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## THE MICROSTRUCTURAL EVOLUTION OF THE WELDED JOINT OF SUPER 304H STAINLESS STEEL AFTER 1000 HOURS OF AGEING AT 700 AND 750°C

This study presents the results of microstructure and hardness tests of a homogenous welded joint made of Super 304H stainless steel after 1000 h of ageing at the temperature of 700 and 750°C. Super 304H steel is commonly used for high-pressure elements of modern power units for supercritical steam parameters. The article compares hardness with the microstructure images in the initial state and after ageing. The microstructure tests were carried out based on scanning microscopy, while the hardness tests were carried out on a stationary hardness tester in the HV10 scale. An increase in hardness after ageing was observed, which is directly related to the initiated precipitation process, both in the weld and base materials.

*Keywords:* Steel Super 304H; microstructure; ageing

### 1. Introduction

The current and prospective state of Poland's energy sector is and will continue to be based on the so-called energy mix, which consists mainly of the share of energy production from fossil fuels and the percentage of production from renewable energy sources [1].

Nevertheless, the continuous growth of energy demand in Poland requires further development of the energy industry using conventional energy sources for the country's energy security, despite the increase in the share of energy from renewable sources [2]. The primary direction of development is related to the successive increase in power generation efficiency by raising the operating parameters of steam. Achieving supercritical parameters in power units requires using new-generation materials with higher creep strength and heat resistance than steel with a ferritic matrix capable of operating at temperatures as high as 700°C [2]. These include, among others, new steels consisting of an austenitic matrix [1], including Super 304H stainless steel (X10CrNiCuNb18-9-3), which was created by modifying the chemical composition of classic 18/8 type steel.

Electricity demand continues to grow worldwide and is directly related to countries' economic development, industrial production, energy intensity and management [3,4]. Economies

worldwide are heavily dependent on energy; the main sources are conventional power plants, of which ~40% are coal-fired units [5]. However, despite the European Union's increasingly stringent environmental criteria, the domestic power industry will, for many years, be based on burning coal with a gradual reduction of its share in the so-called energy mix [1].

The PEP (Polish Energy Policy) assumes that fossil-fuel power generation will decrease from the current 76% to about 56% in 2030. This shows that high-temperature power generation will remain Poland's primary energy source for at least another 20 years [1,6]. Therefore, some changes have been made in the conventional power industry over the past few years due to economic and environmental conditions. The main challenge has become raising the efficiency of power units. This goal is being realized, among other things, by increasing the parameters of steam, which determines the search for new grades of materials.

The modernity of power technologies can be determined by two main parameters: the efficiency of the unit and the associated amount of environmentally harmful emissions [7]. To reduce the level of CO<sub>2</sub> in the atmosphere and save fossil fuels, it is necessary to increase the efficiency of electricity production. This necessitates the development of new steels and alloys for operation at elevated and high temperatures. In addition, materi-

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als used for boiler components must ensure stable retention of mechanical properties: yield strength, creep strength, low susceptibility to embrittlement growth, and corrosion resistance for up to 200000 h. This is possible by maintaining stable material structure and physical properties during operation [8].

Currently, a viable direction for the development of the national power industry is the modernization of old units with elevated steam parameters ( $>25$  MPa/ $570^{\circ}\text{C}$ ) with an efficiency of 40% and the construction of modern units with supercritical parameters ( $>29$  MPa/ $600^{\circ}\text{C}$ ) and an efficiency of 45% net [9]. Super 304H stainless steel is used to construct pressure components of boilers with supercritical operating parameters, primarily for steam superheater elements, mainly for top-stage coils [10]. Super 304H steel tubes also show high oxidation resistance [11]. However, the strength properties are negatively affected by sulfur and phosphorus, the presence of which in the alloy is considered to be its impurity, which can lead to a decrease in the plasticity of the steel. In addition, large non-metallic inclusions contribute to the material's brittleness [12].

Of all the mechanical properties, those determined by creep testing are the most important and decisive in deciding suitability for service under creep conditions. Creep strength, the basis for design calculations, determines the ability to carry service loads of components made of the tested steel. Long-term operation results in a decrease in the temporal creep strength. It is, therefore, necessary to know, for different states of the material after other operating times, the value of this creep strength, defined as residual life or residual creep strength [1].

