Aging behaviour of particular stainless-steels and NiFeCr alloy suitable for heat exchangers

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Abstract. This paper deals with the testing of three materials for special heat exchanger for short-time application. Mechanical and microstructural properties after aging at 650 and 850 °C were tested and analysed. The results will serve as an input data for the design and construction of plate heat exchanger.

1 Introduction

The use of stainless steels as a construction material is often the only possible solution in terms of operation (parameters, environment), lifetime, safety. The application of stainless steels is mainly concerned with the possibility to increase the parameters of the technological equipment, guaranteeing long-term service life and maximum safety. Austenitic stainless steels are especially used in non-standard environments where high resistance is required. The matrix of austenitic stainless steels is formed by austenite and according to the content of other alloying elements, ferromagnetic phases - delta ferrite and deformation martensite, carbides and precipitates of various types, sigma and chi-phase, nitrides and inclusions can be present in the structure. All of these components affect the durability of austenitic stainless steels. [1]
When these steels are slowly cooled after heat treatment or welding, the $Cr_{23}C_6$ precipitates along the grain boundaries in the critical temperature range of approximately 420 ° C to 820 ° C for these steels. Of course, this process also occurs when the austenitic component is exposed to a high operating temperature. [2-4]. Various producers of austenitic stainless steels guarantee the creep resistance of particular grades in the range from 15 to 100 MPa for temperatures 750 – 600 °C. [5]
Other materials which have become standard for high temperature application are nickel-based alloys. These alloys generally offer high corrosion and wear resistance when exposed to high temperatures. [6]

2 Experiment description

2.1 Experimental material

Three alloys – 2 austenitic steels and one nickel alloy were selected for this experiment. The steel grades were austenitic stainless steel 1.4541 (AISI 321) – marked as specimen 4 in following tables and figures and 1.4571 (ASI 316Ti) – marked as specimen 7. Incoloy alloy 800 HT (1.4959) – marked as specimen 5, represented the nickel-iron-chromium alloys. Standard chemical composition of experimental alloys is listed below (see table 1).

Table 1. Chemical composition of experimental materials (Fe is balance).

Steels	C	Si	Mn	P	S	Cr	Mo	Ni	Ti
1.4541	Max 0.08	Max 1.00	Max 2.00	Max 0.045	Max 0.030	17.0–19.00	x	9.00-12.00	5xC
1.4571	Max 0.08	Max 1.00	Max 2.00	Max 0.045	Max 0.030	16.5-18.50	2.00-2.50	10.5-13.50	5xC
NiCrFe Alloy	**C**	**Si**	**Mn**	**P**	**S**	**Cr**	**Ni**	**Al**	**Ti**
1.4959	Max 0.10	Max 0.70	Max 1.50	Max 0.015	Max 0.010	19.0-22.0	30.0-34.0	0.25-0.65	0.25-0.65

2.2 Parameters of aging

There were applied two temperatures of aging with different holding times (dwell on the temperature) and water quenching afterwards. Particular parameters are summarised in the table 2. Only one holding time was chosen for the temperature 850 °C.

Table 2. Parameters of heat treatment – aging.

Temperature [°C]	Holding time [h]		
650	5	25	150
850	150		

2.3 Mechanical testing

Tensile tests were performed on an Inova servo-hydraulic test machine with a capacity of 200 kN. The tests were carried out according to EN ISO 6892-1 and according to

EN ISO 6892-2 (at elevated temperatures). A standard tensometric extensometer was used to detect deformation. Prior to and after the test, the characteristic dimensions of the sample for the determination of stress and strain characteristics (YS, TS, E, A5) were measured. The hardness of the individual materials was measured by the STRUERS DuraScan 50 laboratory hardness tester – by means of Vickers method with the HV10 load. Hardness HV10 was measured according to EN ISO 6507-1 - Vickers hardness test. Notch toughness tests were performed according to ISO 148-1. Mechanical testing was performed on samples which were in two states – as received state and the state after aging. Tensile tests were performed at room temperature and at elevated temperatures which were identical with the ageing temperature (650 and 850 °C).

