AN OVERVIEW OF SIGMA PHASE FORMATION IN 317L GRADE AUSTENITIC STAINLESS STEEL

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Abstract

Austenitic Stainless Steel is known as responsible for the largest manufacturing share of stainless steel. Different processes, as well as the addition of alloying elements affects the microstructure, which provides specific properties to each steel grade. One of the microstructural characteristics that affects the austenitic stainless steels properties is related to the intermetallic phases, especially the sigma phase. Its presence and distribution on the microstructure may impair several characteristics of the alloy, such as the reduction of the tenacity and ductility, generating important discussions on engineering. The present paper aims to overview the effects of the presence of the sigma phase in 317L grade austenitic stainless steel, welded, without and with heat treatment at 850 °C and 1080 °C with different exposure times. The effect of the heat treatments was followed by the mechanical tests of hardness and tensile strength. The investigation of the evolution of the microstructure was accompanied by metallographic analysis in samples in the original condition and after heat treatment. The microstructure was revealed by means of electrolytic etching, with subsequent analysis by optical microscopy, scanning electron microscopy and energy dispersive spectroscopy microanalysis techniques. The presence of stabilizing elements of ferrite, chromium and molybdenum tend to accelerate the precipitation of the sigma phase at 850 °C, even at short exposure intervals at this temperature. It was observed a greater hardening, by the precipitation of the sigma phase in the welded region, emphasizing also that its presence is related to a decrease of the resistance and ductility

Keywords: Austenitic stainless steel, delta ferrite, sigma phase, welding

1. INTRODUCTION

Among the main categories of stainless steels, one may be highlighted as the most produced by the industry and for this reason one of the most studied, the austenitic. Different process as well as well as the addition of alloying element affects the microstructure, which provides specific properties to each steel grade. One of the microstructural characteristics that affects the austenitic stainless steels properties is related to the intermetallic phases, especially the sigma phase (σ). The σ phase is a body-centered tetragonal (BCT), with 30 atoms per unit cell and its precipitated between 600 °C up to 1000 °C [1] may have increase the hardness and decrease the ductility [2], it's non-magnetic and thermodynamically stable up to 950 °C, temperature that its maximum volumetric fraction may be observed [3]. The σ phase formation tend to occur from delta ferrite (δ) presented on the microstructure [4], which may be related to the fact of diffusion rates of chromium (Cr) are lower on this phase than austenite [5]. Beyond its precipitation from δ ferrite, there is also the eutectoid transformation of δ ferrite, which when transformed into σ phase induces the impoverishment of Cr and molybdenum (Mo) contents, the same stabilizers elements of δ ferrite, therefore, having in this reaction another phase with low contents of Cr and Mo, however with residual nickel (Ni) from δ ferrite, therefore having $\delta \rightarrow \sigma + \gamma_2$ [6]. The phase γ_2, it is called as secondary austenite due of its chemical composition, which is different from original austenite γ [7]. And the last mechanism known tends to occur after δ ferrite consumption, precipitating from austenite, once this phase also has Cr and Mo, but the reaction are much slower due to the low diffusivity rates of the elements into the compact structure of austenite [8].

The **Figure 1** shows a 304 grade austenitic stainless steel aged at 650 °C up to 31000 h, evidencing the transformation $\delta \rightarrow \sigma + \gamma_2$ [6]. σ phase morphology varies in according to the temperature that its exposed, through the **Figure 2** morphological changes on the microstructure may be observed at different temperature rates in a duplex stainless steel S31803 [9].

