

The effect of annealing time on microstructure and impact energy of stainless steel AISI 316L

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18th INTERNATIONAL FOUNDRYMEN CONFERENCE

**Coexistence of material science and sustainable
technology in economic growth**



Sisak, May 15th – 17th, 2019

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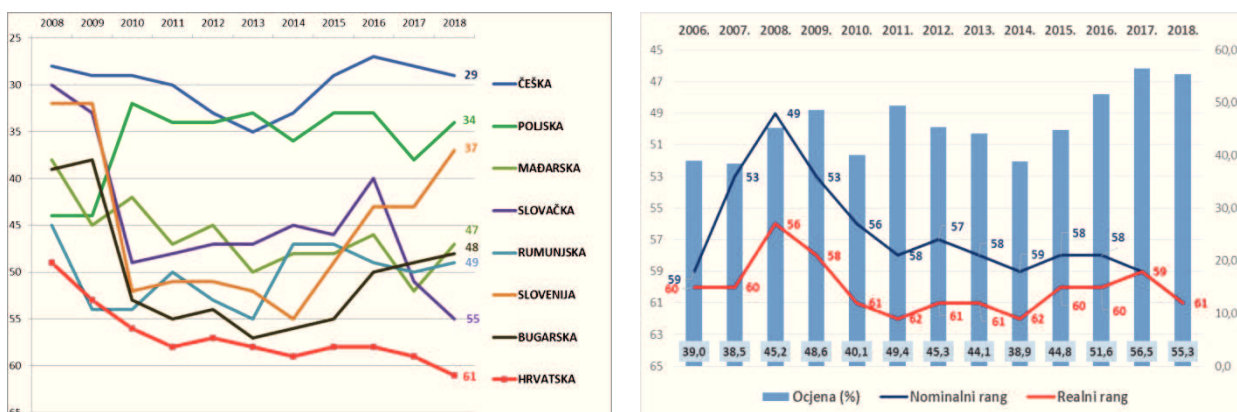
PREFACE

Foundry industry as a base branch represents an important factor contributing to the economic potential of each country. Current market development as well as technical and economic objective, the production of high-quality, low-cost and environmentally friendly casting, requires application of recent and advanced materials, as well as production technologies, followed and supported by understanding of production process.

Production imperative is pointed into the recent technologies and improved materials for everyday usage in our homes, workplaces, as well as materials with special requirements for specific applications such as those for the automotive or space industry. Industrial activities, which are defined as strategic activities in the Republic of Croatia are **Metal Casting** and **Production of Final Metal Products**, recognized as "*economic growth drivers*" because they are expected to realize higher rates of growth and employment.

According to the data of the Central Bureau of Statistics (DZS) and Financial Agency (FINA) and on the basis of analysis of the Sector for Financial Institutions, Business Information and Economic Analysis of the Croatian Chamber of Commerce and for the last analyzed 2017, the primary production of metals in the structure of Croatian industrial production is only 1.35% due to lack of economically viable primary raw materials and market fluctuations in their prices but also of the lack of modern production capacities. However, the valorization and export component of finished metal products stands out with a high share of almost 8.78%. Overall, this represents **10.13% of the industrial production of the Republic of Croatia**.

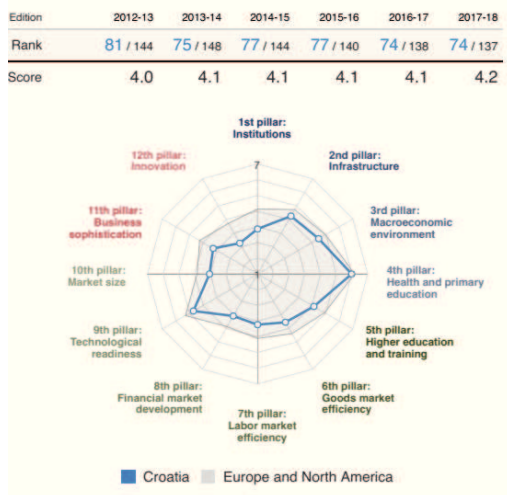
Croatia is also included yearly World Competitiveness Yearbook issued by Institute for Management Development (IMD) in Lausanne. "The World Year of Competitiveness" has been published every year since 1989 for the purpose of analysing and ranking the nation's ability to create and maintain an environment that maintains the company's competitiveness. In 2018 report, Croatia has ranked at 61th place from 63 world economy. The IMD methodology is based on an analysis of 4 factors of competitiveness, namely: economic results, public sector efficiency, business sector efficiency and infrastructure, and 5 indices for each area. Also, an overview of the status of the Republic of Croatia in the period from 2006 to 2018 is presented in the following graph.



IMD World Competitiveness Yearbook 2018

An overview of the status changes suggests that the economic crisis with its negative effects since 2008 for many comparable countries ended in 2014, while the Republic of Croatia in 2018 shows no recovery. Economic results are based on high revenues from tourism but also on exports. Despite a skilled workforce, a high level of education and a reliable infrastructure, a bad business environment, a slow administration and the burden of parafiscal charges still dictate a relatively

low labor price. Progress can be expected through stronger collaboration between the academic community and the economy, with emphasis on investment in innovation, knowledge transfer and technology optimization, with the prerequisite for the management structure to recognize the importance of such cooperation. In addition, the "Competitiveness Report" for 2017-2018 goes to this year, according to which the Republic of Croatia shows a continuous decline and this year it has 74th position out of 137 world economies, as shown in the following graph.



