INFLUENCE OF HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SUS 316L ALLOY

Jaromír DRÁPALA, Gabriela KOSTIUKOVÁ, Monika LOSERTOVÁ

VSB - Technical University of Ostrava, Ostrava, Czech Republic, EU
Jaromir.Drapala@vsb.cz

Abstract

Grade 316L is the low carbon austenitic stainless steel with molybdenum content and is immune from sensitisation (grain boundary carbide precipitation). Thus it is extensively used in heavy gauge welded components. The austenitic structure also gives excellent toughness, even down to cryogenic temperatures. The molybdenum gives 316L good overall corrosion resistant properties, particularly higher resistance to pitting and crevice corrosion in chloride environments. Compared to chromium-nickel austenitic stainless steels, 316L stainless steel offers higher creep, stress to rupture and tensile strength at elevated temperatures. These properties are specified for flat rolled product (plate, sheet, pipe, bar and coil) in ASTM A240/A240M.

Thermal treatment of SUS 316L alloys allows significantly influence microstructure and mechanical properties. The aim of this work was the study of microstructure of SUS 316L before and after annealing. The heat regimes were performed at these conditions: 1050, 1100 and 1150 °C for 20 minutes followed by slow cooling in vacuum. Microstructures features were studied by means of optical and scanning electron microscopies, EDX microanalysis and microhardness measurement. Higher aging temperatures and annealing time led to decreasing microhardness and microstructure changes (grain growth).

Keywords: Alloy SUS 316L, heat treatment, microstructure, EDX analysis, microhardness

1. INTRODUCTION

The austenitic stainless steel grade 316L is the low carbon standard molybdenum-bearing alloy. The molybdenum gives good overall corrosion resistant properties, particularly higher resistance to pitting and crevice corrosion in chloride environments. Grade 316L is immune from sensitisation (grain boundary carbide precipitation). Thus it is extensively used in heavy gauge welded components (over about 6 mm). The austenitic structure also gives this grade excellent toughness, even down to cryogenic temperatures, 316L offers higher creep, stress to rupture and tensile strength at elevated temperatures [1]. In addition to excellent corrosion resistance and strength properties, types 316 and 316L alloys also provide the excellent fabricability and formability which is typical of the austenitic stainless steels.

Chemical composition and typical mechanical properties of 316L are presented in **Table 1** and **Table 2.**

Table 1 Composition ranges for 316L stainless steels (wt.%) [1, 2]

Grade	C	Mn	Si	P	S	Cr	Mo	Ni	N
316L	<0.03	<2	<1	<0.045	<0.03	16-18.5	2-3	10-14	<0.1

Table 2 Typical mechanical properties of 316L stainless steels [1]

Grade	Yield strength **0.2% min.** (MPa)	Tensile strength **min.** (MPa)	Elongation **min.** (% in 50 mm)	Hardness Rockwell **max.** (HRB)	Hardness Brinell **max.** (HB)
316L	170	485	40	95	217

Grade 316 has good oxidation resistance in intermittent service to 870 °C and in continuous service to 925 °C. Grade 316L is resistant to carbide precipitation and can be used in the above temperature range. It has excellent weldability by all standard fusion and resistance methods, both with and without filler metals. Fully austenitic weld deposits are more susceptible to cracking during welding. For this reason Types 316 and 316L "matching" filler metals are formulated to solidify with a small amount of ferrite in the microstructure to minimize cracking susceptibility. 316L stainless steel tends to work harden if machined too quickly. For this reason low speeds and constant feed rates are recommended. 316L stainless steel is also easier to machine compared to 316 stainless steel due its lower carbon content.

316L stainless steel can be hot worked using most common hot working techniques. Optimal hot working temperatures should be in the range 1150-1260 °C, and certainly should not be less than 930 °C. Post work annealing should be carried out to induce maximum corrosion resistance. Most common cold working operations such as shearing, drawing and stamping can be performed on 316L stainless steel. Post work annealing should be carried out to remove internal stresses. 316L stainless steel does not harden in response to heat treatments. It can be hardened by cold working, which can also result in increased strength.