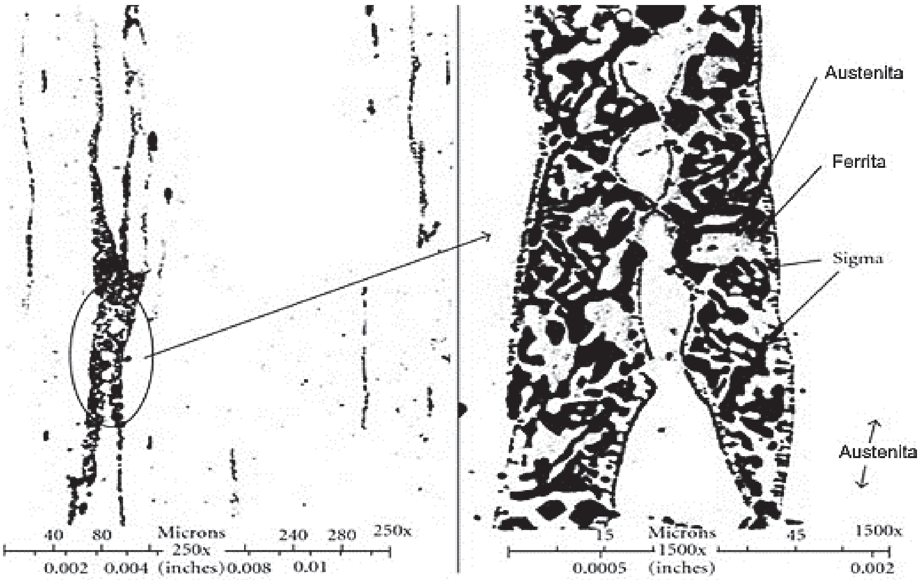

Figure 1 AISI 304 aged at 650 °C up to 31000h, magnetic etched [6]

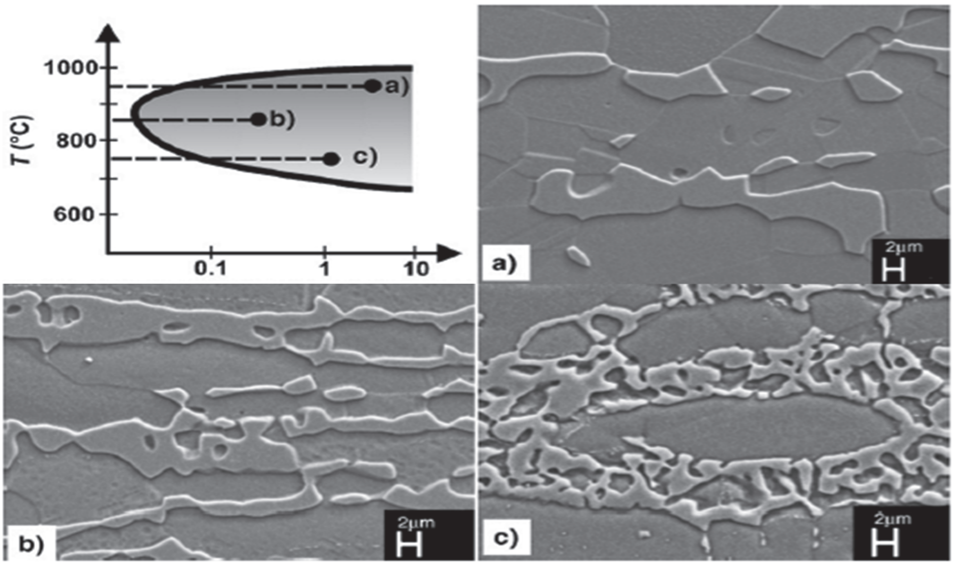

Figure 2 Morphology of the sigma phase with respect to the isothermal annealing temperature, S31803 DSS; (a) 950 °C, (b) 850 °C, (c) 750 °C [9]

317L grade austenitic stainless steel is well known and applied due to its higher corrosion resistance, especially due to the % wt. % of Mo, but whose presence tends to increase the precipitation rates of σ phase, especially when exposed at 650 °C and 870 °C [3]. **Figure 3** highlights to the σ phase formation in a rolled 316L grade austenitic stainless steel evidencing its precipitation from δ ferrite, its morphology, the contrast between them and the brownish appearance [10].

In this context, the continuous study upon the effects of σ phase on austenitic stainless steels, both in relation to the microstructure and mechanical properties as well as the heat treatment, is deemed opportune.