2.4 Microstructural analysis

The analysed samples were subjected to standard metallographic preparation - ie grinding and subsequent polishing. The microstructure of the sample was revealed by etching in the V2A reagent and documented using a Carl Zeiss-Observer.Z1m optical microscope. The microstructure was also analysed by SEM analysis performed on the Jeol JSM 6380 scanning electron microscope. The local chemical composition was measured using the INX x-sight EDX analyser.

3 Results and discussion

3.1 Mechanical properties

The results of the mechanical tests are shown in tables 1 to 3 below. The development of mechanical properties tends to better illustrate the graphs in fig. 1 - 3.

Table 3. Mechanical properties of as received states and aged states obtained after testing at room temperature.

Specimen	Temp.	YS	TS	El
	[°C]	[MPa]	[MPa]	[%]
4_As received	23	289.5	575.1	60.8
4_650°C - 5 h	23	270.9	573.0	59.1
4_650°C - 25 h	23	286.5	570.0	56.4
4_650°C - 150 h	23	293.5	576.7	54.5
7_As received	23	298.2	572.8	55.5
7_650°C - 5 h	23	275.2	575.5	56.6
7_650°C - 25 h	23	307.3	581.9	55.3
7_650°C - 150 h	23	274.6	576.8	53.2
5_As received	23	249.9	561.9	46.0
5_650°C - 5 h	23	256.5	590.9	47.2
5_650°C - 25 h	23	283.8	618.2	42.1
5_650°C - 150 h	23	334.3	671.9	33.2

In case of testing of aged materials at room temperature, the yield strength (YS) is increasing for 1.4541 (longer holding time) and Incoloy (see fig. 1 and table 2). Steel 1.4571 shows drop of yield strength for sample aged for 5 h and 150 h. The values of tensile strength (TS) were not so affected due to aging process for both steels (1.4541 and 1.4571). Substantial increasing of mechanical properties at room temperature is visible for Incoloy where yield strength and tensile strength of aged specimen increased more than 1,3 times and 1,2 times respectively in comparison to as received state.

