

Mechanical properties of Super 304H steel after long-term ageing at 650 and 700°C

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Abstract. The paper shows the degradation process of the modern austenitic Super 304H (X10CrNiCuNb18-9-3) steel which was subjected to long-term ageing for up to 50 000 h at 650 and 700°C. The investigations include microstructure examination (SEM), identification and analysis of the precipitation process, and mechanical properties tests. The Super 304H steel has a structure characteristic of austenitic steels with visible annealing twins and single primary NbX precipitates. Long-term ageing in steel leads to numerous precipitation processes of $M_{23}C_6$, MX carbides, σ phase, Z phase, and ϵ -Cu phase. Precipitation processes lead to a decrease in plastic properties and impact energy as well as alloy over ageing. Yield strength and tensile strength values after 50 000 h of ageing were similar to those as delivered. The yield and tensile strength value strongly depend on the applied ageing temperature.

Key words: super 304H steel; mechanical properties; ageing.

1. INTRODUCTION

One of the most important parameters determining the modernity of a power boiler is its efficiency, thus meeting environmental requirements, i.e. minimising emissions of greenhouse gas and harmful pollutants into the atmosphere [1–3]. These two factors determine the development of conventional energy [4–6]. The first is to ensure the security of the electricity supply [7–10]. The second factor is the environmental aspect. The use of supercritical steam parameters allows us to obtain an efficiency of power units above 45% [11–13]. It should also be remembered that more favourable combustion conditions for fossil fuels reduce the emission of pollutants into the atmosphere. On the other hand, by using the flue gas treatment installation, it is possible to significantly reduce their emission further [14–16].

The operation of power units in conditions of supercritical steam parameters requires the use of steel with creep strength and greater creep resistance higher than in the case of standard materials in critical boiler components [17–20]. One of the modern steel grades is Super 304H (X10CrNiCuNb18-9-3) [21,22]. It is an austenitic chromium-nickel steel with the addition of copper, with an average chromium content of approx. 18%, nickel 9% and copper 2%, resistant to corrosion [13]. This steel shows a sufficiently high creep strength, which is 68 MPa at 700°C for 100 000 h [23], which is the result of strong hardening with solution heat treatment and precipitation

of the steel [24]. In the power industry, this steel is intended for seamless pipes for applications in the operating temperature range from 600 to 700°C.

The use of a new grade of steel for critical boiler components should be preceded by a series of long-term tests [25–27]. This, in turn, requires thorough tests in both laboratory and industrial conditions. One of the main criteria for using the material for boiler pressure part components is the stability of its microstructure and mechanical properties at the assumed operating temperature [28–30]. This stability can be determined by the use of long-term ageing simulating operating parameters, as described in detail in [31,32].

The service life of a structural element, including steam superheater coils, is mainly influenced by preserving the strength and plastic properties and ductility, the stability of the microstructure during long-term operation, and the conditions of supervision and operation [1, 2, 12, 24]. For these reasons, in all considerations and analyses related to the service life of structural elements operating under creep conditions, each object should be treated individually, especially when it is made of a material for which the available knowledge, as in the case of Super 304H steel, is incomplete [24]. This is especially true in situations where operating conditions may deviate from and exceed design intent, including operation in a regulatory system often associated with the operation of renewable sources of energy [27].

Characteristics are developed for the materials of the boiler components to obtain knowledge about their behaviour in real operating conditions [26, 33–35].

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Table 1
Chemical composition of Super 304H steel tube

	Chemical composition, [wt %]											
	C	Si	Mn	P	S	Cu	Cr	Ni	Nb	B	N	Al
Follow-up analysis	0.09	0.20	0.80	0.003	0.001	2.99	18.40	8.80	0.48	0.004	0.11	0.006
VDTÜV 550:12 2012	0.07 0.13	max 0.30	max. 1.00	max 0.040	max 0.010	2.50 3.50	17.0 19.0	7.5 10.5	0.30 0.60	0.001 0.010	0.05 0.12	0.003 0.030

2. MATERIAL AND METHODOLOGY

Here, a new generation of austenitic Super 304H steel (X10CrNiCuNb18-9-3) was considered. The steel test samples were taken from finished products in 42.4×8.8 mm tube sections. The chemical composition of the tested steel is presented in Table 1.