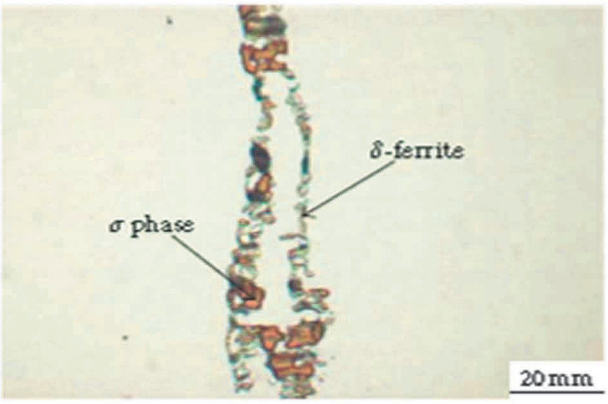

Figure 3 AISI 316L showing σ phase formation from δ ferrite [10]

2. MATERIALS AND METHODS

The austenitic stainless steel 317L was welded by gas tungsten arc welding (GTAW) process. The chemical composition of base metal, weld consumable and weld metal, by means of x-ray spectroscopy, are shown into the **Tables 1, 2** and **3**, respectively.

Table 1 Chemical composition of base metal

wt. %	C	Mn	Si	P	S	Cr	Ni	Mo
AISI 317 L	0.018	1.42	0.85	0.017	0.02	17.91	11.31	3.16

Table 2 Chemical composition of weld consumable

wt. %	C	Mn	Si	P	S	Cr	Ni	Mo
AISI 317 L	0.010	1.50	0.44	0.022	0.010	18.70	13.57	3.54

Table 3 Chemical composition of weld metal

wt. %	C	Mn	Si	P	S	Cr	Ni	Mo
AISI 317 L	0.021	1.54	0.69	0.018	0.015	18.58	12.58	3.02

All the samples (before and after heat treatment) were chemically etched by means of sodium hydroxide (NaOH - 40 % w / w) using 3V during 10s.

Heat treatment have been conducted on two different temperatures, 850 °C and 1080 °C, in order to check the effects on the microstructure and mechanical properties, by means of tensile test (only samples heat treated at 15 and 120 min were tested). The samples were heat treated at 5 min / 2.5 mm [10], starting from 7 min, doubling the condition of it, up to 480 min, therefore, 7 samples for each temperature. By means of a ferritescope the average of δ ferrite, ferrite number (FN), were determined for each condition, being this related to the σ phase precipitation on the microstructure. By means of optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) the assessments were completed and concluded.

3. RESULTS AND DISCUSSION

The microstructure on the welded regions presented higher ferrite contents, with a random distribution in the austenitic matrix and different morphologies compared to the base metal, due to the dendritic solute redistribution related to the cooling rate of the welding process.

During the heat treatment at 850 °C, even short times of exposure were sufficient to start the ferrite decomposition, probably associated to the σ phase. **Figure 4** shows "σ phase" precipitated at 7 and 15 minutes, respectively.