To guarantee the energy security of a country where power plants are based on the combustion of fossil resources, it is necessary to constantly improve and increase the efficiency of power equipment. This task can be accomplished by using increasingly modern steels and alloys that will enhance the performance of boilers and ensure safe and trouble-free operation for a long time [13].

An important issue in determining the service life of the austenitic stainless steel – type Super 304 is the preparation of the material database, both in the initial state and after long-term ageing and creep. A data bank in the form of an atlas of microstructures with the identification of secondary phase precipitates and mechanical properties will allow for the analysis of this steel's loss of service life in real operating conditions.

## 2. The methodology and AIM of the study

This study aimed to carry out mechanical tests and observe the microstructure of a welded joint in the as-delivered state and after ageing at temperatures of  $700$  and  $750^{\circ}\text{C}$  for 1000 h. This research is part of a broader study aimed at assessing the service life of welded joints made of Super 304H steel, defined as the material's ability to maintain the required service properties until a contractual limit state is reached, at which further operation involves the risk of rupture.

Observation of the microstructure was carried out using a scanning electron microscope (SEM) Inspect F on electrolytically etched samples.

The hardness test was carried out using the Vickers method per the standard of EN ISO 6507-1.

## 3. The preparation of samples for testing

Samples for laboratory testing were taken from  $48.4 \times 6.3$  pipe made of austenitic stainless steel – type Super 304 (X10CrNi-CuNb18-9-3). The chemical composition of the tested material is shown in TABLE 1. The pipe sections were joined using circumferential welds using the traditional TIG (141) method with a filler material of trade name Thermanit 617 and chemical composition shown in TABLE 2.

The welds were examined for weld inconsistencies using a set of volumetric non-destructive tests and surface tests. Positive verification of the welds allowed the cutting of specimens as-delivered and aged for 1000 h at  $700^{\circ}\text{C}$  and  $750^{\circ}\text{C}$  in the air atmosphere.



Fig. 1. Characteristic areas in the welded joint

After ageing, cut-outs were taken from the samples in characteristic sections of the welded joint, taking into account: base metal 1 and 2, heat-affected zones 1 and 2, and the weld (Fig. 1). From the cuttings, metallographic samples were made.

TABLE 1

Chemical composition of Super 304H steel (% wt.)

Chemical analysis of Super 304H steel (% wt.)											
C	Si	Mn	P	S	Cu	Cr	Ni	Nb	B	N	Al
0.08	0.19	0.80	0.03	0.001	3.02	18.5	8.7	0.84	0.0004	0.11	0.009

TABLE 2

Chemical composition of the TIG rods Thermanit 617 (% wt.)

Chemical composition of the TIG rods Thermanit 617 (% wt.)									
C	Si	Mn	Cr	Mo	Ni	Co	Al	Ti	Fe
0.05	0.1	0.1	21.5	9.0	Bal.	11.0	1.3	0.3	0.5

## 4. Research results

### 4.1. Observation of steel microstructure

In the as-delivered condition, the tested steel is characterized by a fine-grained austenitic structure with grain size number 7-9 according to ASTM E112 standards and visible annealing twins with both coherent and incoherent boundaries and single primary NbCrN (MX) precipitates of varying size, distributed inside the grains (Fig. 2). Previous studies [14,15]

have shown similar results typical of this steel grade. The fine-grained structure provides the steel with good tensile properties expressed in terms of impact strength, strength, and ductility. In addition, it positively affects oxidation resistance compared to coarse-grained steels.

