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# THE EFFECT OF LONG-TERM IMPACT OF ELEVATED TEMPERATURE ON CHANGES IN THE MICROSTRUCTURE OF INCONEL 740H ALLOY

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This paper presents the results of investigations on microstructure changes after the long-term impact of temperature. The microstructure investigations were carried out by light microscopy and scanning electron microscopy. The qualitative and quantitative identification of the existing precipitates was carried out using X-ray phase composition analysis. The effect of elevated temperature on precipitation processes of test material were described. The obtained results of investigations form part of the material characteristics of new-generation alloys, which can be indirectly associated with the stability of functional properties under the simultaneous effect of high temperature and stress.

Key words: inconel 740H, microstructure, hardness, carbides, ageing

#### INTRODUCTION

The development of the coal-fired energy industry is conditioned by many factors that should be analysed in four focus groups:

- legal (EU strategy for a low-carbon economy),
- improvement in technical and economic indicators, including mainly the efficiency of above 46% for the boilers being designed and constructed as well as compliance with environmental requirements
- ensuring the security of energy supply,
- material-related development of materials and technologies to enable progress in energy technologies.

The above-mentioned conditions shape the direction of development for the coal-fired energy industry, and their aim is the improvement in efficiency and the significant reduction in harmful emissions by increasing the values of thermodynamic parameters of the steamwater circulation system [1-3].

The achievement of the goal set by the EU, and also by the American and Asian countries, which is the target efficiency of 50+ power units, is conditioned by the availability of an adequate range of pipes from the new materials, which meet high requirements for functional properties. These materials include austenitic steels (HR3C, Super 304H, Sanicro 25) [4-6] and nickel-base alloys (HR6W, Alloy 617, Alloy 740H) [7-8]. This requires the acquisition of an adequate knowledge not

only on the stability of structure and functional properties of the materials used, but also mastering the technologies for manufacturing of structural components from these materials, particularly in view of the welding and bending technology [1], as it is known that durability of material is not identical to durability of a finished structural component. Therefore, the functional properties of the materials to be used in the as-received condition should be distinguished by sufficiently high values (e.g. temporary creep strength, impact energy) so that they will not get worse during the technological processes (welding, bending, heat treatment) [9-10].

## MATERIAL FOR INVESTIGATIONS

The material for investigations was nickel-base superalloy Inconel 740H (NiCr25Co20TiAlNb). The chemical composition of test alloy with reference to the requirements of the standard is presented in Table 1. Inconel 740H is intended for components of ultra-critical steam boilers (temperature 700 - 760 °C, pressure approx. 35 MPa). This grade is a derivative of Nimonic alloy 263 the development of which in its initial stage has resulted in obtaining Inconel 740 alloy followed by, though the modernisation of its chemical composition (optimisation of Nb, Ti, Al levels and reduction in Bi, Si levels), Inconel 740H alloy.

This alloy is characterised by high creep strength compared to other nickel-base alloys, which is 210 and 120 MPa at 700 and 750 °C, respectively, for extrapolated time of 100 000 h. It is a result of the solution hardening by cobalt and molybdenum and the precipitation process with  $\text{Cr}_{23}\text{C}_6$  carbides and  $\gamma'$  Ni<sub>3</sub> (Ti, Al, Nb) intermetallic phase precipitating at 700  $\div$  800 °C. Due

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Table 1 Chemical composition / wt. %

	С	Mn	Cr	Ni	Ti	Мо	Со	Nb	Al
Test material	0,03	0,10	24,37	REST	0,87	1,56	20,67	0,65	1,44
To UNS N07740	0,005	≤1,0	23,50	REST	0,50	≤2,0	15,00	0,50	0,20
	0,08		25,50		2,50		22,00	2,50	2,00

to the high chromium content, this alloy shows an excellent resistance to high-temperature corrosion and steam oxidation. Its creep strength is a good recommendation for use in supercritical boilers. Test specimens were sampled from pipe pieces of  $\phi$  31,8 x 6,3 mm.

