

Influence of structure sensitising of the AISI 316Ti austenitic stainless steel on the ultra-high cycle fatigue properties

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Abstract. Austenitic stainless steels are the wide-spread materials, used mainly in the power industry. In that kind of engineering application, structural parts of rotating elements reach during their lifetime very high numbers of loading cycles, exceeding 10^7 numbers of cycles. With regard to this fact, the data of ultra-high cycle fatigue properties are needed to be used in the qualified design. Increasing demands on the efficiency cause the increase of the operating temperature, and exposition of these materials to the elevated temperatures can cause some important structural changes, which result in the sensitising of the structure. In this study authors present their own experimental results about fatigue properties of AISI 316Ti austenitic stainless steel after sensitising, in the ultra-high cycle region ($N_f = 10^6 \sim N_f = 3 \times 10^9$ cycles). Fatigue tests were carried out using ultrasonic fatigue testing device with frequency $f = 20$ kHz at the coefficient of cycle asymmetry $R = -1$, and temperature $T = 20 \pm 5^\circ\text{C}$. In the ultra-high cycle region was observed the continuous decrease of the fatigue properties of the AISI 316Ti, and there was recorded the negative effect of the sensitising on the ultra-high cycle fatigue properties of the AISI 316Ti.

Keywords: AISI 316Ti, sensitisation, ultra-high cycle fatigue

1 Introduction

Austenitic stainless steels are the important category of the technical materials, which have found the wide range of the applications. These steels are highly resistant to many of corrosion environments, and they are successfully used in the chemical and power industry. Excellent corrosion properties of these steels are given by the chemical composition, where the high chromium content enables to create the stable passive layer which prevents degradation by the corrosion processes. In special cases where these steels are exposed to increased temperatures for some time (after welding or in the application in the power industry), significant changes of the microstructure can occur, which affect and decrease the ability to create the protective passive layer. This phenomenon is called sensitisation [1, 2]. Sensitisation of austenitic stainless steels is caused by the extensive precipitation of the

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chromium-rich phases on the grain boundaries, mainly carbides Cr_{23}C_6 and the σ phase, which consume chromium dissolved in the matrix and locally decrease its concentration under the critical value, 11.6%, which is considered as a lowest concentration to provide creation of the stable passive layer [3-5]. Precipitation of these phases occurs preferentially in the grain boundaries regions, which can result in the formation of the continuous network of the carbides on the grain boundaries, which leads to corrosion resistance decrease and the formation of the intercrystalline corrosion and the failure even at very low loadings [6]. In austenitic stainless steels containing molybdenum (as a 316 grade) these carbides also contain Mo, so these areas are also depleted in Mo [6]. Susceptibility of the austenitic stainless steels to sensitisation strongly depends on the chemical composition. Decreasing of the C content and increasing amount of Cr, Mo, and N decrease the susceptibility to sensitisation of austenitic stainless steels [6].

To increase the resistance to sensitisation, special grades of austenitic stainless steels were developed. Improving resistance to the sensitisation could be obtained by the decreasing the carbon content to the lowest possible level. Grades AISI 304L, 316L and 321 has the maximum carbon content 0.03%. The second way how to improve resistance to sensitisation is the alloying steels by the small addition of the strong carbide former, as a Ti or Nb (Grades 316Ti and 347). Recently was introduced another option of reducing susceptibility to sensitisation, using grain boundary engineering [2, 7]. Stabilized steels exhibit a higher level of resistance to sensitisation than steels with lowering carbon content, while niobium is more effective stabilizer than titanium. Despite this fact, even in these more resistant grades, sensitisation can occur, especially in low-temperature exposures, what was documented in the work [1], where austenitic stainless steel AISI 321 (austenitic stainless steel stabilized with the Ti) sensitised after exposures at temperatures around 600°C.