In the tested steel, after 1000 hours of ageing (Fig. 3), secondary  $M_{23}C_6$  and primary MX precipitations inside the grains are present at the grain boundaries. Similar test results after ageing at 650°C were published in [16]. A characteristic feature is the discontinuous arrangement of  $M_{23}C_6$  carbide particles across

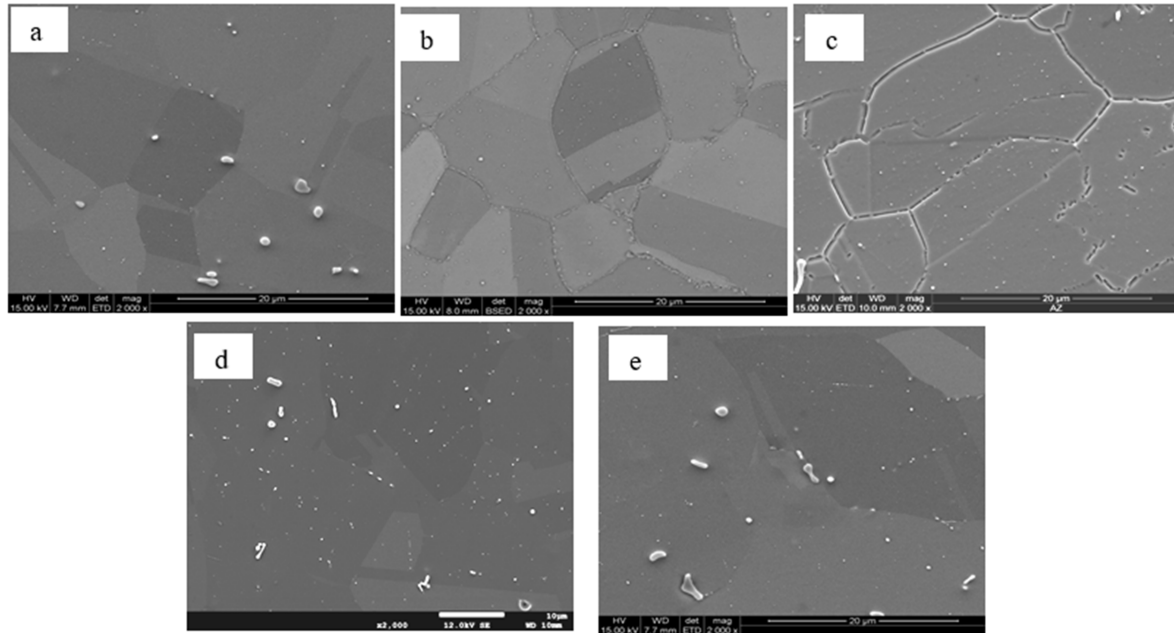


Fig. 2. Microstructure of Super 304H steel in the initial state, (a) – Base material 1, (b) – Heat affected zone 1, (c) –Weld, (d) – Base material 2, (e) – Heat affected zone 2