The microstructural studies were carried out using a scanning electron microscope (SEM) on conventionally prepared electrolytically etched metallographic microsections.

The tests of the mechanical properties of Super 304H steel in the initial state and after long-term ageing included a static tensile test at room temperature of 21°C using a universal testing machine with a maximum load of 200 kN, impact test on non-standard samples with dimensions of $7.5 \times 7.5 \times 55$ mm³ with a cut V-type notch, and Vickers hardness measurement with an indenter load of 10 kg (49.1 N). The strength properties used specimens with a diameter of 5 mm, a measuring length of 50 mm was used, and the total length of the specimen was 85 mm. The strength and impact properties were tested in three samples for each material condition prepared from previously aged material in an air atmosphere.

3. RESULTS

Steels and alloys with austenitic matrix used in the power industry are most often delivered in a supersaturated state. They are mainly characterized by solid solution hardening, which ensures high plasticity and good ductility, with a relatively low yield strength and hardness [36,37]. The chemical composition of steels and alloys developed for operation in high temperature and stress conditions, as well as its optimal heat treatment, should be selected to ensure maximum microstructure stability during operation.

Therefore, it is important to understand, often individually for a given type of material, the mechanisms of structure degradation and their influence on broadly understood mechanical properties. Such knowledge is of fundamental practical importance not only in the design of new steels and alloys but more importantly in the diagnostics and durability assessment of very expensive power boiler installations [6,27]. In the case of the tested Super 304H steel, the secondary phase precipitation processes occurring during ageing lead to its additional strengthening through the precipitation mechanism which is the dominant mechanism in austenitic steels used at elevated and high tem-

peratures. As a result of the precipitation and then the increase in the secondary phase particle size, the content of elements included in the precipitates is decreased in the matrix, which visibly lowers the ability to block the dislocation movement by the elements deliberately introduced into the composition of the tested steel and dissolved in the matrix. Additionally, the precipitation strengthening depends on the amount and size of the secondary precipitates referred to as dispersion.

Sample maps of the distribution of alloying elements, enabling the identification of phases released in the tested steel as a result of long-term ageing at 700°C , are shown in Fig. 1.

The tested Super 304H steel was characterized by approximately 9% higher tensile strength (TS) than the required minimum of 590 MPa; however, it did not exceed the maximum allowable value of 850 MPa. The tested steel also had an approximately 25% higher yield strength (YS) compared to the requirements (235 MPa) [24] and elongation was 13% above the required minimum of 35%.

In the initial period of ageing, very small precipitates of M_{23}C_6 , MX, and ϵ -Cu contribute to a significant increase in YS and TS with a slight reduction in elongation (Fig. 2). Both the increase in strength properties and the decrease in plasticity are related to the presence of precipitates. The degree of increase/decrease in properties depends highly on the ageing temperature. The maximum strengthening and the related increase in YS were visible for the temperature of 650°C after 5000 h of ageing and for the temperature of 700°C after 1000 h (Figs. 2a, 2b). A similar level of YS was also presented in [38]. A similar tendency to increase the strength properties of the tested steel due to ageing was observed for TS. The maximum value of TS increased by about 12% after an ageing time of 20 000 h at 650°C and 5000 h at 700°C .