Sensitisation caused degradation of the austenitic stainless steels properties, increase the susceptibility to intercrystalline corrosion, stress corrosion cracking and also degradation of mechanical properties. Sensitisation affects the fatigue properties indirectly, as the premature fatigue crack initiation occurs on the corrosion pits, which can form when the sensitised steel has decreased the corrosion properties [8]. The direct effect is the increasing the fatigue crack propagation by the intercrystalline manner, which is an important factor in the low cycle fatigue. Other aspects of the direct effect of the sensitisation on the fatigue properties, especially in the ultra-high cycle area, where the fatigue crack initiation and the propagation of short cracks plays a dominant role in the fatigue process, are still not well examined. Ultra-high cycle fatigue is the region beyond the conventional fatigue limit ($N_f = 10^7$), and during cyclic loading in this region, different mechanisms of the failure may take places, which makes necessity to experimental evaluation of the effect of the microstructural changes on the fatigue properties in this area.

Authors in this study present their own experimental results of the influence of sensitisation on the ultra-high cycle fatigue properties of the AISI 316Ti austenitic stainless steel.

2 Experimental procedures

2.1 Material

As the experimental material in this study was used steel AISI 316Ti in the form of bars with the diameter of 15 mm, which represents the austenitic stainless steel with improved susceptibility to the sensitisation by the addition of Ti stabilizer. The chemical composition of the tested material is shown in Table 1, and it was obtained by the emission spectrometry device ICP (JPY 385). Evaluation of the basic mechanical properties was carried out by the

standard tensile tests. These were conducted on the ZWICK Z050 testing machine at an ambient temperature $20\pm 5^\circ\text{C}$ and the initial strain rate was $\epsilon_m = 10^{-3} \text{ s}^{-1}$. Specimens used for the tensile test fulfilled the requirements of the EN 10002-1 standard for tensile tests, five specimens were used and the average values of the mechanical properties were than calculate. Mechanical properties are shown in Table 2. The microstructure of the tested material was examined using optical microscopy and the scanning electron microscopy, and the samples were prepared by the standard procedure for the preparation of metallographic samples and etched by the solution of 30 ml HCl, 10 ml HNO_3 and 30 ml glycerol.

Table 1. Chemical composition of experimental material

Cr	Ni	Mo	Mn	Ti	C	Si	P	S	Fe
17.55	12.96	2.54	1.63	0.37	0.058	0.81	0.033	0.037	Bal.

Table 2. Mechanical properties of experimental material

YS [MPa]	UTS [MPa]	Elongation[%]	Reduction in area [%]	HV10
251	773	54	48	213

Microstructure observation (Fig. 1) reveals austenitic polyedric structure, with a high density of the annealing and deformation twins. In the structure were also present different kinds of secondary particles, their nature was examined using EDX analysis, and they were identified as a Cr, Ti, and Mo-rich particles, with the average size up to the 5 μm . The structure also contains considerably large Ti-rich particles, which were identified as carbides and nitrides of titanium, with the average size 10-20 μm . A small amount of delta ferrite, in a form of elongated particles, was present and the structure also contains a small amount of Si-rich inclusion. No σ phase was observed in the structure of the as-delivered state.

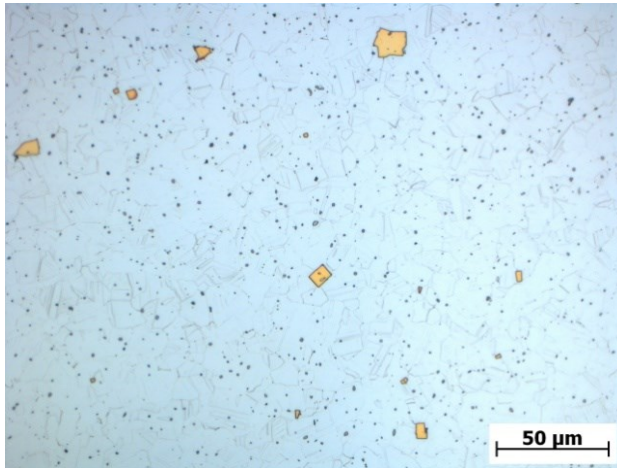


Fig. 1. Microstructure of AISI 316Ti in as-delivered state

Specimens for the fatigue tests were machined from the bars, according the drawing on the Fig. 2. Dimension of the specimens were calculated to fulfill the resonance conditions of the ultrasonic fatigue testing machines. After machining, the surface of the specimens was polished, to prevent any affecting of testing results by the specimen's surface state.