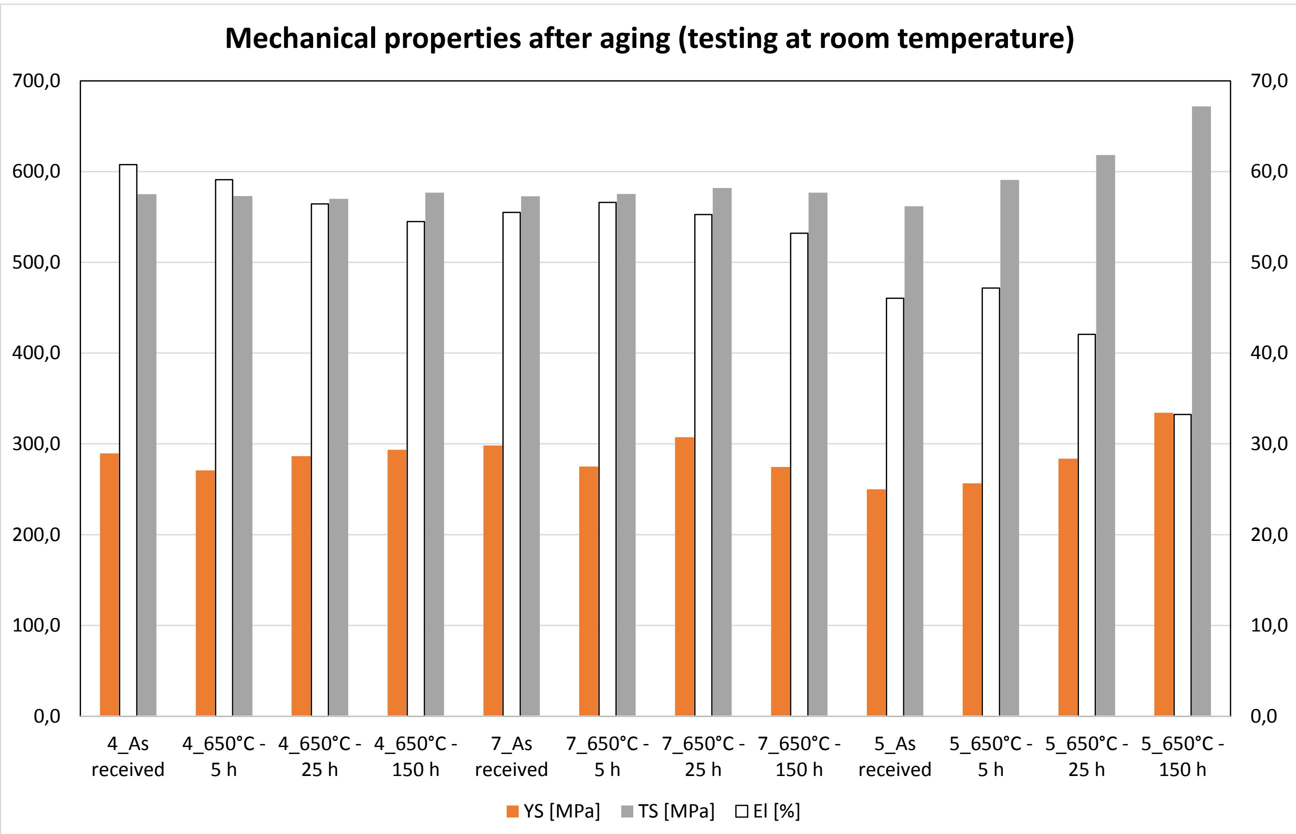

Figure 1. Mechanical properties after aging. Tests were performed at room temperature.