World Economic Forum, The Global Competitiveness Report 2017–2018

The problematic pillars of competitiveness are the continuity of business and innovation. The 5 most problematic factors for doing business in terms of efficiency valorization are identified: inefficient public administration, instability of legal regulation, tax regulations, and corruption and tax rates. These 5 factors can be regulated by public policy. Thereafter, there are four factors that the economy needs to recognize and impose as prerequisites for its competitiveness: inadequate capacity for innovation, availability of funding, limiting labor regulations, inadequately educated workforce. Identifying their own niche for competitiveness on the global market and following the stated public policy requirements for recognizing and incorporating them into development and funding strategies, as well as the education system in designing competent, creative and innovative workforce, can provide a synergy of positive moves towards increasing competitiveness.

Therefore, the importance of *coexistence of material science and sustainable technology in economic growth* reveals in collaboration between small and medium enterprises' (SMEs'), industry and higher education institutions (HEI). **International Foundrymen Conference** organized by University of Zagreb Faculty of Metallurgy, Sisak, Croatia in cooperation with University of Ljubljana Faculty of Natural Sciences and Engineering, Ljubljana, Slovenia, University North, Koprivnica, Croatia, Technical University of Košice Faculty of Materials, Metallurgy and Recycling, Košice, Slovakia, and ELKEM ASA, Norway found its significant position due to aforementioned reasons.

Coexistence of material science and sustainable technology in economic growth comprehends to recent technology and educated and skilled engineers. The Conference topics were designed as presentations of the current *"state of the art"* research in collaboration with industry, and production innovation with the aim to improve the competitiveness.

The scope of **18th International Foundrymen Conference (IFC)** covers scientific, technological and practical aspects concerning research, development and application of casting technology with the common perspective – increase of competitiveness. Special attention will be focused towards the

competitiveness ability of foundries, improvement of materials features and casting technologies, environmental protection as well as subjects connected to the application of castings.

During this Conference 35 paper will be presented. Book of Abstracts of the 18th International Foundrymen Conference includes summaries of the papers. The Proceedings book consists of papers *in extenso* published in electronic format (USB). Full length papers have undergone the international review procedure, done by eminent experts from corresponding fields, but have not undergone linguistic proof reading. Sequence of papers in Proceedings book has been done by category of papers in following order: plenary lectures, invited lectures, oral and poster presentation, and inside the category alphabetically by the first author's surname.

Within the Conference Student section is organized. This is an opportunity for industry to meet and recruit human resources as a main potential for business development. Coexistence of material science and sustainable technology in economic growth represent a knowledge transfer between small and medium enterprises' (SMEs'), industry and higher education institutions. Higher education at the Faculty of Metallurgy (HEI), conceived through the program and the learning outcomes, is based, inter alia, on promoting students' scientific and research work on applied topics, enabling ambitious and creative young people to become independent problem solvers, developing and supporting their curiosity, analytics and communication: **Graduates like the labour market need!**

This occasion represents an opportunity to discuss and increase the mutual collaboration between HEIs' and industry with the aim of information exchange related to advanced experience in foundry processes and technologies, gaining the new experience in presentation and / or teaching process within lifelong learning process.

The organizers of the Conference would like to thank all participants, reviewers, sponsors, auspices, media coverage and all those who have contributed to this Conference in any way.

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**THE EFFECT OF ANNEALING TIME ON MICROSTRUCTURE AND IMPACT
ENERGY OF STAINLESS STEEL AISI 316L**

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Poster presentation

Original scientific paper

Abstract

In this work the results of microstructural analysis and impact energy testing of austenitic stainless steel AISI 316L were carried out. Investigations were performed before and after annealing at 850 °C. Annealing time in this investigation varied from 30 to 90 minutes. After annealing, the samples were cooled in room temperature air. Microstructural analysis of initial rolled and different annealed states was performed by optical microscopy (OM) and scanning electron microscopy (SEM) equipped with device for energy dispersive spectroscopy (EDS). Impact tests were performed on Charpy V-notch specimens at room temperature. Initial rolled state of investigated steel showed the presence of typical elongated polygonal grains austenite and delta ferrite while annealed states showed the presence and evolution of sigma phase in microstructure. Impact energy value of initial rolled state was 260 J and by increasing annealing time it decreases.

Keywords: *stainless steel, microstructure, heat treatment, annealing, impact energy*

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INTRODUCTION

Stainless steels (SS) are based on the binary Fe-Cr systems, the properties of which are modified by the additional alloying elements like nickel, molybdenum and manganese. Molybdenum is added usually to type 316 steel to enhance the corrosion properties, primarily the pitting and crevice corrosion resistance [1]. In the world's total stainless steel production austenitic type steels take about 60% [2]. Austenitic stainless steels are often used in nuclear power plants, boilers, heat exchangers, chemical reactors etc. because their



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high resistance to corrosion and high temperature [3-5]. Also, stainless steel offers exceptional advantages for applications in construction [6]. Mostly stainless steels are used for construction as flat products and bars. Besides acceptable yield strength and tensile strength austenitic stainless steels are characterized by high impact energy and relatively low hardness. When considering the operational performance of austenitic stainless steel, the most important points to be taken into account are corrosion resistance, mechanical properties and the integrity of the welded joint in the case of welding these steels. Their high corrosion resistance resulted from formation of a continuous and protective surface oxide layer (passive film). This film is only a few nanometers thick and enriched in Cr (III) oxide/hydroxide species.