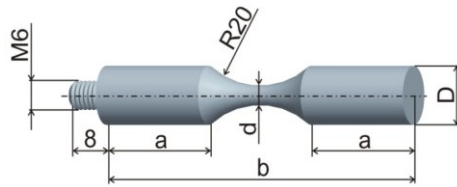


Fig. 2. Drawing of the fatigue test specimen

2.2 Sensitisation treatment and evaluation

Part of samples for the fatigue test was heat treated to provide sensitising of the structure. Sensitising heat treatment has been carried out in resistance furnace in the air atmosphere. Heat treatment consists of the heating to the 600 °C, holding at this temperature for 100 hours followed by the slow cooling on the air. Conditions of sensitisation were purposely selected, and this treatment represents the standard operating temperature of these steels in the power industry. Based on the published studies, precipitation of carbides on the grain boundaries can occur at this temperature, and also diffusion rates of chromium are considerably low, so the chromium content in the depleted zones cannot be restored by the diffusion from the grains interiors. Another important fact is that at 600 °C precipitation of Cr-rich carbides is more favourable than Ti-rich carbides [1].

For the determination of the degree of sensitisation, the ASTM standard 262 practice A has been used. This method is based on the electrolytic etching of the specimens, prepared by the standard method for the preparation of the metallographic samples, in the 10% water solution of oxalic acid at the constant potential 10V to reveal the chromium depletion zones. Recommended practice for evaluation of sensitisation degree based on the ASTM standard 262 practice A characterize three degrees of sensitisation. Step – represent no sensitised structure, where no attack on the grain boundaries is observed. Dual – structure contains a small amount of pits on the grain boundaries, without pits surrounding whole grains. Ditch – strong attack on the grain boundaries, pits surround at least one whole grain.

2.3 Fatigue tests

Determination of fatigue properties in the ultra-high cycle area was carried out on the ultrasonic fatigue testing device KAUP, which schematic diagram is shown on the Fig.3. KAUP ultrasonic fatigue testing device loads specimen with the symmetrical tension-compression loading, at the resonant frequency 20 kHz. The detailed description of this device and the principles of the ultrasonic fatigue testing are more detailed described in the references [9-11]. During whole tests, constant temperature 20±5°C was maintained. Due to a high level of internal friction, typical for austenitic stainless steels, additional cooling of the specimens was needed. Some authors in their study of ultrasonic fatigue of austenitic stainless steels [12] used a stream of cold air to cooling specimens, but this was not to be effective enough in our case, thus water cooling was used. Specimens were submerged in the water with additives, to effectively lead away heat generated during high-frequency loading. Tests were carried out in the range $10^6 < N_f < 3 \times 10^9$ number of cycles. Two series of specimens were used, the initial state and after sensitisation at 600°C/100h. Fatigue tests results were shown in the S-N diagrams, and the regression of the curves was done by the Basquin equation (1), in the form:

$$\sigma_a = \sigma'_f (N_f)^b \quad (1)$$

where b is exponent of fatigue live curve and σ'_f is the coefficient of fatigue strength obtained by extrapolation of stress amplitude σ_a on the first loading cycle.

