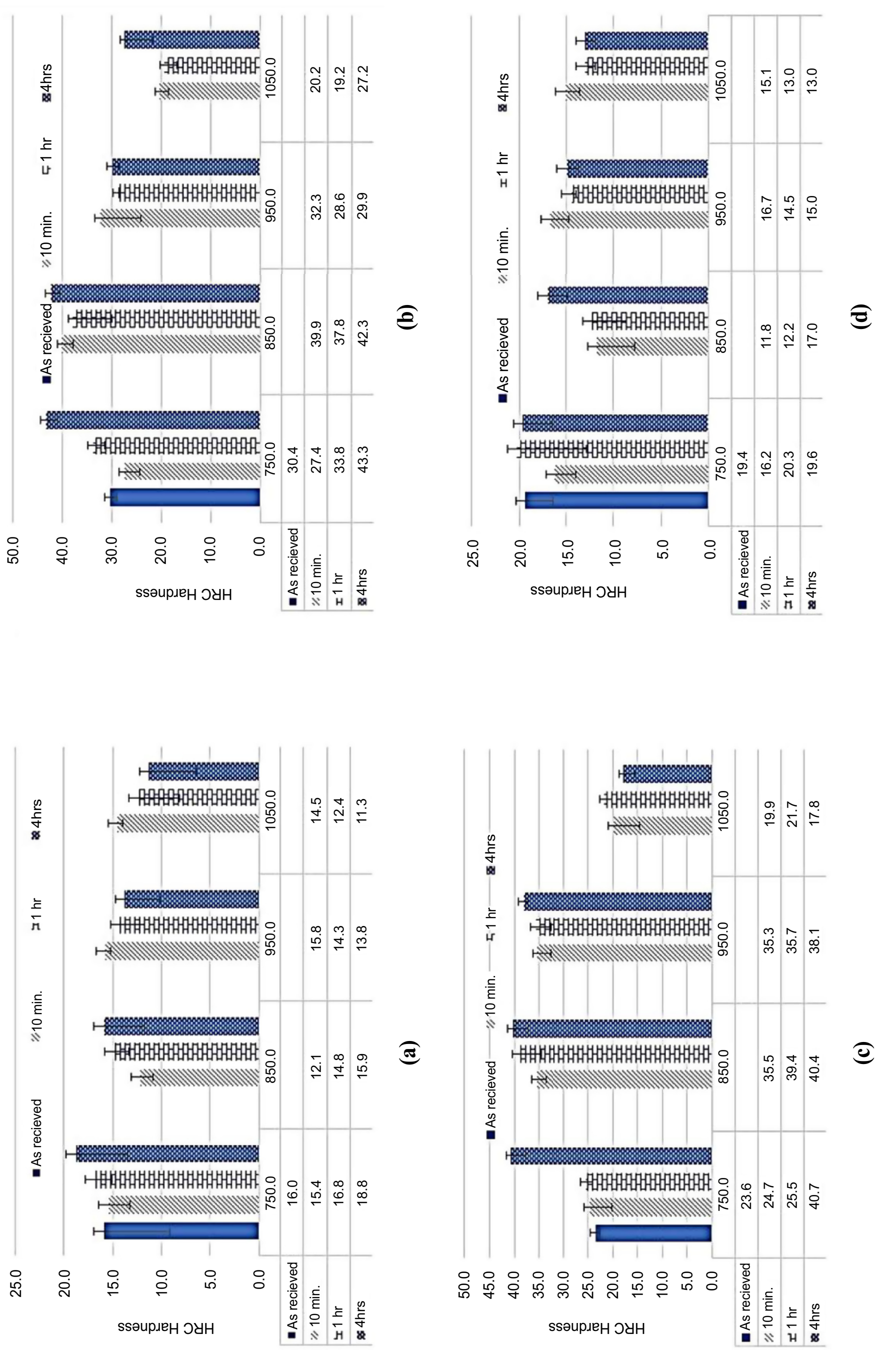

Figure 1.
Hardness Rockwell C values of as-received and heat-treated alloys at different temperatures and times: (a) 2304 alloy, (b) 2205 alloy, (c) 2507 alloy and (d) 316 L

microstructures of samples that were heat treated at different temperatures and times. Figure 3a shows the as-received DSS alloy type 2205, which almost shows a 50:50 ratio of the ferrite:austenite ratio. Figures 3b, 3c and 3d show the microstructure of heat-treated samples at 850°C for 10 min, 950°C for 1 h and 1,050°C for 4 h, respectively. Large similarities can be