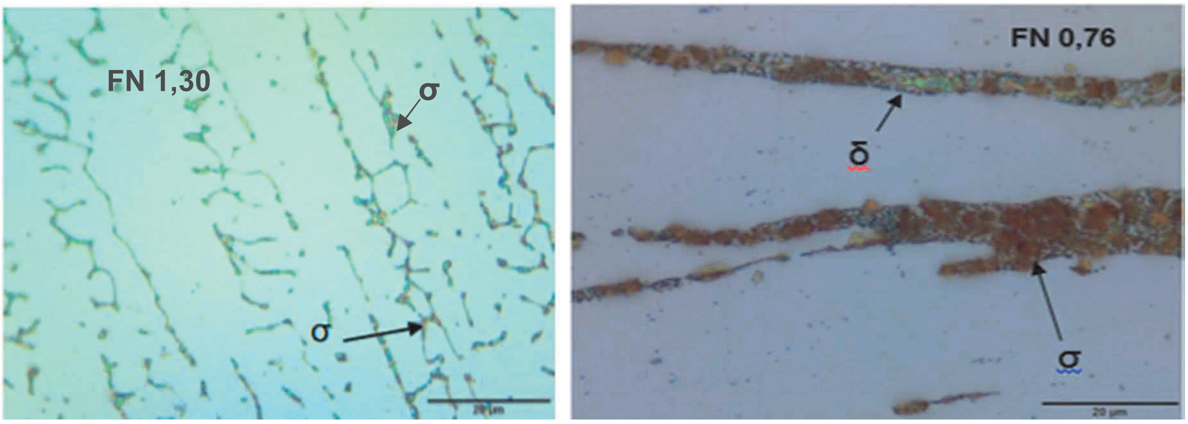

Figure 4 Welded zone after 7 min exposure and transition, weld/base metal, at 15 min of exposure

By increasing the heat treatment time morphological changes may been observed, such as coral-like [16], without changes on its volume fraction, which means the precipitation is accelerated in the presence of δ ferrite and then it gets stable or so when the initial amount get consumed. **Figure 5** shows an EDS analyzes together with an OM image, where it may be seen that no morphological changes was observed compared to the beginning of heat treatment, but it's possible to observe through **Tables 4** and **5** that δ ferrite have been consumed at all and now might have the reaction from $\gamma_2 \rightarrow \sigma$.

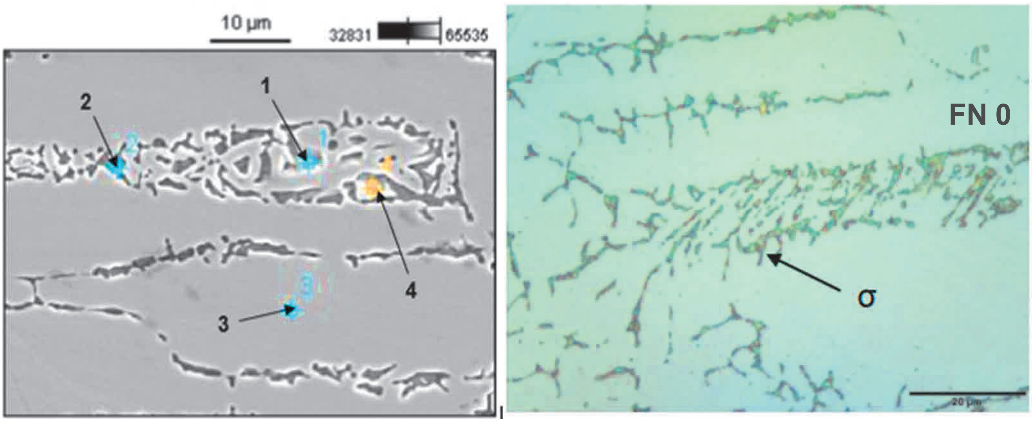

Figure 5 AISI 317L during EDS analyzes and OM image on the transition region, weld to base metal

Table 4 EDS analyzes at 850 °C on 240 minutes of exposure

Element (w %. %t)	Point 01	Point 02	Point 03	Point 04
Cr	22.68	22.83	18.82	23.18
Mn	--	1.42	1.48	--
Fe	67.14	66.52	67.64	66.62
Ni	6.16	5.47	9.49	5.58
Mo	4.02	3.77	2.57	4.62

Table 5 Average of ferrite number (FN) by means of ferritescope at 850 °C with different time of exposure

Exposure time (minutes)							
0	7	15	30	60	120	240	480
Average FN in the weld metal - Heat treatment 850 °C							
8.11	1.30	0.76	0.42	0.16	0	0	0
Average FN in the base metal - Heat treatment 850 °C							
4.94	1.13	0.56	0.27	0.13	0	0	0

EDS analyzes in AISI 317L, shown the σ phase formation starting from δ ferrite content, highlighted by points 1, 2 and 4, showing a higher content of Cr, compared to the point 3, beyond the Mo contents, which once again suggest that Mo has an important role on σ phase precipitation, especially when δ ferrite it's presented. In addition, it is possible to observe that the δ ferrite it is totally consumed in about 120 min, but even short intervals it has a great decrease, confirming the σ phase as shown on **Figure 4**.Heat treatment at 1080 °C did not present any σ phase precipitation as expected. But an important value may be observed through the **Table 6**, δ ferrite content were not totally consumed in the base metal, which may be related to the morphology in this area and its distribution.