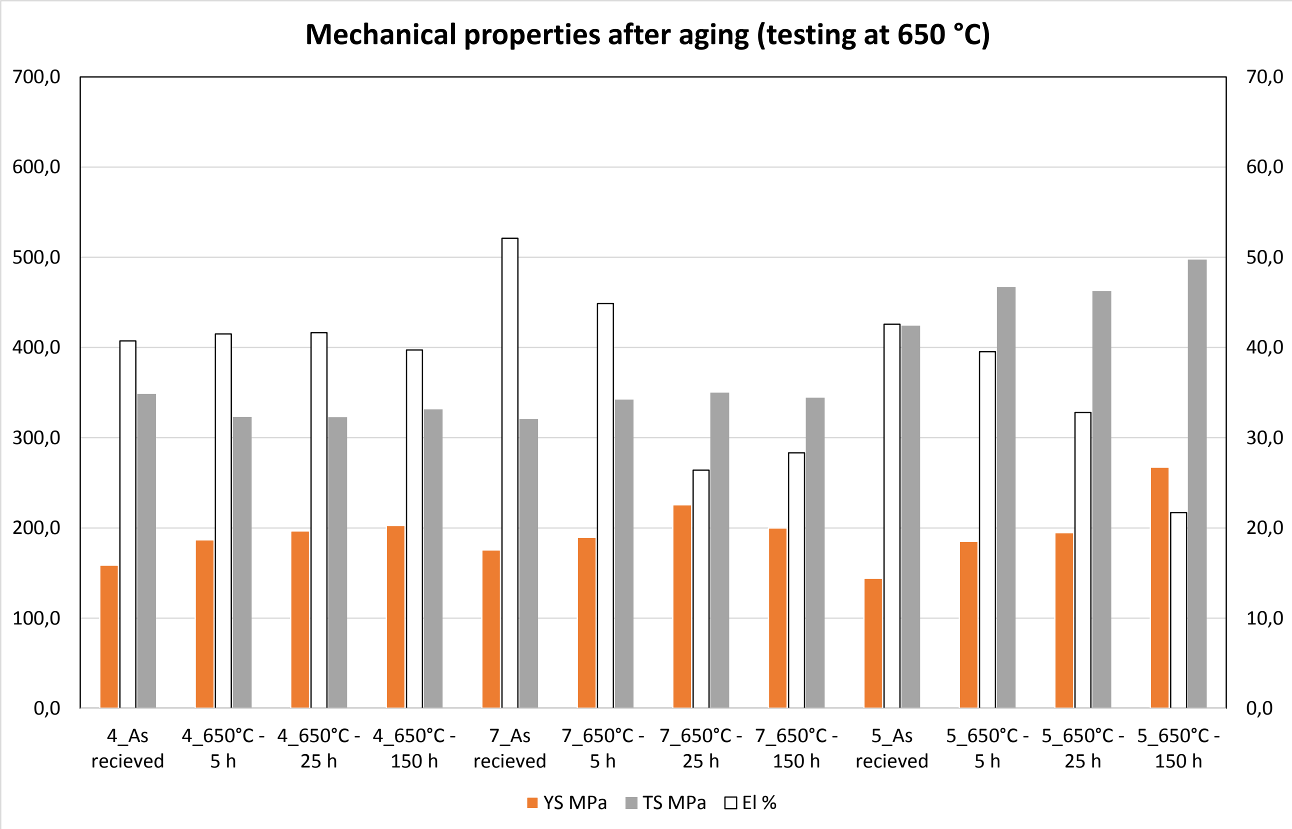

Figure 2. Mechanical properties after aging. Tests were performed at 650 °C.

Trends of increasing both YS and TS were observed in case of mechanical properties measured at 650 °C (see fig. 2 and table 4).

Table 4. Mechanical properties of as received states and aged states obtained after testing at 650 °C.

Specimen	Temp. [°C]	YS [MPa]	TS [MPa]	El [%]
4_As received	650	158.8	348.6	40.7
4_650°C - 5 h	650	186.8	323.0	41.5
4_650°C - 25 h	650	196.6	322.9	41.6
4_650°C - 150 h	650	202.7	331.5	39.7
7_As received	650	175.6	320.8	52.1
7_650°C - 5 h	650	189.7	342.2	44.9
7_650°C - 15 h	650	225.7	350.0	26.4
7_650°C - 150 h	650	200.0	344.4	28.3
5_As received	650	144.1	424.2	42.6
5_650°C - 5 h	650	185.2	467.0	39.5
5_650°C - 25 h	650	194.7	462.7	32.8
5_650°C - 150 h	650	267.4	497.5	21.7

Increasing of aging temperature to 850 °C lead to further drop of both yield strength and tensile strength for all three materials. However, the elongation was increased substantially for all three materials. The results of mechanical properties obtained after testing at increased temperature 850 °C are shown in figure 3. Hardness values (tab. 5) show similar trends in comparison to the values of YS measured at room temperature.

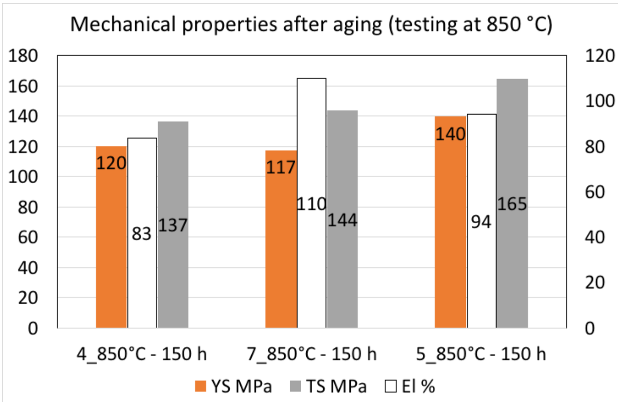

Figure 3. Mechanical properties after aging. Tests were performed at 650 °C.

Table 5. Results of hardness testing.