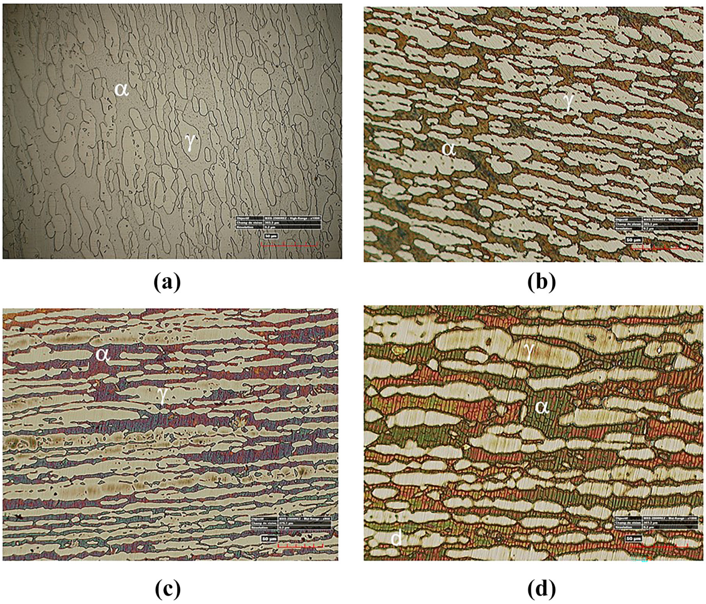

Figure 2.
The microstructure of duplex stainless steel 2304: (a) as received, (b) 750°C for 10 min, (c) 950°C for 1 h and (d) 1,050°C for 4 h

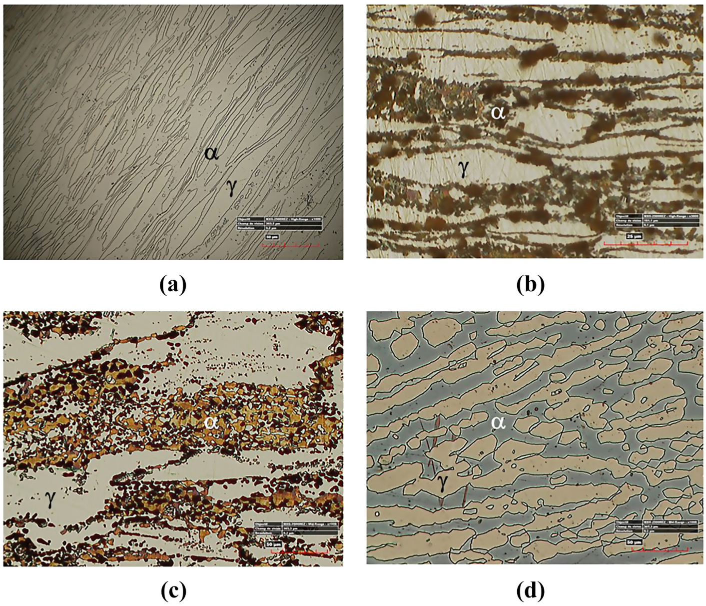

Figure 3.
The microstructure of duplex stainless steel 2205: (a) as received, (b) 850°C 10 min, (c) 950°C for 1 h and (d) 1,050°C for 4 h

seen between the as-received and heated-treated samples at 1,050°C, while the other samples heated at 850 and 950°C (especially for 4 h) showed the lowest FC% and their microstructures, see Figures 3b and 3c, show residual stress-free ferrite grains, shapes and sizes due to the occurrence of the recrystallization phenomenon (Alinejad *et al.*, 2016; Badji *et al.*, 2008; Ciuffini *et al.*, 2017; Vijayalakshmi *et al.*, 2011; Kashiwar *et al.*, 2012). On the other hand, Figure 1b shows that the hardness was increased to the highest values, around 43 HRC, during heating at 750 and 850°C (both for 4 h) – compared to the hardness value of the as-received condition which equals to 30 HRC. At 950°C, for all times, the hardness values were almost similar to the as-received alloy, while at 1,050°C, the hardness values were noticeably reduced to around 20 HRC, especially for 10 min and 1 h heating.