Table 6 Average of ferrite number (FN) by means of ferritescope at 1080 °C with different time of exposure

Exposure time (minutes)							
0	7	15	30	60	120	240	480
Average FN in the weld metal - Heat treatment 1080 °C							
8.11	2.54	2.07	1.41	0.89	0.42	0.17	0
Average FN in the base metal - Heat treatment 1080 °C							
4.94	1.62	3.34	3.17	2.87	2.62	2.36	1.80

In **Table 7**, may be observed that the samples with higher contents of δ ferrite, without treatment, have got the higher tensile strength, followed by those with σ phase precipitation (850 °C), which also may increases the tensile strength.

Table 7 Mechanical properties by means of tensile test

	Without HT	850 °C - 15 min	850 °C - 120 min	1080 °C - 15 min	1080 °C - 120 min
Tensile strength (MPa)	677.44	663.13	640.56	579.97	582.88
Elongation (mm)	11.61	11.98	11.80	13.5	14.82
Area reduction (%)	64.52	29.06	28.38	65.52	72.1

Based upon **Table 6** data, may be observed that the presence of δ ferrite, at 850 °C, increases the tensile strength to the detriment of ductility, but its influence is still lower than that of σ phase. The latter evidenced in the small reduction of area that the materials presented, besides to the fact that no uniform behavior during the tensile test was observed, especially on the welded region, highlighting to the strain hardening on this region. The material heat treated at 1080 °C despite to present lower tensile strength rates, higher ductility may be observed, which may be related to the dissolution of δ ferrite in the matrix and no presence of σ phase. Results of hardness test comparing to the δ ferrite content, FN are shown in **Table 8.** It's evidenced that the FN at 850 °C have been reduced on base and weld metal, as well as the hardness have been increased, at

1080 °C the FN have been decreased, but with no significate changes on the hardness, compared to the original without any heat treatment after welding, 170 Vickers hardness (VH).

Table 8 Vickers hardness, VH, 1000g

	Hardness (VH)		σ Ferrite content (FN)	
Heat treatment	Weld metal	Base metal	Weld metal	Base metal
1080 °C - 156 min	193	174	1.85	4.653
1080 °C - 120 min	153	180	0.68	3.02
850 °C - 15 min	248	224	1.04	1.506
850 °C - 120 min	225	227	0.15	0.11

4. CONCLUSION

Based upon the studies carried out, as per technical references as well as the results found through the techniques used it's possible to conclude that the 317L grade austenitic stainless steel tends to precipitate σ phase at 850 °C even at short times of exposure, which might indicate that its precipitation it's related to the presence of δ ferrite on the microstructure, being it the first mechanisms to precipitate σ phase, once the same elements that stabilizes σ phase are those that stabilizes δ ferrite, especially chromium and molybdenum. In addition, molybdenum shows an important role as on the σ phase precipitation at 850 °C and δ ferrite formation as at 1080 °C to retard the δ ferrite's reduction. A strain hardening was observed on the welded region, which may suggest by comparing the microstructures' morphology (base vs welded metal), that it's dimension and distribution all over the matrix may influence on material's behavior, once dendrites formed after weld solidification hadn't a pattern, being neither organized nor spaced among each other, which tends to difficult the discordance movement comparing to an organized region, such as the lamellar structure found on base metal. Results might also suggest, but it has to be more studied, that σ phase's presence on the microstructure may not affect the mechanical behavior as bad as it was supposed to, apparently related to its disposition and morphology in the matrix

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