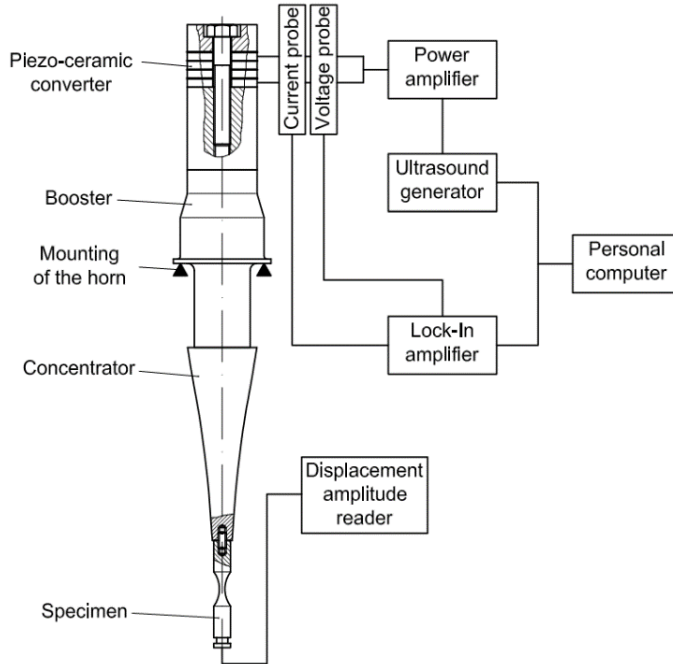


Fig. 3. Ultrasonic fatigue testing device KAUP

3 Results

3.1 Sensitisation evaluation

Sensitisation degree was evaluated according to ASTM 262-A standard. This test reveals, that applied heat treatment (600°C/100h) result in sensitisation of austenitic stainless steel AISI 316Ti. The degree of sensitisation was evaluated as a ditch, because the careful observation taken on the multiple places shows a high degree of intergranular attack and individual grains were wholly surrounded by the pits (Fig. 4).

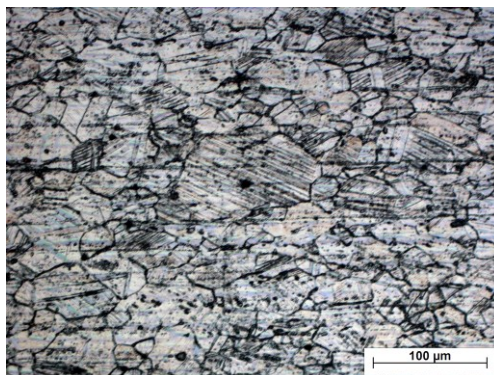


Fig. 4. Microstructure of sensitised AISI 316Ti electrolytically etched by oxalic acid

3.2 Fatigue tests

S-N curves were constructed from the fatigue tests results. Curves were approximated by Basquin equation, using the least square method [13]. Regression curves coefficients are shown in Table 3. In both testes states was recorded decrease of fatigue strength with increasing number of loading cycles, even after conventional fatigue limit (10^7 loading cycles). Figure 5 shows a comparison of the S-N curves for initial state and a state after sensitising treatment. In both curves was not recorded the presence of any plateau, which suggests no changing of preferential mechanisms of fatigue crack initiation.

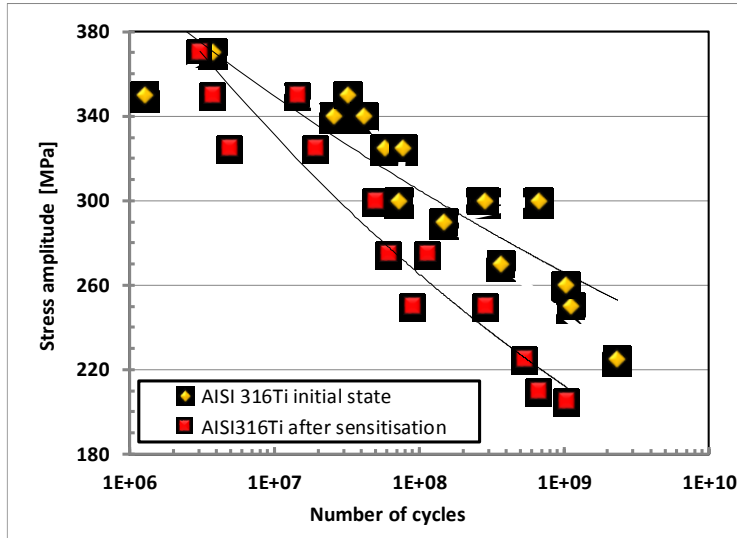


Fig. 5. S-N curves of AISI 316Ti in initial state and after sensitisation

Using regression curves coefficients; fatigue strength for different numbers of loading cycles was calculated for both tested states. Results are shown in Table 3, as it can be seen, the decrease of fatigue strength for the sensitised state is more significant at higher numbers of loading cycles. For the 10^9 number of cycles exhibits sensitised state 20% decrease of fatigue strength in comparison with the initial state.