Super duplex stainless steel alloy type 2507

Table 2 shows that the FC% of the as-received SDSS alloy type 2507 was 66.2%. At 750°C, FC % has started the reduction with increasing the heating time, while at 850 and 950°C, the values were dramatically reduced, reaching 14%. At 1,050°C, the FC% was increasing with the time, almost recovered to reach the same value of the as-received alloy. The investigations of microstructural images support these changes, Figure 4a shows the as-received alloy of 2507, which shows a balanced ratio of both ferrite and austenite. Figures 4b, 4c and 4d show the microstructure of samples which were heat treated at 850°C for 10 min, 950°C for 1 h and 1,050°C for 4 h, respectively. These figures show that there are large similarities between the as-received and 1,050°C heat-treated samples, while the other samples, treated at the other temperatures, show different grain shapes and sizes. In terms of the hardness values, Figure 2c shows that the hardness value of the as-received 2507 alloy was 24 HRC, and the highest hardness values, around 40 HRC, were measured for samples heated at 850 and 950°C for all time intervals, also at 750°C but only for 4 h. Similar hardness values of the as-received alloy were obtained only at 750°C for 10 min and 1 h. All the samples heat treated at 1,050°C had hardness values lower than the as-received alloy, especially for 4 h was equal to 18 HRC.

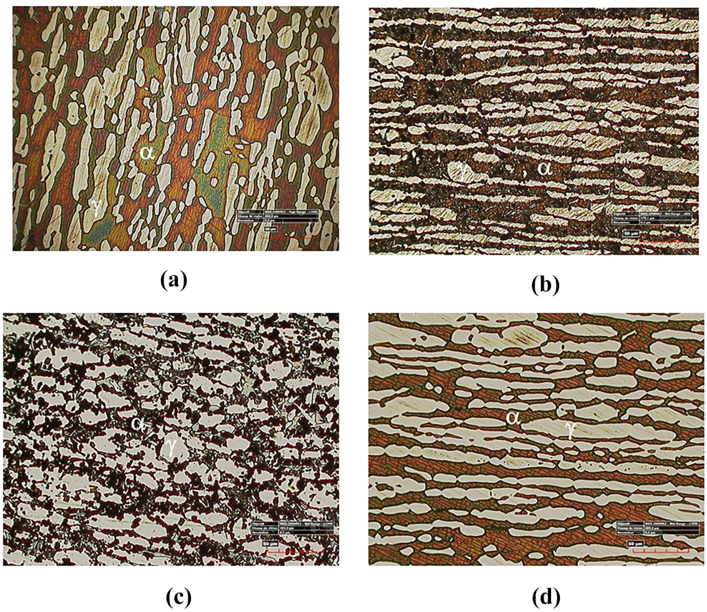

Figure 4.
The microstructure of duplex stainless steel 2507: (a) as received, (b) 850°C 10 min, (c) 950°C for 1 h and (d) 1,050°C for 4 h

Austenitic stainless steel alloy type 316L

In general, this type of alloy did not show noticeable changes – after applying the heat treatments – on the microstructure and hardness values. Figure 5a shows austenite as the microstructure of the as-received alloy, which is similar to Figure 5d, and the heat-treated sample at 1,050°C for 10 min. However, Figures 5b and 5c show the microstructure of the heat-treated alloys at 750 and 850°C – both for 1 h – which show carbide precipitation on the grain boundaries, which is attributed to passing the alloy through the sensitization range (Khoshnaw *et al.*, 2019). In terms of FC%, Table 2 shows that, because the microstructure is austenitic, almost no ferrite was observed as the highest FC% did not exceed 0.6%. Figure 2d shows that the hardness of the as-received alloy was 19 HRC, the hardness values were decreased for samples heated at higher temperatures and longer time, and the lowest hardness value, 12 HRC, was observed at 850°C for 10 min and 1 h.

Discussion

The results indicated that the response of the alloys to the applied thermal treatments used in this study was different from one alloy to another. Table 2 showed that the austenitic stainless steel type 316L showed the least or almost no response to the heat treatments than DSS type 2304, while the highest and effective responses to retrieve the original phases were observed in alloys 2205 and 2507. Figure 6 shows that DSS alloy type 2304, which is a two phase-based alloy, showed a steady balance of both austenite and ferrite, regardless of the applied heat treatments as heating at high temperatures did not lead the alloy to change significantly. Table 2 and Figure 6 show that the FC% equals to 54.8 for the samples heated to 750°C (1 h and 4 h) and 850°C for all times, while the heating at 1,050 for 4 h brought the FC% similar to the ratio of the as-received sample, which was equal to 62.2%. Figure 6 shows that both 2205 and 2507 alloys behave in a similar way, after heating at 750°C, the FC