To reduce or prevent of microfissures in austenitic stainless steel, a minimum delta ferrite is required. Beneficial effect of delta ferrite is in dissolving more of harmful elements such as sulfur, phosphorus and boron in austenite [7]. But, this ferrite can be transformed in sigma phase. Austenitic steels may undergo microstructural changes when they are exposed to elevated temperature for a shorter or longer period of time. Microstructural variations caused by heat treatment are responsible for changes in the mechanical properties and corrosion resistance. Usually, three intermetallic phases which can be occurred in austenitic stainless steels are sigma phase, chi phase and Laves phase [8-10]. The precipitation mechanism in austenitic stainless steels has been the subject of many investigations motivated by the detrimental effects of the precipitated phases on impact energy and corrosion resistance of steels [11-13]. Padilha et al. [11] and Sourmail [13] reported the precipitation of carbides ($M_{23}C_6$, MC, M_6C , M_7C_3), primary nitrides (MN, M = Zr, Ti, Nb and V), and secondary nitrides (M_2N , M = Cr, Fe) in austenitic stainless steels during thermal treatment (annealing) or welding. Dománková et al. [10] mentioned the following sigma phase composition in AISI 316 which observed after ageing at 800 °C: 56–61%Fe, 21–26%Cr, 12–21%Mo and 1–5%Ni. As the sigma phase composition tends to vary it is difficult to define it by a formula but it is certain that it negatively affects on the steel properties.

The sigma phase has significant influence on properties of stainless steels and has been researched for some time [14]. Generally, the sigma phase forms via thermal ageing but also can be formed via radiation-induced segregation in FeCr alloys. Sigma phase is an intermetallic compound with a complex tetragonal crystalline structure and a typical sigma phase composition for the AISI 316L steel type is 44 % Fe - 29 % Cr - 8 % Mo. Sigma phase can be responsible for reduction in impact energy at room temperature.

Due to AISI 316L stainless steel can be particularly useful at very high temperatures (e.g. in nuclear reactor) it becomes important to study the material microstructure and impact energy at elevated temperatures. The aim of the present work is to show possibility the sigma phase appearance and whether relative short annealing time (up to 90 minutes) can have an influence on microstructure and impact energy of austenitic stainless steel AISI 316L.



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MATERIALS AND METHODS

The material used in this study was AISI 316L type stainless steel which was delivered in hot rolled state. The chemical composition of the investigated steel is listed in Table 1. Specimens for investigation were produced from steel plates of 15 mm thickness. Austenitic stainless steel AISI 316L was studied before and after heat treatment. Heat treatment consisted from annealing at 850 °C for 30, 60 and 90 minutes followed by cooling in the air. Microstructural analysis was carried out by optical microscopy Olympus GX 51 (OM) and scanning electron microscopy TESCAN VEGA 5136 MM (SEM) equipped with device for energy dispersive spectroscopy (EDS). Samples used for microstructural characterization were subsequently ground (papers grid 240-1200), polished ($0.3 \mu \text{Al}_2\text{O}_3$) and electrolytically etched in two different solutions. To expose austenite boundaries the etching solution 1 containing 60 ml HNO_3 and 40 ml water solution was used at 1V DC for 20 s. Sigma phase were identified with etching solution 2 composed from 56 g KOH in 100 ml water at 2V DC for 10 s. Impact tests were performed on Charpy V-notch specimens (7.5x10x55 mm) at room temperatures on device MLF System PSW 300.

Table 1. Chemical composition of investigated austenitic stainless steel AISI 316L, wt.%

C	Mn	Si	Cu	V	Mo	Al	Cr	Ni	W	Ti	Nb	Fe
0.018	1.50	0.33	0.39	0.078	1.91	0.006	17.34	10.56	0.121	0.003	0.025	balance

RESULTS AND DISCUSSION

From the results of this paper it was possible to establish a correlation between the microstructure, impact energy and various annealing time at 850 °C for investigated AISI 316L stainless steel. The material in delivered (hot rolled) and thermal treated (annealed) state was microstructural characterized firstly after electrolytically etching to expose austenite boundaries (etching solution 1). Figures 1-3 show a typical microstructure of the austenitic stainless steel of the present study. Optical micrographs (Figure 1a) and SEM micrographs (Figures 2a and 3) of microstructure of rolled state exhibited typical elongated grains of polygonal austenite and delta ferrite. Stringers of delta ferrite are elongated in the direction of rolling. In AISI 300 series stainless steels, during casting firstly is formed delta ferrite, and then this ferrite transforms to austenite by diffusion of chromium and nickel between the phases. Chromium diffuses to the ferrite and nickel to the advancing austenite. The austenite grains nucleate and grow into delta ferrite grains. The presence of residual delta ferrite retained at room temperature can be ascribed to the slow diffusion of chromium and nickel. The microstructure, according to Schaeffler diagram (Figure 4) [15], consisted of austenite and up to 10% of delta ferrite because the $\text{Cr}_{\text{eq}}/\text{Ni}_{\text{eq}}$ ratio was 1.69 (Equations 1 and 2).