The austenitic stainless steel 316L is provided in the mill annealed condition ready for use. Heat treatment may be necessary during or after fabrication to remove the effects of cold forming or to dissolve precipitated chromium carbides resulting from thermal exposures. For the Type 316 alloy the solution anneal is accomplished by heating in the 1040 to 1175 °C temperature range followed by air cooling or a water quench, depending on section thickness. Cooling should be sufficiently rapid through the 816 - 427 °C range to avoid re-precipitation of chromium carbides and provide optimum corrosion resistance. In every case, the metal should be cooled from the annealing temperature to black heat in less than three minutes. Type 316 cannot be hardened by heat treatment [2].

Typical applications of the 316L include: food preparation equipment particularly in chloride environments, pharmaceuticals, marine and architectural applications, fasteners, medical implants, including pins, screws and orthopaedic implants like total hip and knee replacements [2]. Next information on the effect of heat treatment on microstructure, mechanical and other properties of austenitic stainless steel 316L you can find in [3-7] and elsewhere.

Thermal treatment of SUS 316L alloys allows significantly influence microstructure and mechanical properties. The aim of this work is the study of microstructure and mechanical properties of 316L alloy before and after thermal treatment.

2. EXPERIMENT

The initial material for the experiments were 316L thin tubes with outside diameters 8 mm (thickness 0.42 mm) delivered by Continental Automotive Czech Republic s.r.o. One batch of samples (No. 1) was delivered in a condition after air-annealing (the oxide layer on a surface of the tubes), the second batch of samples (No. 2) was in a condition after deformation forming. For experimental high vacuum annealing of the samples the following heat treatment modes were used - see **Table 3**:

Table 3 Regimes for heat treatment of 316L tubes

Regime	Heating	Pre-annealing	Annealing	Cooling	Samples No.
A	12 °C / min	---	1100 °C / 20 min	Free in the furnace	**1, 2**
B	30 °C / min	---	1050 °C / 20 min	Free in the furnace	**2**
C	24 °C / min	----	1150 °C / 20 min	Free in the furnace	**2**
D	24 °C / min	800 °C / 10 min	1050 °C / 20 min	Free in the furnace	**2**

Figure 1 presents as an example a histogram of regime B for sample No. 2.