#### **TESTING METHODOLOGY**

The microstructure investigations were carried out with an Inspect F scanning electron microscope (SEM) on the conventionally prepared electrolytically etched metallographic microsections. The analysis of precipitation processes was carried out by X-ray analysis of carbide isolates with Philips diffractometer.

Hardness measurements were taken by Vickers method with Future – Tech FM – 7 hardness testing machine at the indenter load of 10~kG.

The quantitative analysis of precipitates was carried out with NIKON EPIPHOT200 & LUCIA G v.5.03 image analysis system. The image analysis system was calibrated with scale marker in the photos. Calibration coefficient: 1 pixel =  $0.040 \mu m$ .

The above-mentioned tests were performed on material in the as-received condition and after long-term ageing at 700 and 750  $^{\circ}$ C for 1 000 and 5 000 h.

#### **RESULTS AND DISCUSSION**

The microstructure of Inconel 740H alloy in the asreceived condition (after solution heat treatment) observed by light and scanning electron microscopy is shown in Figure 1. The test alloy was characterised by the austenitic microstructure with numerous annealing twins and relatively thick grain with the average grain diameter of approx.  $100 \, \mu m$  (similar to the size as specified in the ASTM cards).

15.00 kV ETD 10.1 mm] 250 x a)

The X-ray phase composition analysis of the precipitates showed the presence of M(C,N) niobium and titanium carbides and nitrides,  $Cr_{23}C_6$  carbides and  $\gamma'$  phase in the as-received condition of the test alloy (Table 2).

Table 2 Change of the phase composition of the Inconel
740H precipitates depending on the annealing
time

Material state	Phase composition				
as-received condition	M(C,N), Cr <sub>23</sub> C <sub>6</sub> γ' phase				
ageing 700°C / 1 000 h	main Cr <sub>23</sub> C <sub>6</sub> min. γ' phase				
ageing 750 °C / 1 000 h	min. $\operatorname{Cr}_{23}C_6$ main $\gamma'$ phase				

The ageing of Inconel 740H alloy in the temperature range of 700 - 750 °C for 1000 h affects the development of the precipitation process and change in morphology coherent with the matrix of  $\gamma'$  phase and  $M_{23}C_6$  carbides. The examples of Inconel 740H microstructures observed after ageing are shown in Figures 2 and 3. As the temperature increased, the growth of  $Cr_{23}C_6$  carbide concentration was observed at the austenite grain boundaries and twins with evenly distributed regular  $\gamma'$ -phase precipitates inside the austenite grains and in the immediate vicinity of carbides at the grain boundaries.

The X-ray phase composition analysis of the precipitates in Inconel 740H alloy revealed that following ageing at 700 °C for 1 000 h the main phase was  $Cr_{23}C_6$  carbide and, to a lesser degree,  $\gamma'$  phase (Table 2). Raising the ageing temperature up to 750 °C significantly intensifies the precipitation process of  $\gamma'$  phase, which shows the highest percentage after a period of 1 000 h (Table 2).

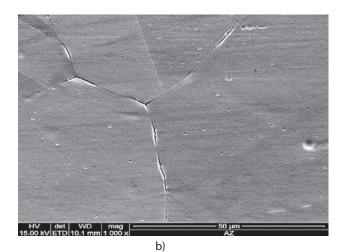
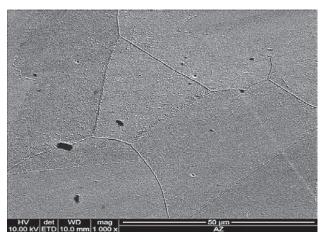
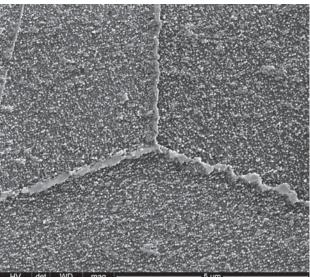