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$$Cr_{eq} = \%Cr + 1.5\%Si + \%Mo + 0.5\%(Ta+Nb) + 2\%Ti + \%W + \%V + \%Al \quad (1)$$

$$Ni_{eq} = \%Ni + 30\%C + 0.5\%Mn + 0.5\%Co \quad (2)$$

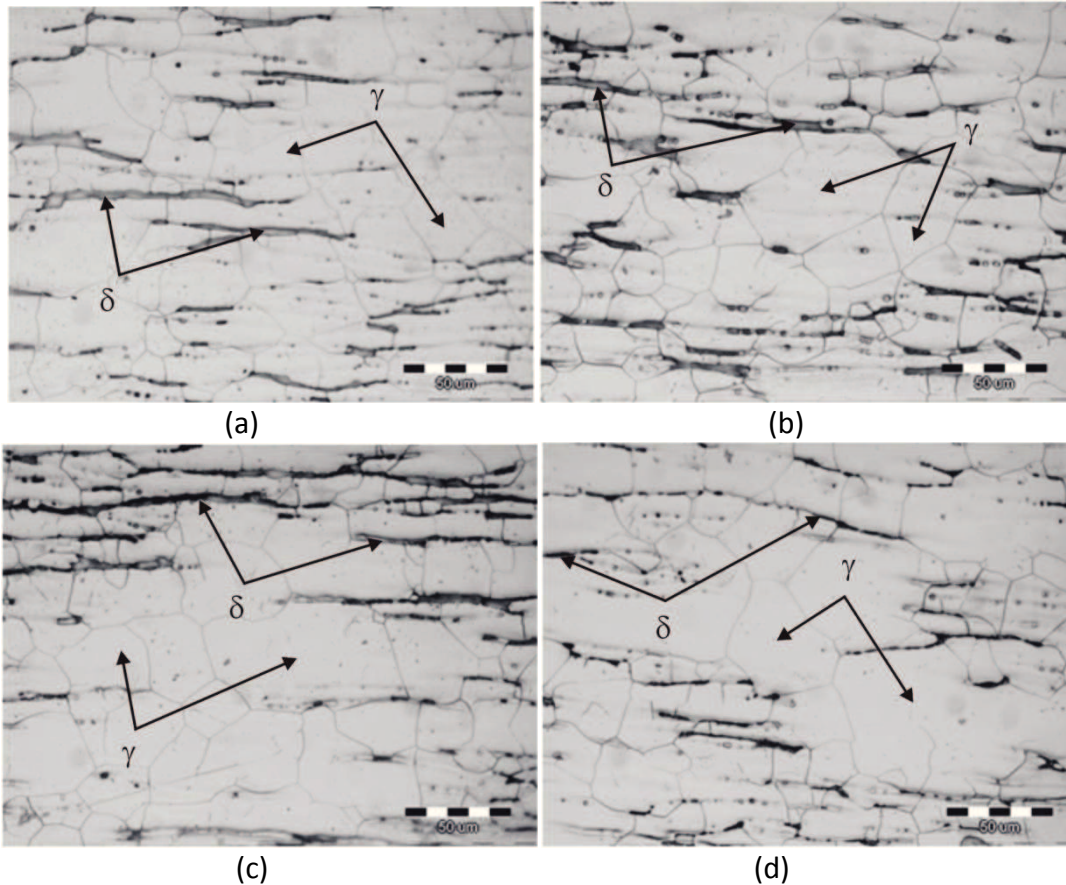


Figure 1. Optical micrographs of AISI 316L stainless steel in rolled (delivered) state (a), annealed state 850 °C/30 min (b), annealed state 850 °C/60 min (c), annealed state 850 °C/90 min (d); etching solution 1



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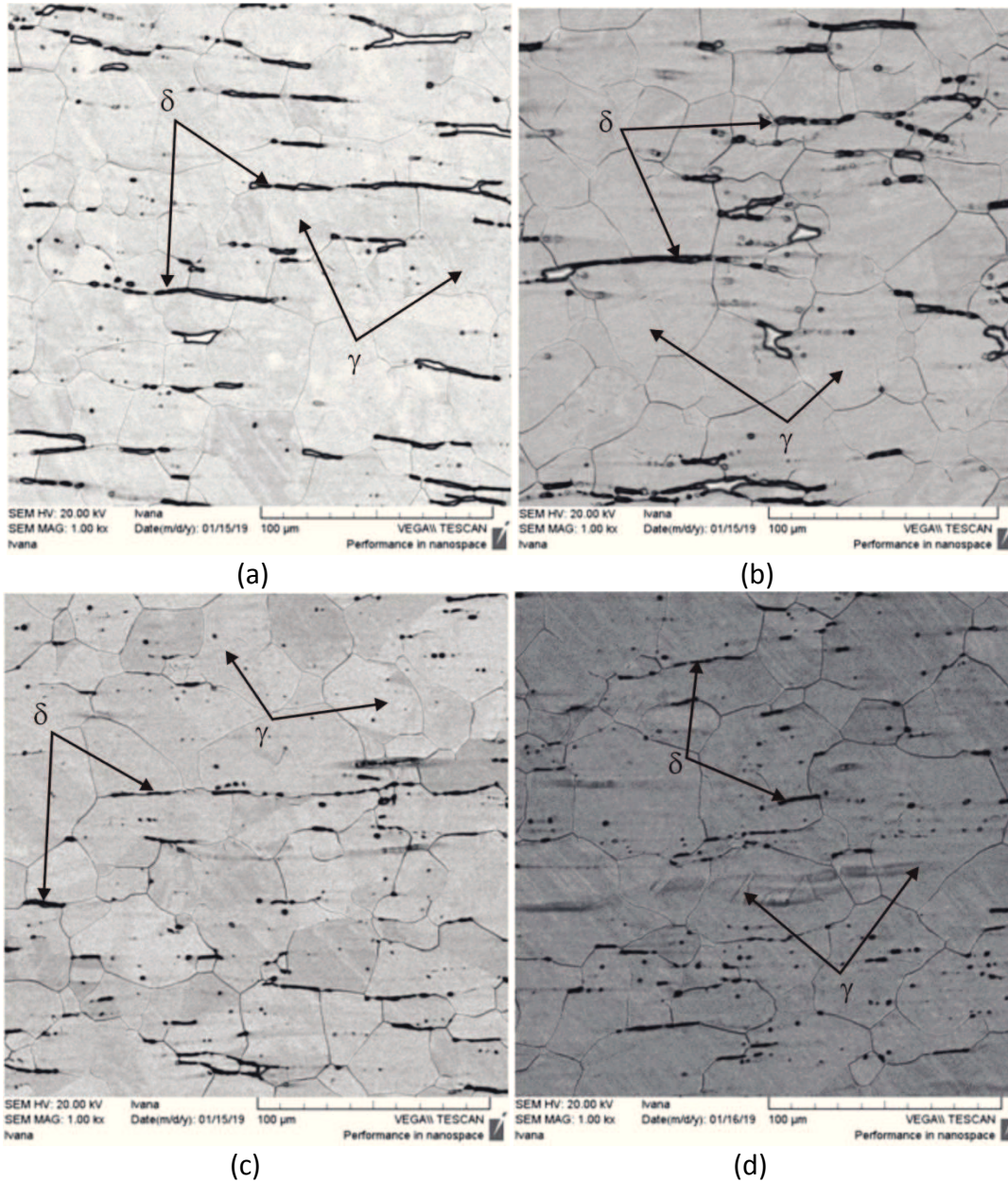