Specimen	HV10	Specimen	HV10
4_As received	158	7_650°C - 150 h	168
4_650°C - 5 h	169	7_850°C – 150 h	150
4_650°C - 25 h	175	5_As received	120
4_650°C - 150 h	183	5_650°C - 5 h	142
4_850°C – 150 h	150	5_650°C - 25 h	153
7_As received	157	5_650°C - 150 h	183
7_650°C - 5 h	171	5_850°C – 150 h	142
7_650°C - 15 h	209		

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Notch toughness (Charpy test) measured at room temperature showed interesting behaviour for steel 1.4541 and Incoloy 1.4959. The notch toughness values decreased with increasing aging time, however they increased after aging at 850 °C. The notch toughness of steel 1.4571 showed the lowest value at 850 °C. This behaviour is typical for some austenitic stainless steel and it is in compliance with the study of O. H. Ibrahim et al. [2].

Table 6. Results of Charpy test.

Specimen	KV
4_As received	111.1
4_650°C - 5 h	98.8
4_650°C - 25 h	88.4
4_650°C - 150 h	79.5
4_850°C - 150 h	91.5
7_As received	59.9
7_650°C - 5 h	56.7
7_650°C - 25 h	50.9
7_650°C - 150 h	48.8
7_850°C - 150 h	21.7
5_As received	204.3
5_650°C - 5 h	173.4
5_650°C - 25 h	154.4
5_650°C - 150 h	115.1
5_850°C - 150 h	165.5

3.2 Microstructure

3.2.1 Optical microscopy

The microstructure of the 1.4541 material in the as received state (see figures 4. a and b) consists of an austenitic matrix with the presence of a small amount of delta ferrite without the obvious presence of intermetallic phases. The aged states have an austenitic matrix with the presence of a larger proportion of the delta ferrite (see figures 4. c - f). As the time of exposure increases, the carbide phase is formed at the grain boundaries, and in the heat degraded state 150 hours (both 650 and 850 ° C) there is a grain coarsening. At 850 ° C, it is clear that the carbides precipitate within the grain matrix and not only preferentially on their boundaries.
Note: Description of a magnification (100x and 1000x) in the figures caption means the magnification of microscope used for the documentation not the actual magnification of the image.