Table 3. Regression curve coefficients and comparison of fatigue strength for different numbers of loading cycles

	σ'_f [MPa]	b	Fatigue strength for $N = 10^7$ [MPa]	Fatigue strength for $N = 10^8$ [MPa]	Fatigue strength for $N = 10^9$ [MPa]
Initial state	910.58	-0.059	351.8	307.1	268.1
Sensitised	1564.7	-0.096	333	266.9	214

3.3 Fractography analysis

Fractography analysis was carried on the broken specimens, with the aim to describe the fatigue process and to find any differences which can be related to the sensitisation treatment. Every broken specimen was carefully examined, to rule out any affecting of initiation stage by possible cavitation. Despite observations made by other authors [14], no cavitation was recorded on the specimens, even they were submerged in the water to

prevent self-heating of the testing specimens during fatigue tests. Two different types of initiation of fatigue crack were recorded. In both, the crack forms in the surface and subsequently propagate along slip plane, usually at 45° degrees to the loading direction and when the stress intensity increase, the crack will start to propagate perpendicular to the loading axis. At high loading amplitudes, the first stage of the fatigue crack growth is quite small, in just a few μm , because the higher stress level allows to transition to the other slip planes, which are perpendicular to the loading axis. This type of initiation was observed for both states, in similar values of the loading stress (Fig. 6). The second type of crack initiation occurs at the lower loading stress levels. In this type of crack initiation, fatigue crack initiates also on the surface, but due to lower stress intensity, the length of crack propagation along the initial slip plane is much longer, and it may extend across several grain diameters before the increase of the stress intensity allows to promote slip in other systems then primary slip system [15], see Fig. 6. The occurrence of these two types of fatigue initiations was recorded for the both tested states so the change of initiation mechanism cannot be related to the decrease of fatigue strength in sensitised specimens. Occurrence of the second type of fatigue crack initiation was not observed in every specimen broken at lower loading amplitude, and this is caused by the nature of this mechanism, where very important role plays the crystallographic orientation of the grain where the initiation starts.

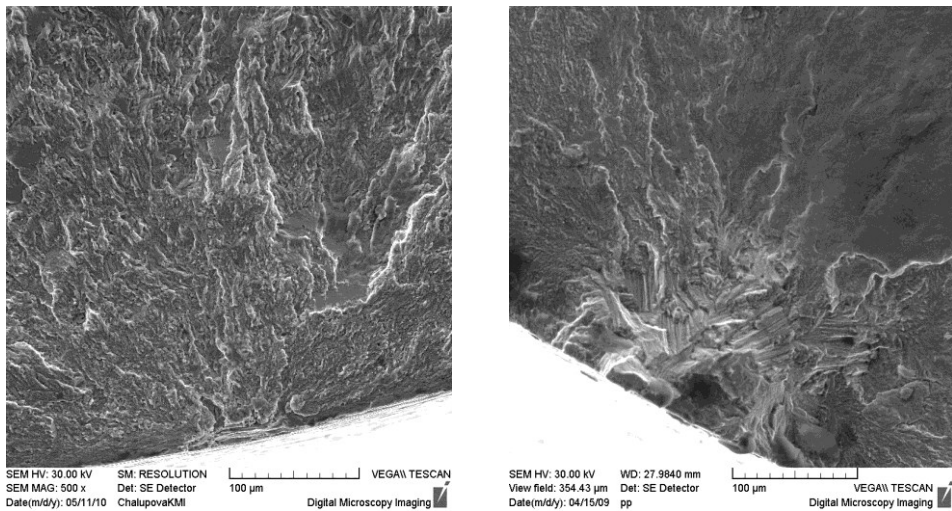


Fig. 6. Initiation of fatigue cracks. Left figure shows sensitised specimen broken after $N_f = 3.8 \times 10^7$ cycles at $\sigma_a = 350$ MPa. Right picture shows crystallographic initiation of fatigue crack, sensitised specimen, $N_f = 2.9 \times 10^8$, $\sigma_a = 250$ MPa

Observation of the crack surfaces shows that after initiation, fatigue cracks propagate mostly by transcrystalline manner (Fig. 7), and on the cracks surfaces were observed ductile striations. In all specimens, a few regions of intercrystalline fatigue crack propagation were recorded (Fig. 7). In the initial state specimens, intercrystalline crack propagation regions did not exceed 1% of the total crack surface area. Sample after sensitising treatment exhibits the higher content of the intercrystalline crack propagation area which however did not exceed 5% of the total crack surface area.