The effect of M_{23}C_6 precipitates along grain boundaries on yield strength was shown in [39]. The size of M_{23}C_6 carbides facilitates easier nucleation of defects in the form of voids and discontinuities at their interfacial boundary. In addition, the resulting continuous lattice of M_{23}C_6 carbides at the grain boundary allows through the intergranular mechanism. Hence, M_{23}C_6 carbides precipitated at grain boundaries are mainly responsible for the decrease in plasticity of the tested steel in the initial period of structure degradation. In the next stage of the ageing process, a significant amount of σ phase precipitates within and outside the grains cause a decrease in plastic properties. The precipitation and increase in particle size of secondary

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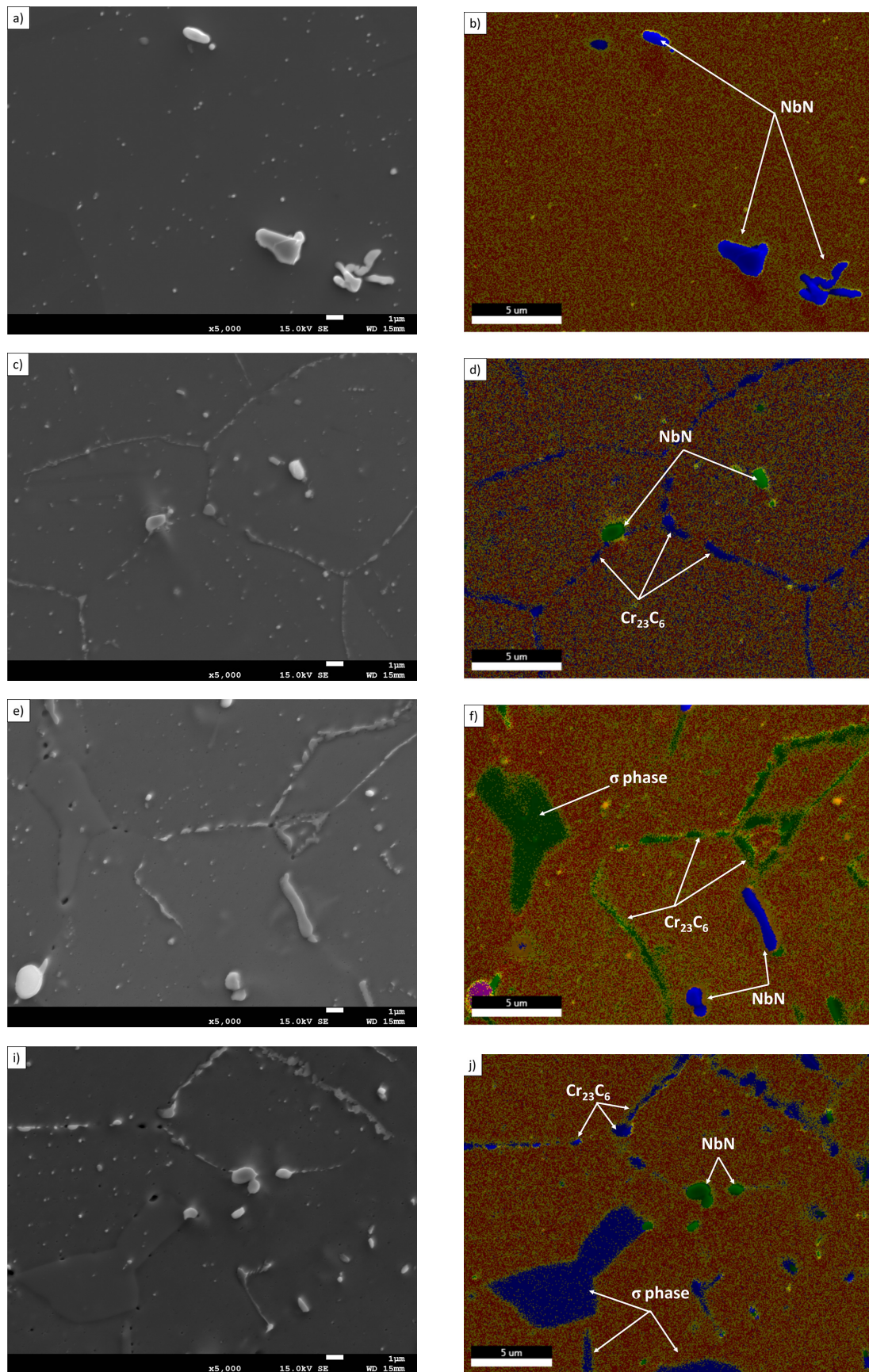