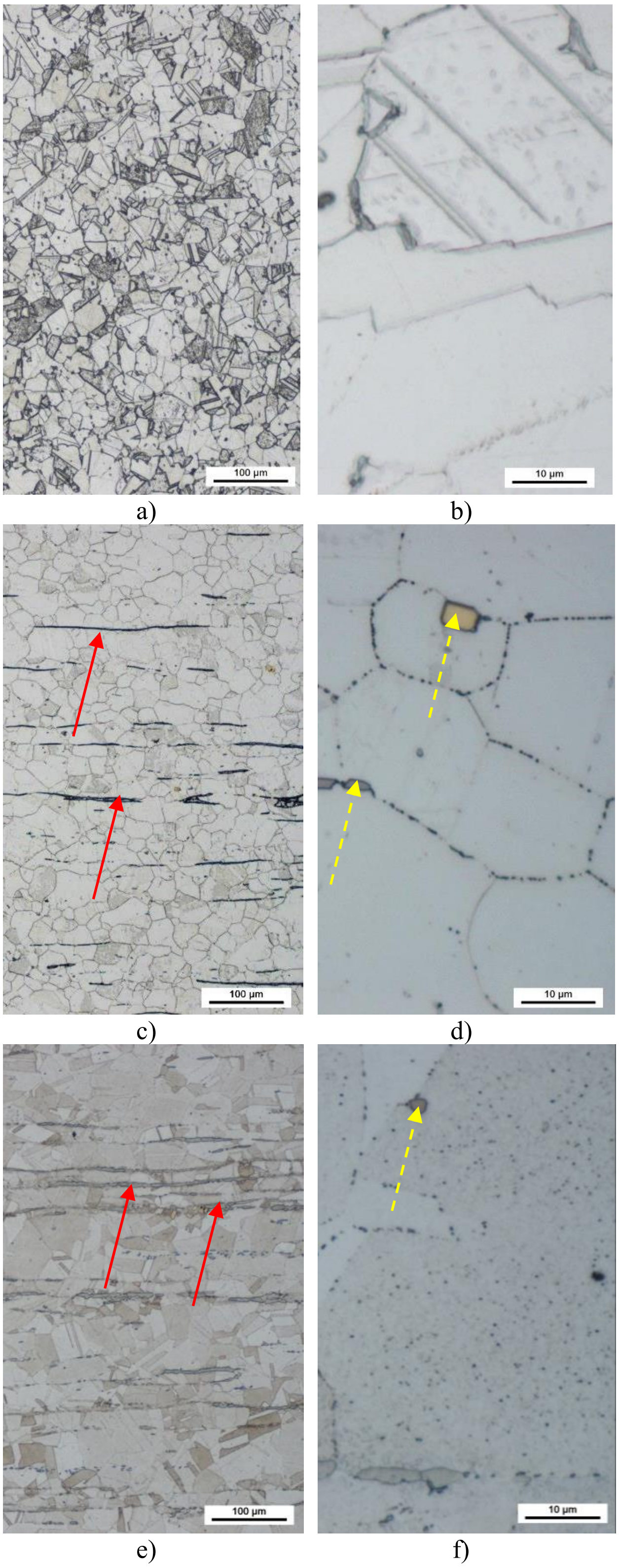


Figure 4. Microstructures of steel 1.4541 in as-received state and after aging. Red arrow marks delta ferrite, yellow arrow with dashed line marks TiC/N particles. a) Specimen 4_as_recieved state – 100x; b) Specimen 4_as_recieved state 1000x, c) Specimen 4_ 650°C - 150 h – 100x; d) Specimen 4_ 650°C - 150 h – 1000x; e) Specimen 4_ 850°C - 150 h – 100x; f) Specimen 4_ 850°C - 150 h – 1000x

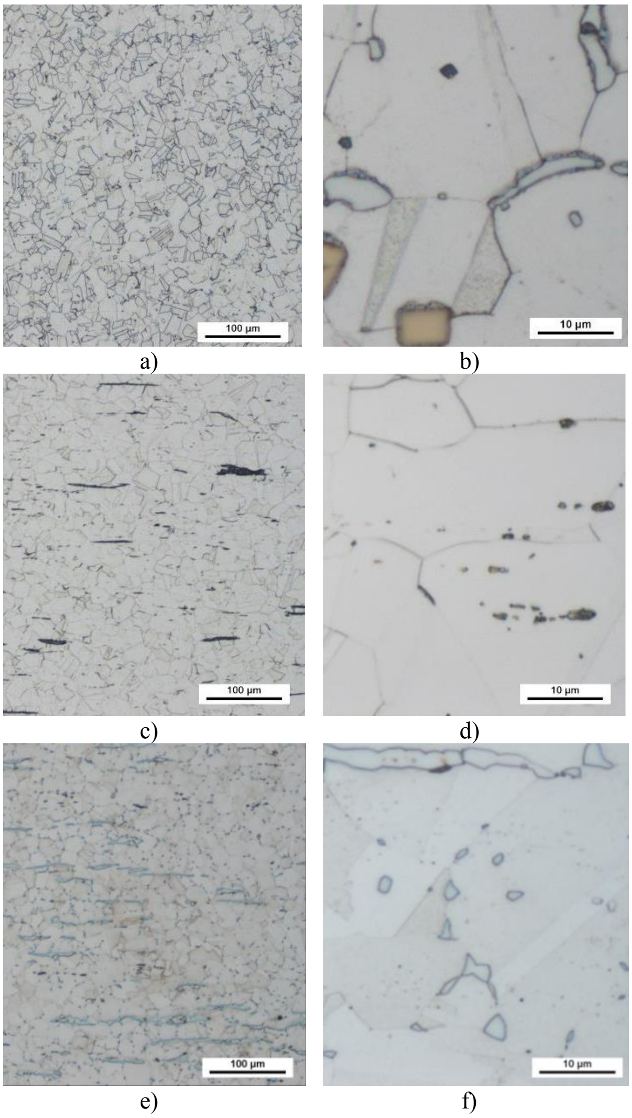


Figure 5. Microstructures of steel 1.4571 in as-received state and after aging. a) Specimen 7_as_recieved state – 100x; b) Specimen 7_as_recieved state 1000x, c) Specimen 7_ 650°C - 150 h – 100x; d) Specimen 7_ 650°C - 150 h – 1000x; e) Specimen 7_ 850°C - 150 h – 100x; f) Specimen 7_ 850°C - 150 h – 1000x