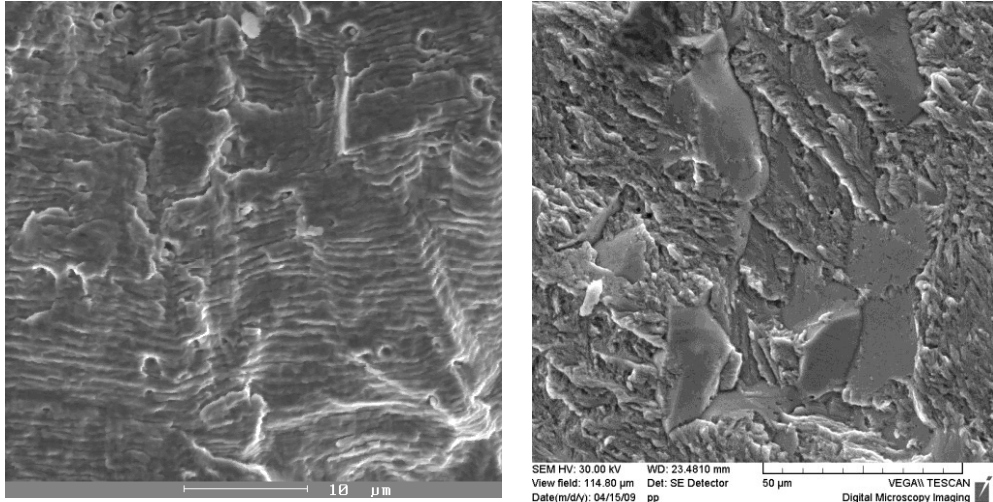


Fig. 7. Fatigue crack propagation. Left figure: transcrystalline fatigue crack propagation, clearly visible striations, sensitised specimen, $N_f = 3.8 \times 10^7$, $\sigma_a = 350$ MPa. Right figure: intercrystalline fatigue crack propagation, the same specimen

4 Discussions

4.1 Sensitisation

At first, the sensitisation treatment will be discussed. Most previous studies were dedicated to sensitisation at high-temperature exposures. In real applications, the sensitisation of austenitic stainless steel can have two causes. The main and the most studied case is the sensitisation of welded joint, where the heat affected zone is subjected to temperature regimes ideal for the precipitation of chromium-rich carbides at the grain boundaries. Heat input causes that all carbon will solute in the matrix and during subsequent cooling there is enough time to precipitation of carbides along the grain boundaries but not sufficient time to chromium diffusion from grain interior to the depleted zone in the grain boundary areas. This can be suppressed by special heat treatment after welding, like cooling with the controlled cooling rate or by the soaking time at elevated temperatures in order to realize desensitisation. For improving resistance to sensitisation after welding, low carbon and stabilized grades of AISI 316 steel were introduced. The second case of sensitisation occurs when these steels are subjected to intermediate (550-650°C) temperatures for long exposures. In the real applications, this could be easily achieved, while these steels are standard using in this temperatures range. According to literature, at these temperatures, chromium-rich carbides precipitate in favor of titanium carbides [1]. During sufficient long exposures at high temperature, desensitisation can occur, which means, that the chromium depleted zones disappeared because there was enough time for diffusion chromium atoms to the depleted zones at grain boundaries from the grain interiors. For the austenitic stainless steels with improved resistance to sensitisation (ex 316L) was fully desensitisation observed after 2h of exposure to 950°C and after 8h at 900°C. As it's seen, the rate of desensitisation process is fully driven by the temperature and by the diffusion rates of chromium [16]. However, at a lower temperature, as in the study used 600°C, based on the observation made in the study [16], no desensitisation occurs in 316Ti grade after 100h at 600°C. For these reasons, temperature regime 600°C/100h was chosen for the sensitisation treatment in these study, and

predictably, this treatment results in fully sensitisation of the structure of austenitic stainless steel AISI 316Ti.