Fig. 1. Microstructure of Super 304H steel, (a, b) – as-delivered and after ageing at 700°C for (c, d) – 1000 h; (e, f) – 10000 h; (g, h) – 30000 h; (i, j) 50000 h, SEM

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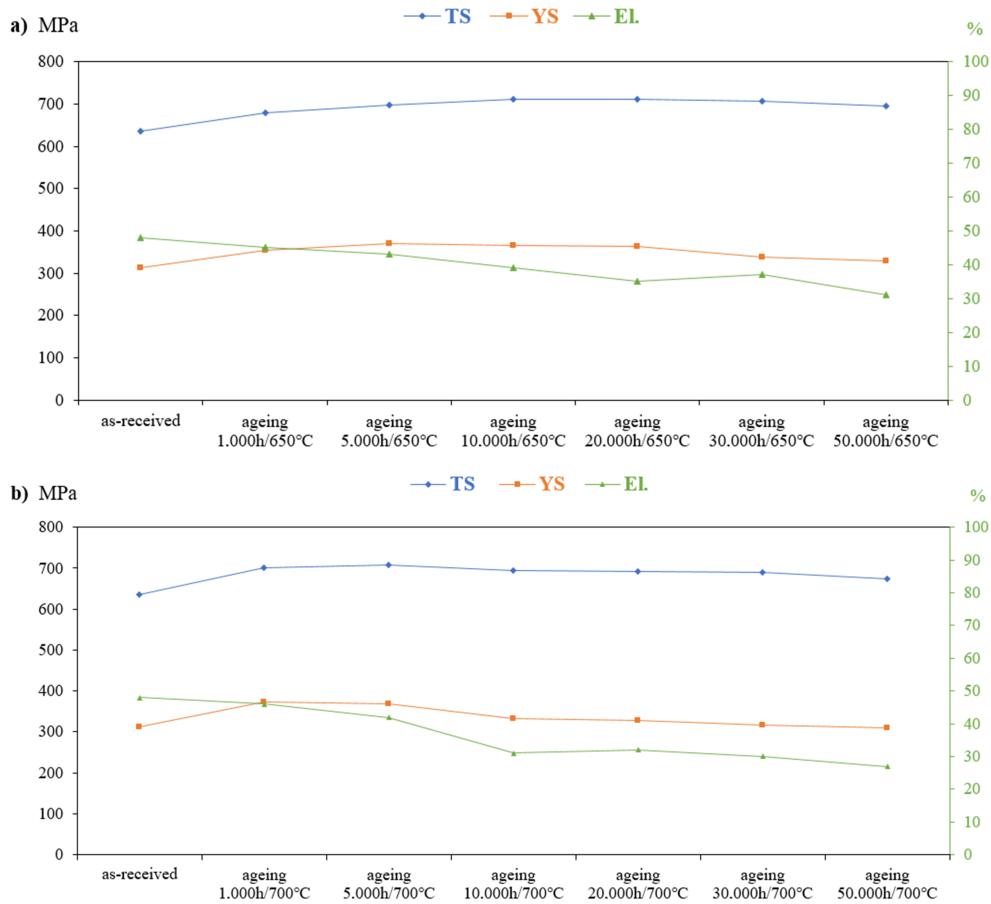


Fig. 2. Influence of ageing time of the Super 304H steel at: a) 650 and b) 700°C, mechanical properties determined by static tensile test at room temperature

phases taking place during ageing also lead to a reduction in the strengthening through the solution mechanism. Additionally, according to [24, 39], during ageing, processes of dynamic recovery and recrystallisation take place in steel and the disappearance of coherent borders of twins is observed. The above changes in the microstructure lead to a gradual decrease in YS, TS, and a further decrease in elongation. The dynamics of these changes strongly depend not only on temperature, but also on the ageing time (Fig. 2). At the lower ageing temperature of 650°C, due to slower nucleation and precipitation growth processes as well as changes in the dislocation structure, the decline in strength properties is slower compared to the higher ageing temperature of 700°C (Fig. 2). The interpretation of the YS and TS values obtained after ageing at 650 and 700°C showed that the values of TS and YS after 50 000 h of ageing at 650°C are similar to the values of TS and YS after 10 000 h of ageing at 700°C (Figs. 2a, 2b). A similar relationship is also observed in the case of the average equivalent diameter of the ϵ -Cu, σ and $M_{23}C_6$ phase precipitates. From here, we conclude that increasing the ageing temperature from 650 to 700°C accelerates the process of the Super 304H steel degradation approximately five times. Nevertheless, for the 50 000 h ageing time in the tested steel, regardless of the ageing temperature, no ageing effect was observed where the strength properties after ageing were still slightly higher than those as delivered (Fig. 2).