Figure 2. SEM micrographs of AISI 316L stainless steel in rolled (delivered) state (a), annealed state 850 °C/30 min (b), annealed state 850 °C/60 min (c), annealed state 850 °C/90 min (d); etching solution 1



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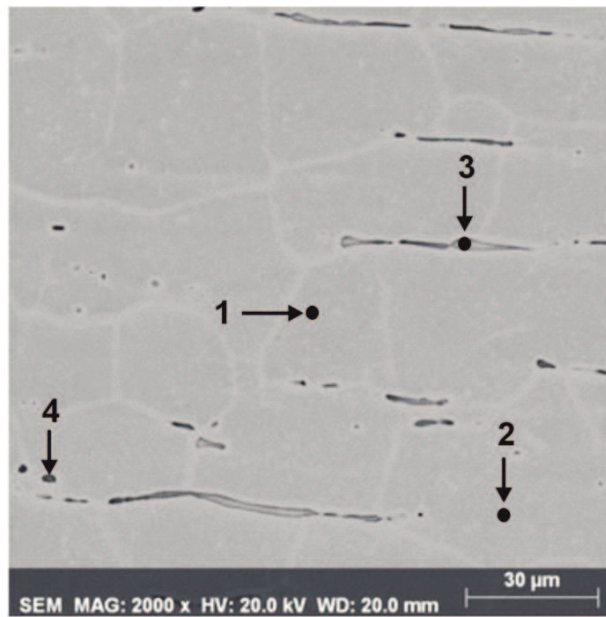


Figure 3. SEM micrograph of AISI 316L stainless steel in rolled (delivered) state with marked positions for EDS analysis; etching solution 1

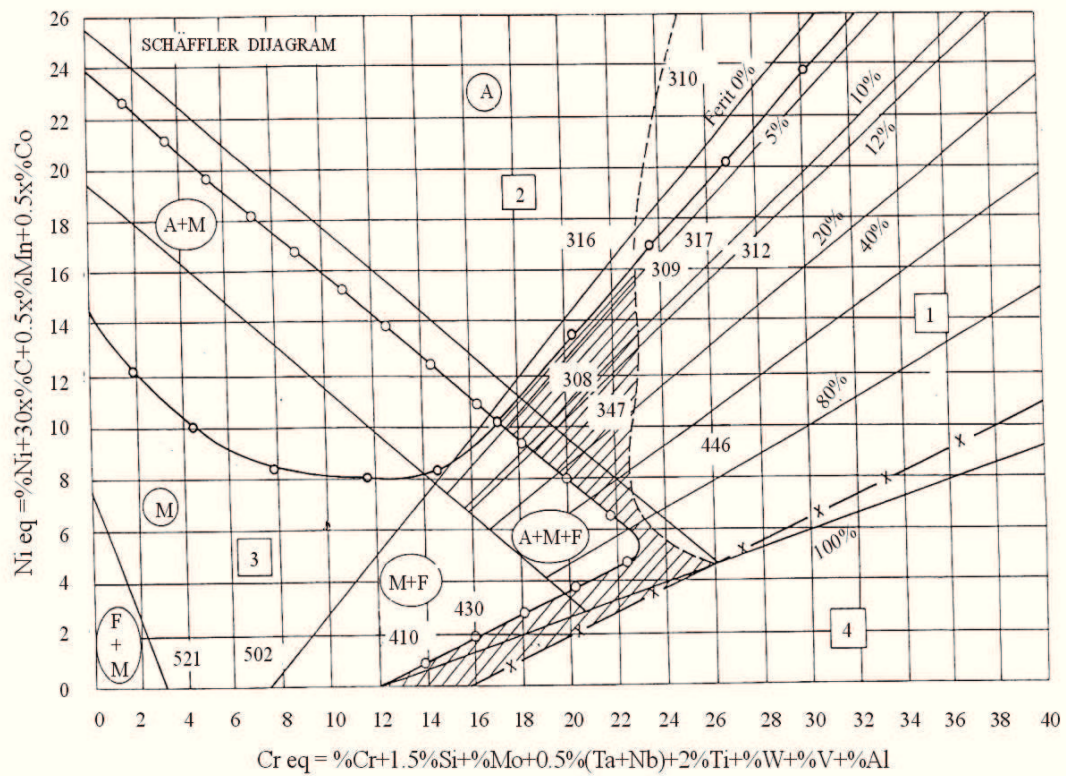


Figure 4. Schaeffler diagram [15]