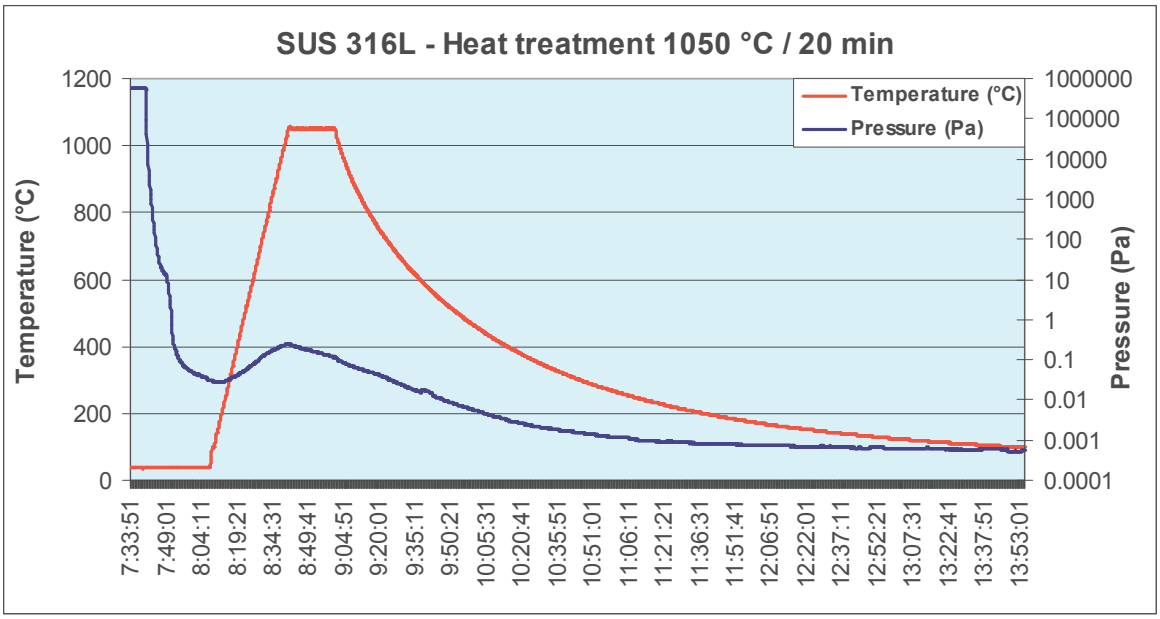


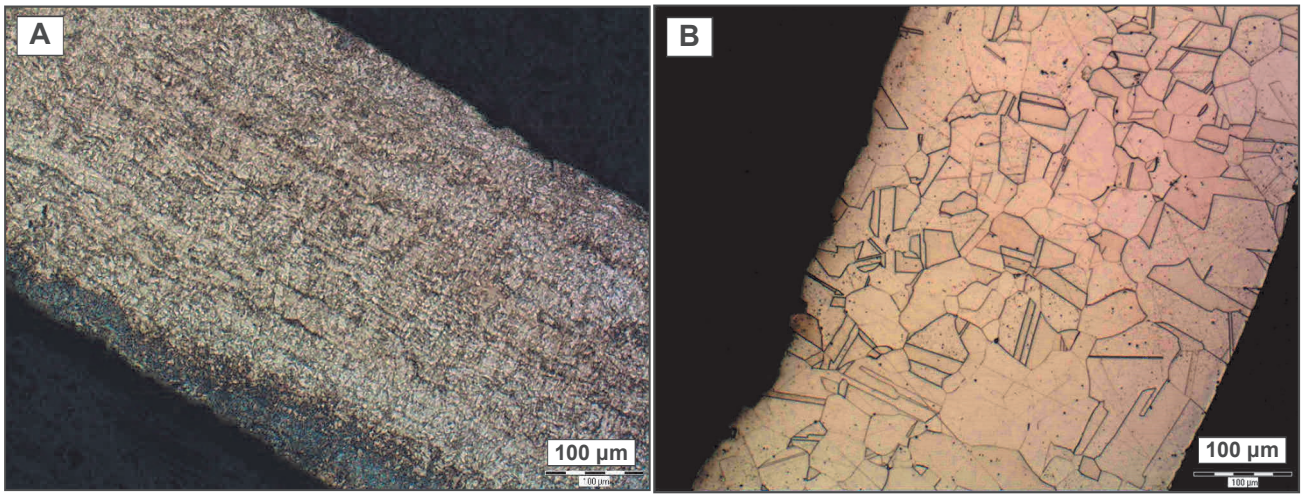
Figure 1 Regime B of heat treatment for sample No 2

The prepared metallographic specimens were studied using the optical method (OLYMPUS GX51 inverted metallographic microscope equipped with OLYMPUS DP12 digital camera). The microstructure and phase analysis was performed using the SEM/EDX method (a scanning electron microscope, JEOL JSM-6490LV type equipped with INCA x-act analyzer). FUTURE-TECH FM-100 automatic microhardness tester with FM-ARS900 control unit was used to determine the Vickers microhardness. The microhardness $HV_{0.1}$ was measured through 5 indents across the wall of tube from inner to outer surface of the specimen at 10 g load.

3. RESULTS

3.1. Microstructure analyses

Figure 2 shows images of the selected specimens after different heat treatment regimes, both in the initial state and after high-temperature annealing in various conditions.