The precipitation and increase in the $M_{23}C_6$ carbides precipitates at grain boundaries, in the case of the tested steel, also leads to a sharp decrease in the impact energy to 1000 h of ageing. According to works with shorter ageing times, this decrease is already observed after 100 h of ageing in 650°C [40–42]. Therefore, it is important to ensure stabilisation of the growth of $M_{23}C_6$ precipitates in order to slow down the decrease not only in TS but also in impact energy. In the case of the tested steel, impact energy after 1000 h of ageing was lower than as-received by 50% depending on the ageing temperature (Fig. 3). After 50 000 h of ageing of the tested steel, the level of impact strength, regardless of the ageing temperature, is high compared to the competing HR3C steel [43]. The decrease in impact energy in the Super 304H steel should be related primarily to the continuous lattice of $M_{23}C_6$ carbides at grain boundaries (Figs. 3, 4), as well as their growth and coalescence, which significantly weakens the grain boundary cohesion. This decrease may also result from the disappearance of the coherent twin boundaries and the precipitation of $M_{23}C_6$ carbide twins on incoherent boundaries [43]. Due to the above, the boundaries of austenite grains and the boundaries of twins are areas of low-energy cracking propagation by the intergranular mechanism. The reduction of plastic properties measured by impact energy is also influenced by the morphology of $M_{23}C_6$ precipitates precipitated on grain boundaries. The σ phase at

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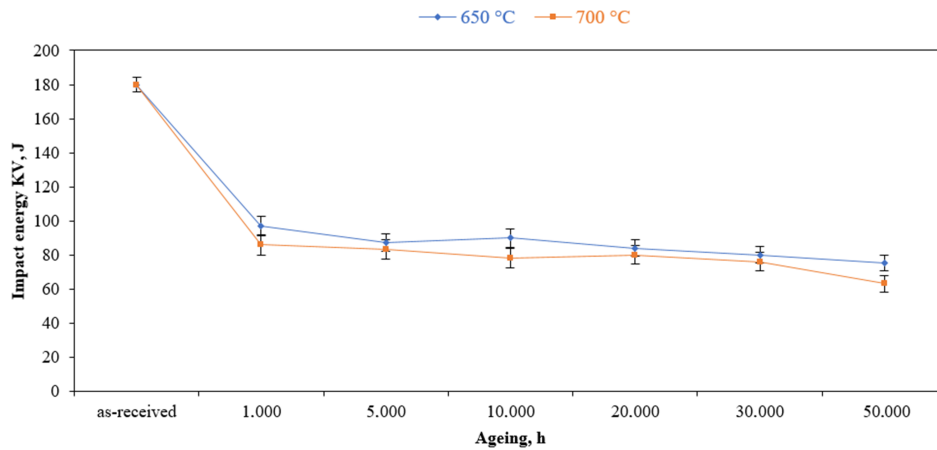


Fig. 3. Impact of temperature and ageing time of the Super 304H steel on impact energy