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It is generally held that up to 10% of delta ferrite in microstructure of austenitic stainless steel is an effective means of offsetting a grain boundary weakness that develops in austenite at high temperatures and leads to fissuring. From detailed analysis of optical and SEM micrographs (Figures 1b, 1c, 1d and 2b, 2c, 2d) can be obtained similar microstructures in OM micrographs. In contrast, SEM micrographs show that increase in the annealing time results in a decrease content of elongated ferrite stringers. Annealing time was too short for visible changes at OM micrographs, probably. Figure 3 show SEM micrograph of AISI 316L stainless steel in as-rolled state with marked positions for EDS analysis. The results of the EDS analysis (Table 2) show similar content of chromium (16.44-16.87 wt.%), nickel (11.19-11.82 wt.%), manganese (1.77-2.00 wt.%), molybdenum (1.77-2.24 wt.%), and silicon (0.23-0.33 wt.%) for all positions.

Table 2. Results of EDS analysis of different positions in as-rolled state AISI 316L stainless steel; positions marked at the Figure 3

Positions	Chemical composition, wt.%					
	Fe	Cr	Ni	Mn	Mo	Si
1	67.19	16.50	11.74	2.00	2.24	0.33
2	67.81	16.44	11.82	1.88	1.77	0.27
3	67.53	16.66	11.75	1.97	1.84	0.26
4	67.87	16.87	11.19	1.77	2.07	0.23

Also, the investigated steel in rolled and annealed state was microstructural characterized after electrolytically etching to expose sigma phase (etching solution 2). Figures 5-7 show a microstructure of the austenitic stainless steel AISI 316L after etching in solution 2. The presence of sigma phase was observed. Optical and SEM micrographs show that the content of the sigma phase is increasing by increasing annealing time. In rolled (delivered) state (Figures 5a and 6a) the sigma phase is not present since it only occurs by exposing the steel to high temperatures. Sigma phase is intermetallic phase which usually forms in the Fe-Cr systems at high temperatures (550-900 °C). The mechanism of sigma phase nucleation can be described by transformation of delta ferrite. Transformation of delta ferrite into sigma phase was a function of the chemical composition and the kinetics of its precipitation which was governed by the rate of diffusion sigma-forming elements, especially chromium and molybdenum. Figure 7 show SEM micrograph of AISI 316L stainless steel in as-annealed 850 °C/90 min state with marked positions for EDS analysis. The results of the EDS analysis show similar content of manganese (1.96-1.98 wt.%), molybdenum (1.88-2.74 wt.%), and silicon (0.24-0.27 wt.%) for all analyzed positions. By contrast, the chromium content is higher at position 2 (Figure 7, Table 3). Based on this it can be assumed that position 2 presents probably a sigma phase evolution with chromium content 21.42 wt.% and nickel content 6.79 wt.%.



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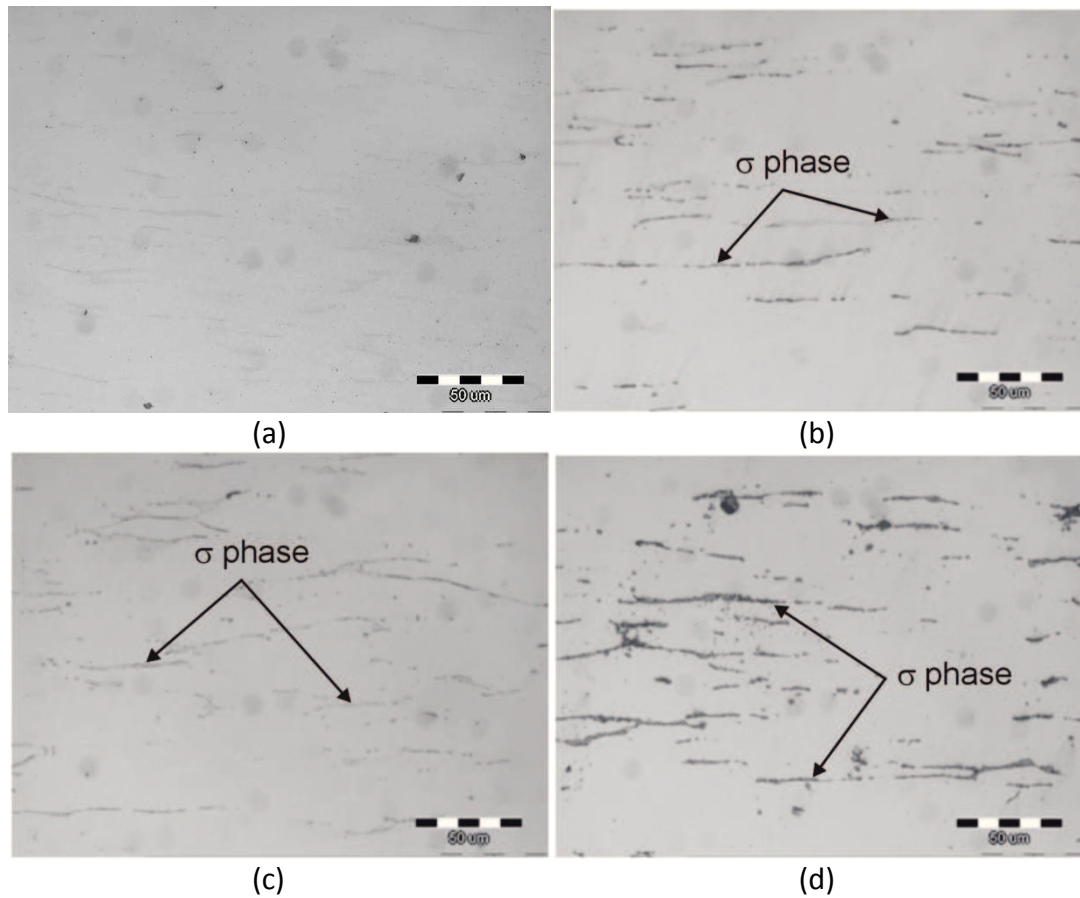