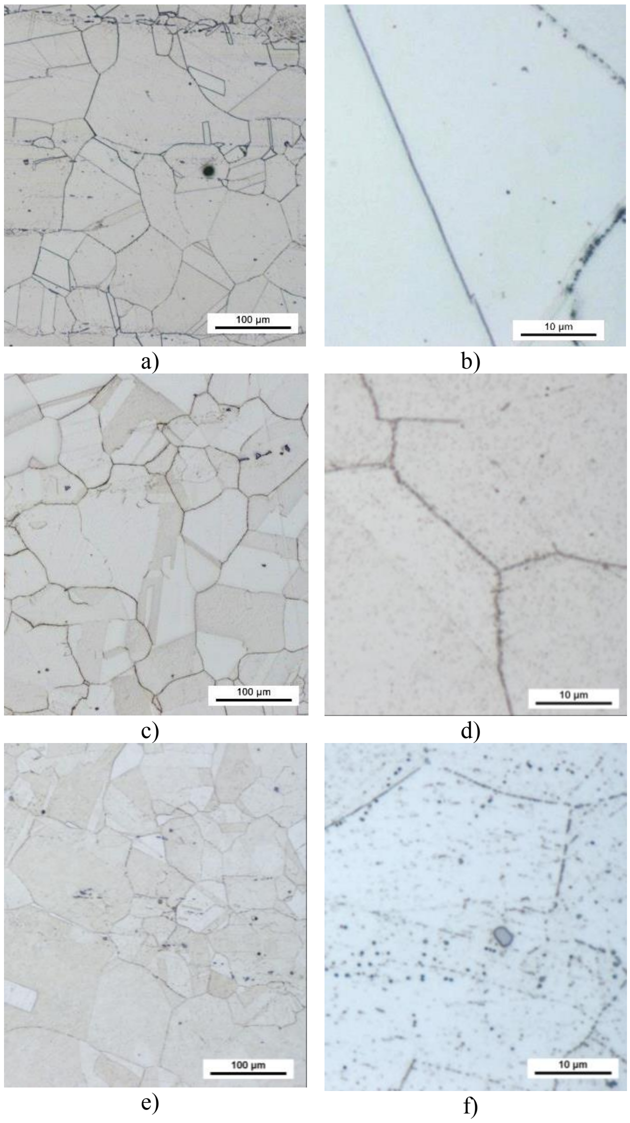
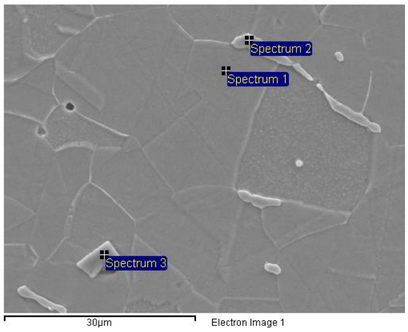


Figure 6. Microstructures of Incoloy 1.4959 in as-received state and after aging. a) Specimen 5_as_recieved state – 100x; b) Specimen 5_as_recieved state 1000x, c) Specimen 5_ 650°C - 150 h – 100x; d) Specimen 5_ 650°C - 150 h – 1000x; e) Specimen 5_ 850°C - 150 h – 100x; f) Specimen 5_ 850°C - 150 h – 1000x

The microstructure of 1.4571 in the as received consists of an austenitic matrix with the presence of delta ferrite. Thermally degraded states have an austenitic matrix with the presence of a larger proportion of delta ferrite. With increasing exposure time, grain growth occurs (see figure 5). The homogeneous microstructure of Incoloy 1.4959 consists of an austenitic matrix with carbide precipitates in grains and at the borders of austenitic grains. With increasing temperature and aging, carbides precipitate inside the grain. Grain coarsening trend with increasing time and aging temperature was not observed in this alloy (see figure 6).