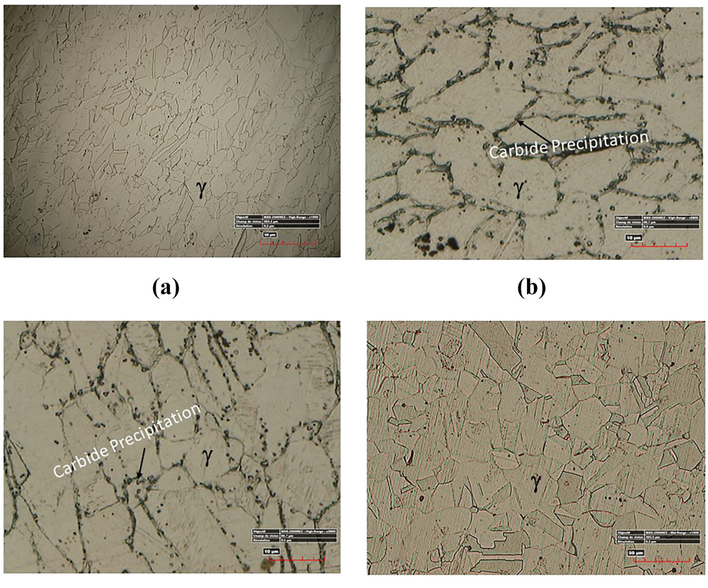

Figure 5.
The microstructure of austenitic stainless steel type 316L: (a) as received, (b) 750°C for 1 h, (c) 850°C for 1 h, (d) 1,050°C for 10 min

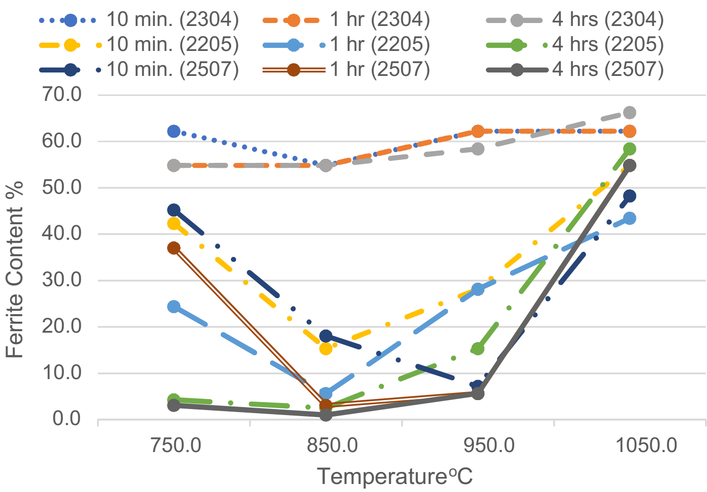

Figure 6.
Ferrite content % changes vs the applied heat treatments

% started to decrease, though 2507 took longer time than 2205 to start this reduction and this can be attributed to the chemical composition, that is, higher Cr%, see Table 1. Both alloys at 850°C show lower FC% than as-received alloys, then the FC% starts to increase again at 950°C and by reaching 1,050°C, the amount of FC% was almost similar to that of the as-received alloys.

In terms of the hardness results, Figure 1 showed that the heat treatments did not change the hardness values significantly as the lowest hardness values were observed on the samples which were heat treated at 850 and 1,050°C.

More microstructural investigations were carried out to build a correlation between the FC% and hardness values for the heat-treated 2304 alloys. Figure 7a shows a type of intermetallic compound phase for a sample heat treated at 850°C for 10 min, which showed the lowest FC% and hardness value. Although the intermetallic compounds are known, generally, as brittle phases, consequently lead to the high hardness; researchers (Argandoña *et al.*, 2017; Pettersson *et al.*, 2015) found that this is not always happening, especially when the other factors, such as grain size and surface roughness, are interacting together. On the other hand, Figure 7b shows the recrystallization phenomenon through reinitiating the ferrite phase on the austenite grain boundaries for the sample heated at 1,050°C for 4 h. Therefore, the low hardness values at this particular temperature can be attributed to the "recrystallization" or re-initiation residual stress-free ferrite phase in the alloy.