Figure 5. Optical micrographs of AISI 316L stainless steel in rolled (delivered) state (a), annealed state 850 °C/30 min (b), annealed state 850 °C/60 min (c), annealed state 850 °C/90 min (d); etching solution 2



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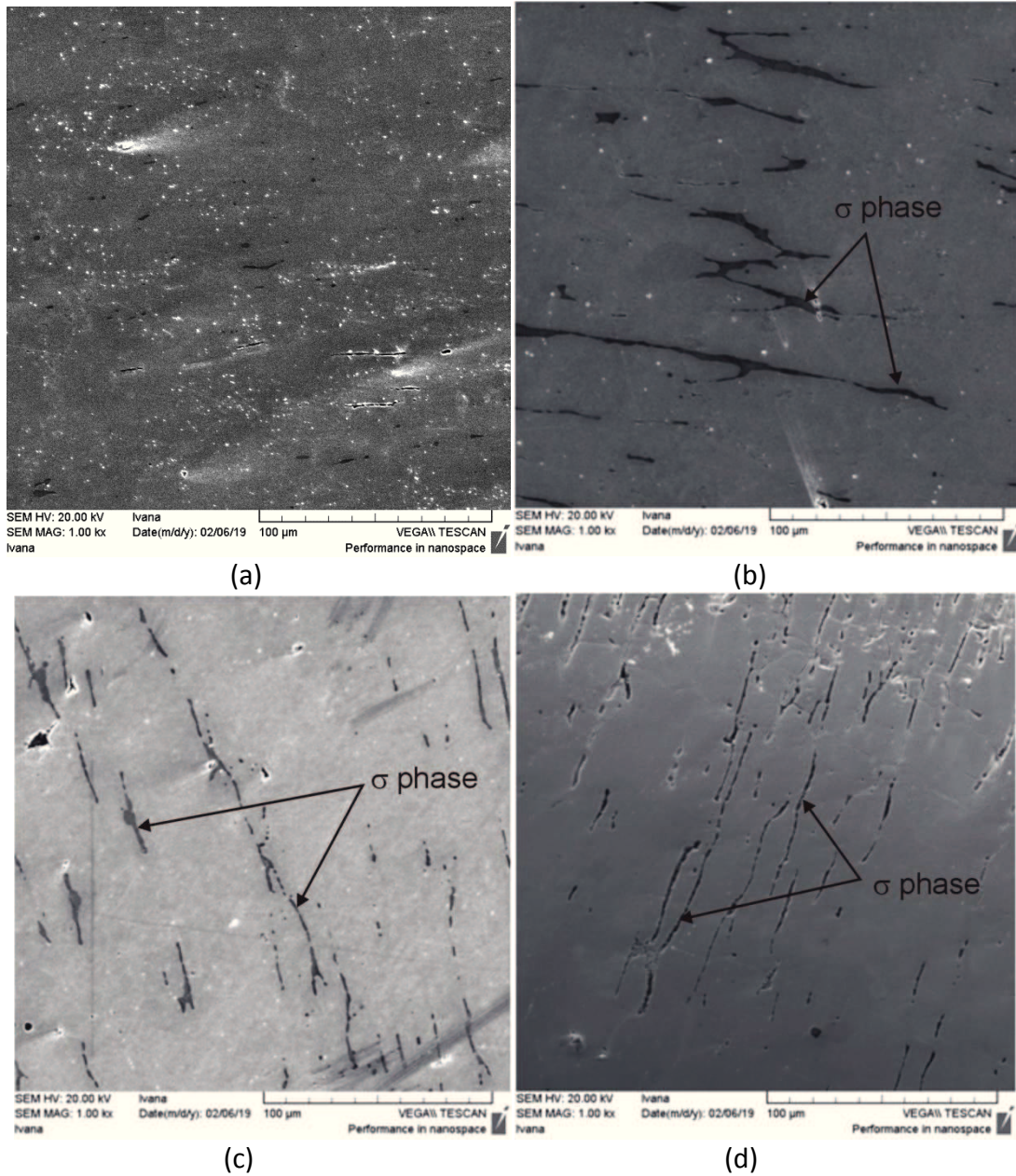


Figure 6. SEM micrographs of AISI 316L stainless steel in rolled (delivered) state (a), annealed state 850 °C/30 min (b), annealed state 850 °C/60 min (c), annealed state 850 °C/90 min (d); etching solution 2