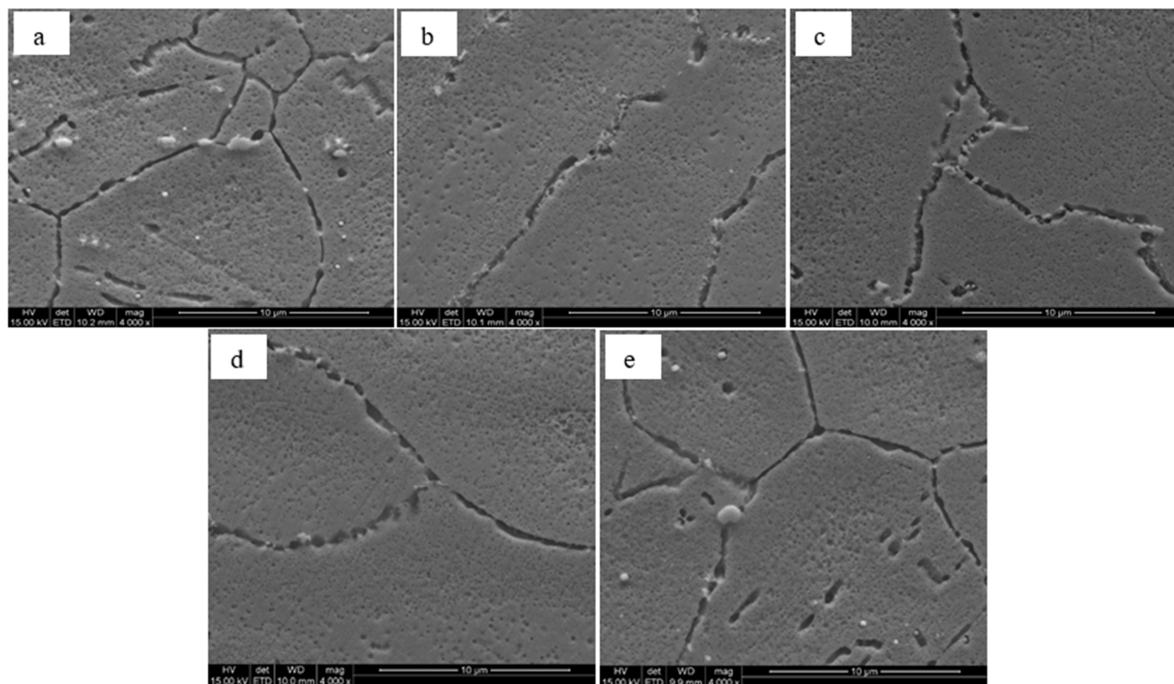


Fig. 3. Microstructure of Super 304H steel after 1000 hours of ageing at 750°C, (a) – Base material 1, (b) – Heat affected zone 1, (c) –Weld, (d) – Base material 2, (e) – Heat affected zone 2

the grain boundaries. Compared to the interior of the grains, the grain boundaries, as surface defects with disordered crystal structures, allow faster diffusion of alloying elements.

#### 4.2. Hardness test

Fig. 4 shows the hardness charts (HV10) of Super 304H steel. In the delivery condition, the material showed the most significant variation in hardness in the welded joint zones. After ageing at 700°C, hardness was observed in all zones with a smoothing of the hardness curve compared to the material in the delivery condition. After ageing at 750°C, a decrease in hardness was observed compared to the material in the delivery state. Similar hardness measurement results for a similar butt joint made of Super 304H steel were also obtained in the article [3].

Long-term ageing is one of the main methods of simulating the operating conditions of materials operating at elevated and high temperatures [1]. Simulations of changes in the microstructure of austenitic steels are carried out through the ageing process at a specific temperature to know their impact on the change in performance properties. This process is carried out at a temperature close to the expected operating temperature for at least tens of thousands of hours. Austenitic stainless steel – type Super 304 and its welded joint cause an improvement in strength properties and a decrease plastic properties in the initial period [1]. Increasing the ageing temperature to a certain level, usually not exceeding 50÷100°C of the maximum long-term operating temperature, also contributes to the increase in the dynamics of the ongoing processes.

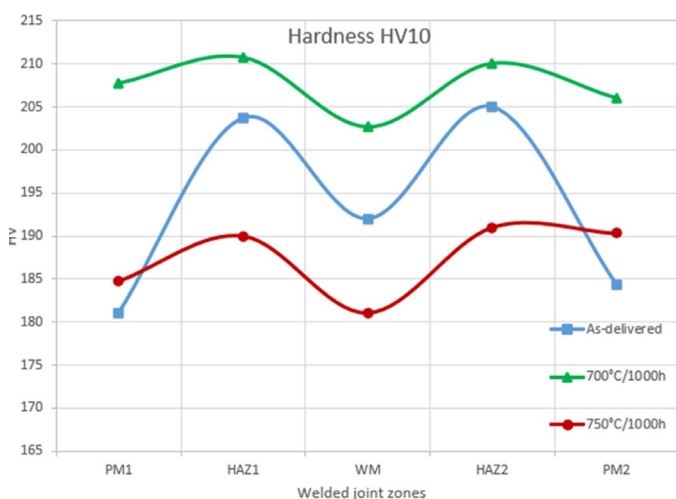


Fig. 4. Results of hardness measurement

#### 5. Conclusions

- In the results of the microstructure and hardness measurements, it can be stated that the strengthening process of this

steel has begun, as evidenced by the observed numerous very fine precipitations of secondary phases in the microstructure and the increase in hardness adequate to the ageing time and temperature.

- No material discontinuities in the form of micro-cracks were found in the tested weld of the joint after 1000 h ageing.
- Ageing the welded joint for 1000 h at 700°C increased hardness in all zones of the joint, while ageing at 750°C decreased hardness below the values obtained for the joint in delivery condition.
- Continued testing at longer ageing times will allow observation of more pronounced microstructural degradation and accompanying changes in mechanical properties in the welded joint.

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