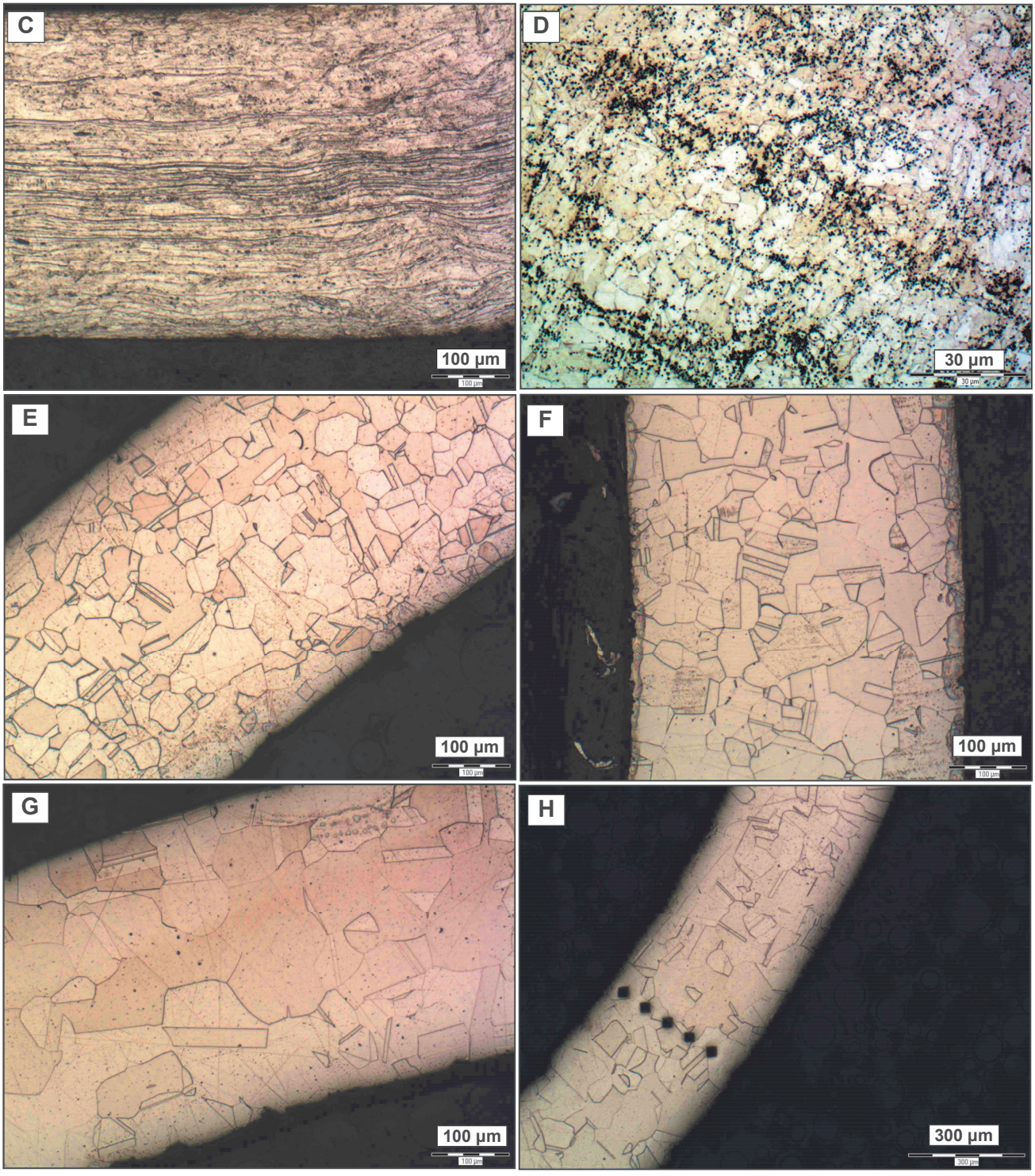


Figure 2 Microstructures of 316L tubes after various conditions of heat treatment. A - sample No. 1 after air-heating - delivered state (longitudinal section), B - sample No. 1 after annealing 1100°C/20 min in vacuum (cross section), C (longitudinal section) and D (cross section) - sample No. 2 after cold working operations - delivered state, E, F, G, H - sample No. 2 after vacuum annealing: E - 1050 °C / 20 min; F - 1100 °C / 20 min; G and H - 1150 °C / 20 min. The microstructure was developed using chemical etching in a solution of 50 ml H_2O + 50 ml HCl + 5 ml HNO_3 acids. In **Figure 2H** are visible marks after Vickers microhardness measurements.