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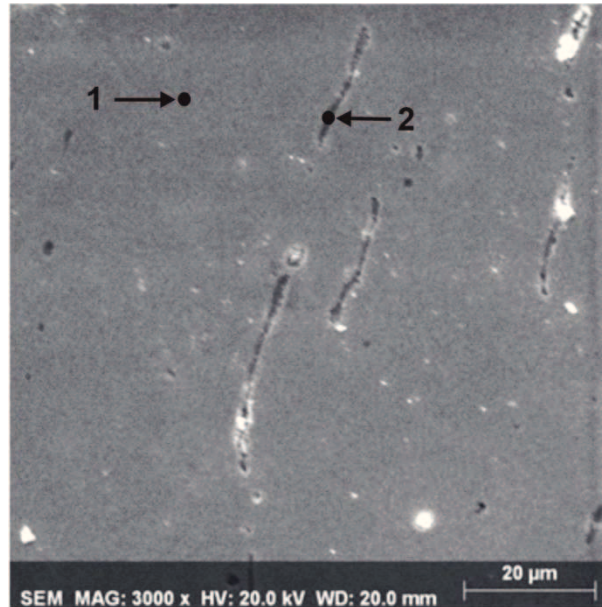


Figure 7. SEM micrograph of AISI 316L stainless steel in annealed state 850 °C/90 min with marked positions for EDS analysis; etching solution 2

Table 3. Results of EDS analysis of different positions in as-annealed state 850 °C/90 min; positions marked at the Figure 6

Positions	Chemical composition, wt.%					
	Fe	Cr	Ni	Mn	Mo	Si
1	68.86	15.81	11.23	1.96	1.88	0.27
2	66.82	21.42	6.79	1.98	2.74	0.24

Figure 8 show the average values of impact energy testing of the investigated AISI 316L stainless steel before and after heat treatment (annealing). The values of impact energy are given as means of mostly three determinations. With a more detailed analysis of the impact energy values it can be seen that increasing annealing time (30-90 min) caused decreasing in impact energy. Before heat treatment impact energy value of AISI 316L stainless steel was 260 J. Heat treated state 850 °C/30 min/air have impact energy 224.5 J and it decreased to 166 J in heat treated state 850 °C/90 min/air. This decrease in impact energy can be related to microstructural changes i.e. occurrence and evolution of sigma phase by increasing annealing time.



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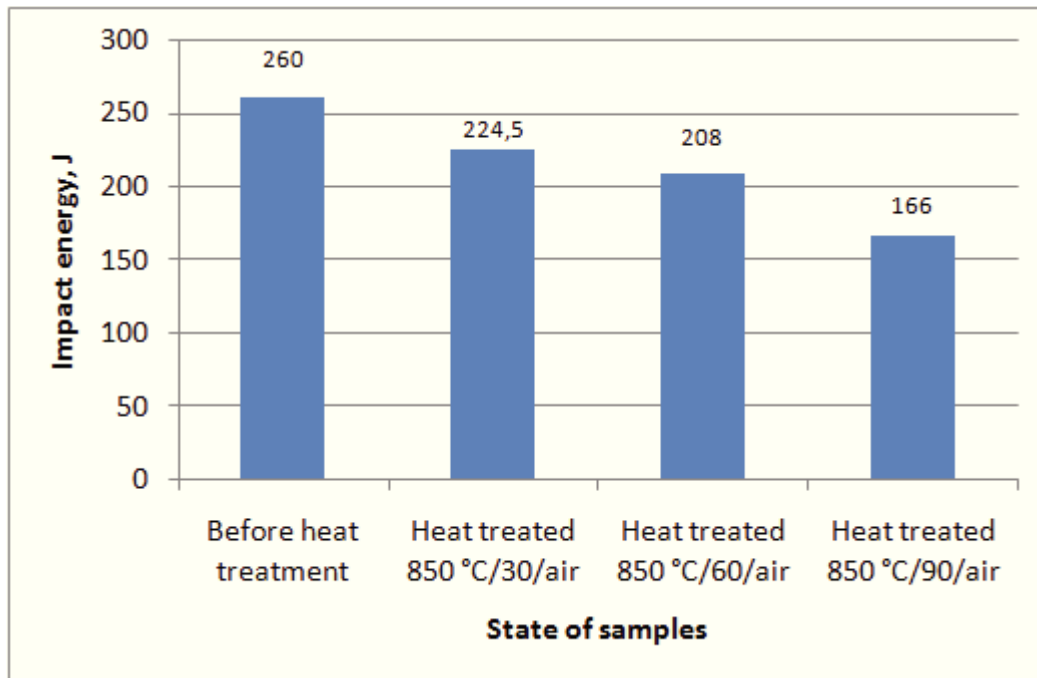


Figure 8. Impact energy vs. annealing time for stainless steel AISI 316L

CONCLUSIONS

Results of investigation the effect of annealing time on microstructure and impact energy of austenitic stainless steel AISI 316L suggest the following:

- Optical micrographs and SEM micrographs of microstructure of initial rolled state confirmed the presence of typical elongated polygonal grains of austenite and delta ferrite stringers.
- Increase in the annealing time from 30 to 90 minutes resulted in a decrease of elongated ferrite stringers content.
- EDS analysis showed similar content of chromium (16.44-16.87 wt.%), nickel (11.19-11.82 wt.%), manganese (1.77-2.00 wt.%), molybdenum (1.77-2.24 wt.%), and silicon (0.23-0.33 wt.%) for different positions in delivered rolled state.
- Optical and SEM micrographs showed the presence of sigma phase in annealed states. According to micrographs the content of the sigma phase is increasing by increasing annealing time.
- EDS analysis of annealed states showed position with higher chromium content (21.42 wt.%) and this position presents a sigma phase evolution, probably.
- Increasing annealing time from 30 to 90 minutes caused a decrease in impact energy. Impact energy value of stainless steel AISI 316L in initial rolled state was 260 J while after 90 minutes of annealing time impact energy decrease to 166 J. This decreasing can be result of occurrence and evolution of sigma phase during annealing.



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