The heat treatments which were applied in this study showed the most significant effects on both DSS alloy type 2205 and SDSS alloy type 2507 to retrieve their microstructures. In general, the results indicated noticeable similarities between these two alloys, for example, the FC% decreased with heating to higher temperatures, mainly at 850 and 950°C, then at 1,050°C, the FC% was noticeably increased, which almost reached the as-received amounts, while – at these particular temperatures – the hardness values for both alloys reached the minimum. Table 2 and Figure 2 shows that the highest hardness of 2205 and 2507 alloys was observed for the samples which were heat treated at 750 and 850 °C (for different times), which had the lowest ferrite contents. While the lowest hardness value was for the samples – which recovered their FC% – heated to the highest temperature at 1,050°C almost all times. This correlation can be attributed to the reinitiation, due to the recrystallization phenomenon, of new free residual stress ferrite grains.

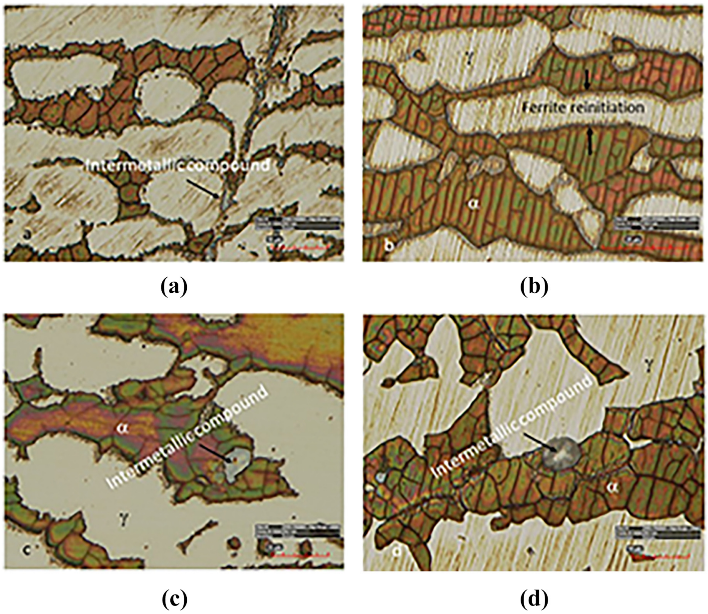

Figure 7.
Microstructure and intermetallic compounds: (a) alloy 2304 heated at 850°C for 10 min, (b) alloy 2304 heated at 1,050°C for 4 h, (c) alloy 2205 heated at 750°C for 10 min and (d) alloy 2,507 heated at 850°C for 10 min

On the other hand, in terms of increasing the hardness values with heating these two alloys, Figures 7c and 7d show the microstructures of 2205 and 2507 heated for 10 min at 750 and 850 °C, respectively. The figures show the initiation of intermetallic compounds which can be the reason behind increasing the hardness. The interpretation about the interrelation between the intermetallic phase, FC% and hardness is not a straightforward approach as researchers noticed such controversial results and have been concluded that the intermetallic compounds and/or brittle phases, such as sigma phase, may present a wide range of hardness values due to the complexity of their structures. Moreover, the hardness results are dependent on the surface finish, which affects the scattering of the results concerning the average value (Guimarães and Mei, 2004; Walter and Mitterer, 2009).

For the austenitic alloy, the results indicated that the microstructure remained stable because the chemical composition of this group of alloys maintains the austenitic phase at room temperature and at high temperatures, except the carbide precipitation phenomenon which occurs at sensitization ranges, see Figure 5. This stable austenite, due to the high Ni and N contents, see Table 1, does not allow heating to a high temperature to change the microstructure, for example, to ferrite. In terms of the hardness values, Figure 1d showed that there are no large changes that happened in the hardness values between the heat-treated samples compared with the as-received alloy. The lowest hardness value at 750 and 850°C can be attributed to the recovery and recrystallization phenomena, forming new grains with free residual stresses due to passing the samples through these particular temperatures (Dehghan-Manshadi *et al.*, 2008).

Conclusions

(1) The alloys, 2205 and 2507, have completely recovered their ferrite:austenite ratio through heating to 1,050°C for all times.

(2) The recovered samples showed low hardness values, which was attributed to the initiation of new ferrite phases, free residual stress grains and/or due to the increase in the austenite phase and dilution of the intermetallics during the heat treatment.

(3) Alloy type 2304 showed noticeable stability of FC% as the ferrite:austenite ratio remained steady.

(4) Austenitic stainless steel did not show changes, except Cr carbide precipitation by passing through sensitization ranges of temperatures at 750 and 850°C.

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