4.2 Fatigue properties

Fatigue test result shows two important conclusions. At first, the fatigue strength of AISI 316Ti decrease with increasing number of loading cycles, what is in strong contrast with the work [12] where the constant fatigue limit in the range 10^7 - 10^9 of loading cycles was recorded and no decrease of fatigue strength was observed. However, results of other authors [17] made on the similar material (316L) show similar result as in our study. The second important conclusion is that sensitisation negatively affects the fatigue properties in ultra-high cycle area, and this influence is increasing with increasing numbers of loading cycles. Data displayed in Table 3 shows that this effect is stronger with increasing numbers of loading cycles. For 10^7 of loading cycles this decrease is about 5%, for 10^8 of cycles it is about 13% and for 10^9 of loading cycles is this decrease more than 20%. The decrease of fatigue strength by 20% is significant and this value must be considered in the design of construction operating under similar conditions. The reason of this decrease is unclear. Fractography analysis did not show any differences in the fatigue crack initiation, although two different mechanisms were recorded but its occurrence was similar for both tested states. Small differences were observed in the fatigue crack propagation, when the crack surface of broken sensitised specimens contains the high amount of intercrystalline fatigue crack propagation areas. In high cycle fatigue and especially ultra-high cycle fatigue, fatigue crack propagation takes only a small number of cycles and the most loading cycles are related to the fatigue crack initiation. For 10^9 cycles fatigue crack propagation takes less than 0.01% fraction of all loading cycles, thus small changes in the fatigue crack propagation in sensitised specimens cannot be the reason for the fatigue strength decrease in the ultra-high cycle area. This decrease must be connected with the fatigue crack initiation stage. Fatigue strength is strongly related with the grain size [18] and long exposures to elevated temperatures could result in the grain boundary migration, but temperature 600°C is not sufficient for the grain growth, what was verified by the grain size observation and measurement, where no signs of boundary migration were recorded. In the similar study [19], made on the AISI 316L stainless steel, sensitised at similar conditions, no effect of sensitisation on the fatigue properties in the ultra-high cycle area was recorded. The main differences between these grades are that 316Ti contains 0.37% of Ti but also, the carbon content is more than double compared with 316L. One theory should be considered in relation to these two studies. Precipitation of carbides on the grain boundaries and formation of the chromium depleted zones has another significant consequence. Chromium in austenitic stainless steels is the substituent in the solid solution. Chromium is the main element responsible for corrosion properties, but also, it strengthens the matrix by the solid solution strengthening mechanism and chromium content affect the strain induced martensitic transformation. Lower chromium content in the depleted zone thus will cause a local decrease of mechanical properties. Fatigue crack initiation and the propagation of the short fatigue cracks determine resulting fatigue properties in the ultra-high cycle region and formation of zones, with a local decrease of mechanical properties can result in a preferential slip in this areas and this can cause the acceleration of fatigue crack initiation process and the decrease of fatigue strength. This theory requires another precise experiment, focused on the determining the effect of the chromium content on the local mechanical properties.

Conclusions

Based on the experiments carried out in this study, the following conclusion could be stated:

- Heat treatment 600°C/100h result in the sensitisation of austenitic stainless steel AISI 316Ti.
- Fatigue test of AISI 316Ti in the ultra-high cycle area show continuously decrease of fatigue strength with increasing number of cycles.
- There was recorded the negative effect of the sensitisation on the fatigue properties of the AISI 316Ti in the ultra-high cycle region. This effect was stronger at lower loading amplitudes and higher numbers of loading cycles, where the decrease of fatigue strength by 20% was recorded in comparing with the initial state, while at higher loading amplitudes and lower numbers of loading cycles was the difference in fatigue strength almost zero. Nature of this decrease is unclear and it must be subjected to further investigation.

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