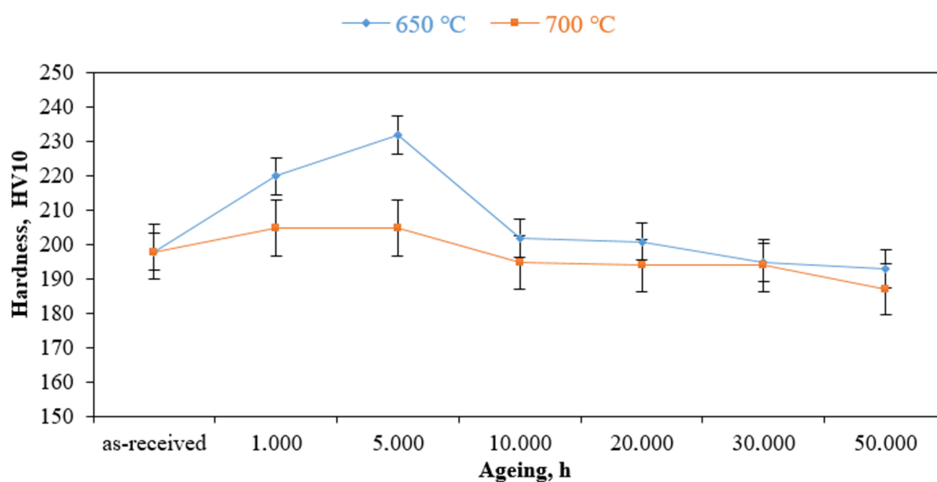


Fig. 4. Change in HV10 hardness depending on the Super 304H steel ageing time and temperature

grain boundary observed after a longer ageing time does not affect the impact strength deterioration of austenitic steel as rapidly as it is in the case of the initial precipitation of $M_{23}C_6$ carbides in the initial period of ageing [43]. In the case of tests on changes in the impact strength of the Super 304H steel as a result of long-term temperature impact, a similar trend in the progress of the degradation process is observed as in the case of tests on strength properties. The impact strength at an ageing temperature of 650°C after 50 000 h of ageing is similar to that of the Super 304H steel after 700°C/10 000 h ageing.

The results of hardness measurement after ageing for up to 50 000 h are presented in Fig. 4. It was shown that, in particular at 650°C, hardness increases by roughly 10% in the first period of ageing (up to 5000 h) compared to the as-received condition, i.e. to the value of 220–230 HV10. The observed precipitation processes taking place during the ageing of the tested steel contributed to the increase in hardness, the dynamics of which depended on the ageing temperature. This is mainly due to the precipitation of the finely dispersed ϵ -Cu phase after ageing for 10 000 h. Additionally, hardness begins to drop to the as-received state and amounts to 193 and 187 HV10 after ageing for 50 000 h for an ageing temperature of 650 and 700°C.

The registered decrease in hardness compared to the as-received condition is approximately 3%. The decrease in hardness is related to the gradual softening of the matrix and coagulation of the precipitates. It is also caused by an increase in the average diameter of the ϵ -Cu phase precipitates, but also by a change in their chemical composition, which means that with the ageing time this phase is richer in copper atoms and poorer in Fe, Cr and Ni [38]. Due to the lack of characteristic differences in the level of hardness of the tested Super 304H steel, the hardness measurement cannot be a criterion for assessing the degree of changes in mechanical properties (for an ageing time of up to 50 000 h) and even more so in estimating the degree of degradation of functional properties.

4. SUMMARY

The study was conducted on the Super 304H austenitic steel in the as-received condition and after ageing at 650 and 750°C up to 50 000 h. The tests were performed to allow the following conclusions to be drawn:

- The Super 304H steel microstructure degradation was mainly related to secondary-phase precipitation occurring

at the boundaries ($M_{23}C_6$, σ phase) and inside the grains (MX, σ phase, ϵ -Cu phase, Z phase). The rate of this process depends on the ageing temperature.

- In the initial period, long-term ageing of the Super 304H steel leads to a dynamic increase in the strength properties (TS, YS) with simultaneous reduction in impact strength, and then, as a consequence of the extended ageing time, it leads to a gradual decrease in both TS, YS and impact strength. The temperature 700°C significantly influences the dynamics of microstructure changes and the corresponding properties.
- Stated hardness fluctuations and reduction of ductility measured by breaking work after various ageing times at both 650 and 700°C.
- The presented test results are applicable for the evaluation of durability of power equipment elements operating in creep conditions.

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