3.2.1 Scanning electron microscopy

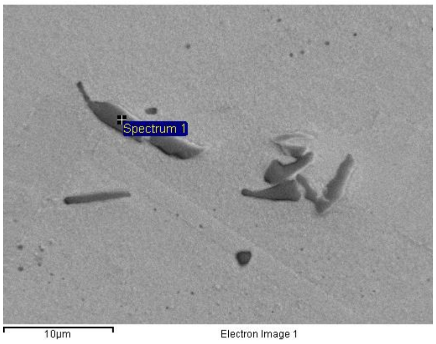
Aged specimens were subjected to EDX analysis for identification of intermetallic particles and secondary phases. Figure 7 shows the localities of EDX measurements of matrix (spectrum 1), delta ferrite (spectrum 2) and TiN particle (spectrum 3) in steel 1.4571.



El.	Spectrum		
	1	2	3
N			16.2
Ti			72.8
Cr	17.4	24.6	
Mn	2.2		
Ni	10.9	5.1	
Mo		4.2	
Fe	bal	bal	bal

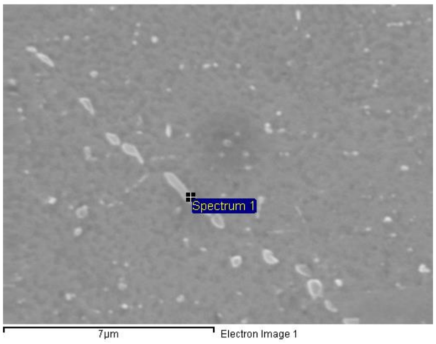
Figure 7. Results of EDX measurements on steel 1.4571 specimen.

EDX analysis of Incoloy revealed the presence of complex carbides composed of Cr, Nb and Ti within the grains (see fig. 8) and chromium carbides (likely $Cr_{23}C_6$) on the grain boundaries (see fig. 9).



El	Spectrum 1
Ti	46.6
Cr	7.5
Ni	5.5
Nb	1.8
Mo	1.8
Fe+C	bal

Figure 8. Results of EDX measurements on Incoloy 1.4959 specimen on particles within the grains.



El	Spectrum 1
C	19.6
Cr	36.7
Mn	0.8
Ni	8.5
Fe	bal

Figure 9. Results of EDX measurements on Incoloy 1.4959 specimen on carbides on the grain boundaries.

4. Conclusion

Three alloys – 2 austenitic steels and one nickel alloy were selected for experiment dealing with microstructural and mechanical properties after aging at two temperatures 650 and 850 °C. Microstructure of both steels shows grain coarsening with increasing of temperature and holding time. Volume fraction of delta ferrite was increased after aging. The carbides precipitation occurs mainly on the grain boundaries and also within the grains in case of 1.4541 steel. Grain size of Incoloy was not affected after aging nevertheless the precipitation of chromium carbides on the grain boundaries was also observed in this material. These microstructural changes affected the mechanical properties. Generally, both YS and TS tested at room temperature were increased and the elongation decreased in case of 1.4541 steel and Incoloy. YS of 1.4571 was lower after aging at 650 °C in comparison to as received state. Results of testing at elevated temperatures showed good combination of YS, TS and elongation for 1.4541 steel and Incoloy. Also the notch toughness of these materials was decent in comparison to poor toughness of 1.4571 steel. Best performance of mechanical properties was obtained with Incoloy alloy. Results of these tests will serve mainly for the design and construction of a short-time service helium-helium heat exchanger. However the tested materials could also be applied in oil, gas and vacuum industry.

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