3.2. Grain sizes and microhardness

Based on the metallographic images, for all the samples the mean grain size was determined including the group classification according to ČSN EN ISO 643. Further, microhardness of the samples was measured in the delivered condition and after different conditions of vacuum annealing with subsequent free cooling in a vacuum furnace - see **Table 4.**

Table 4 Grain sizes and microhardness $HV_{0.1}$ (average value from 5 measurements) of 316L after various regimes of heat treatments

Sample No.	**1**		**2**				
Conditions	Delivered state	1100 °C 20 min	Delivered state	1050 °C 20 min	1100 °C 20 min	1150 °C 20 min	**800 °C/10 min + 1050 °C/20 min**
Figure	2A	2B	2C, 2D	2E	2F	2G, 2H	
Grain sizes (μm)	4.1 ± 1.6	43 ± 17	33 ± 13	42 ± 14	42 ± 12	89 ± 24	50 ± 13
Grain size by ČSN EN ISO 643	G 12-13	G 5-6	G 6-7	G 6	G 6	G 4	G 5-6
Microhardness $HV_{0.1}$	192 ± 6	169 ± 9	424 ± 15	132 ± 3	198 ± 19	133 ± 7	140 ± 6

3.3. SEM/EDX chemical microanalysis

In **Table 5** are presented results of the chemical analysis for sample 2 in the delivered state. The oxidic Cr_2O_3 + FeO and sulphide inclusions of MnS type were accidentally found in the microstructure.

Table 5 Results of chemical analysis of used 316L steel

Element	**Si**	**Cr**	**Mn**	**Fe**	**Ni**	**Mo**
(wt%)	1.07	19.61	1.83	63.44	12.25	1.78

4. DISCUSSION

Vacuum annealing of stainless tubes with a diameter of 8 mm and a wall thickness of 0.42 mm was carried out in order to reduce the delivered material hardness from the original values of about 420 $HV_{0.1}$ to a required value from 140 to 180 $HV_{0.1}$. The tubes after soft annealing were intended for following processing by mechanical forming to final products.

In order to optimize the vacuum annealing technology, several modes were proposed - see **Table 3.** It was found out that both rise time to the annealing temperature and the annealing temperature itself affect structural and mechanical properties of 316L alloy. The annealing time was the same in all the experiments, i.e. 20 minutes. At the high 1150 °C temperature of annealing the grain grow to an average size of approximately 90 μm occurred, whereas more or less the same microhardness was found out both at the annealing temperature of 1050 °C and 1150 °C. Annealing resulted in the total recrystallization of the samples leading to formation of new polygonal-shaped grains. Annealing twins were also found out here - see **Figures 2B, 2E, 2F** and **2G.**

As a result of partial findings the following optimal mode for vacuum annealing of samples was proposed:

1) Rise to the temperature of 800 °C at a rate of 24 °C/min,
2) Holding on 800 °C temperature for a period of 10 minutes,
3) Rise to the temperature of 1050 °C at a rate of 24 °C/min,
4) Holding on 1050 °C temperature for a period of 20 minutes,
5) Free cooling in a furnace under vacuum.

The vacuum during the high-temperature annealing period must be better than 0.1 Pa.

The resulting structural properties of such annealed samples feature required parameters in term of grain sizes and their morphology. Also the microhardness values are within the demanded range, which is required for subsequent mechanical cold processing of the tubes. Holding on 800 °C is necessary to maintain the demanded vacuum level under 0.1 Pa for the whole time of annealing.

The declared occurrence of chromium carbides [2] at exceeding the temperatures between 800 to 450 °C (cooling) was not observed in our experiment either in the metallographic images or in SEM/EDX analysis.

5. CONCLUSIONS

The aim of the work was to propose an optimal heat treatment mode for SUS 316L alloy in a form of thin-walled tubes. On the basis of verifying experiments the following high vacuum annealing process was proposed:

Rise to the temperature of 800 °C at a rate of 0.4 °C/s ➔ holding on 800 °C temperature for a period of 10 minutes ➔ rise to the temperature of 1050 °C at a rate of 0.4 °C/s ➔ holding on 1050 °C temperature for a period of 20 minutes ➔ free cooling of the samples under vacuum.

This mode ensures the required grain size of G 5-6 value according to ČSN EN ISO 643 and microhardness $HV_{0.1}$ on a level of 140 ± 10.

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