Figure 1 Microstructure of Inconel 740H alloy in the as-received condition; a) LM, b) SEM. Hardness 205 HV10

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**Figure 2** Microstructure of Inconel 740H alloy after ageing at 700 °C / 1 000 h; SEM. Hardness 278 HV10

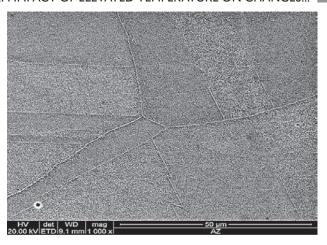
The confirmation of that is the quantitative analysis of  $\gamma'$  phase precipitates, which showed that the average diameter of this phase after ageing at 700 and 750 °C for 1 000 h was 29 and 76 nm, respectively.

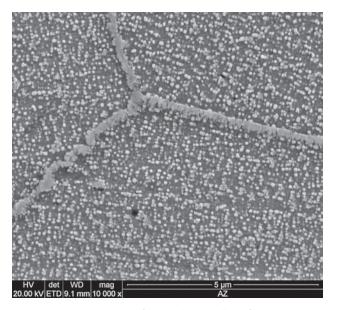
The long-term ageing process in the range of the expected long-term service temperature of 700 - 750 °C contributes to the decrease in impact energy and increase in strength properties due to the precipitation of secondary phases ( $\gamma'$ ,  $\text{Cr}_{23}\text{C}_6$ ).

## **CONCLUSIONS**

The Inconel 740H alloy in the as-received condition is characterised by the austenitic microstructure with numerous annealing twins and the grain with the average diameter of about 100  $\mu$ m. The X-ray phase composition analysis of the precipitates showed the presence of M(C,N) niobium and titanium carbides and nitrides,  $\text{Cr}_{23}\text{C}_6$  carbides and  $\gamma'$  phase in the as-received condition of the test alloy.

Long-term ageing at 700 - 750 °C contributes to the precipitation of  $\text{Cr}_{23}\text{C}_6$  carbide at the austenite grain boundaries and annealing twins and of coherent  $\gamma'$  phase





**Figure 3** Microstructure of Inconel 740H alloy after ageing at 750 °C / 1 000 h; SEM. Hardness 341 HV10

inside the austenite grains the average diameter of which increases with the temperature and ageing time.

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## **REFERENCES**

- [1] L.A. Dobrzański, M. Macek, B. Tomiczek, P. Nuckowski, A.J. Nowak The influence of the dispersion method on the microstructure and properties of MWCNTs/AlMg1SiCu nanocomposites, Archives of Metallurgy and Materials 61 (2016) 2, 1229-1234
- A. Drygała, L.A. Dobrzański, M. Szindler, M.M. Szindler, M. Prokopiuk vel Prokopowicz, E. Jonda, International Journal of Hydrogen Energy 41 (2016) 18, 7563-7567

- [3] Iseda, H. Okada, H. Semba, M. Igarashi, Long term creep properties and microstructure of SUPER304H, TP347HFG and HR3C for A-USC boilers, Energy Materials 2 (2007), 199-206
- [4] L. Żukowska, A. Śliwa, J. Mikula, M. Bonek, W. Kwaśny, M. Sroka, D. Pakułą: Finite Element prediction for the internal stresses of (Ti,Al)N coatings, Archives of Metallurgy and Materials 61 (2016) 1, 149-152
- [5] Ch. Chi, H. Yu, X. Xie, Advanced Austenitic Heat-Resistant Steels for Ultra-Super-Critical (USC) Fossil Power Plants, Alloy steel properties and use, (edited by E. V. Morales) InTech Publ. 2011 171-201
- [6] A. Zieliński, M. Sroka, M. Miczka, A. Śliwa: Forecasting the particle diameter size distribution in P92 (X10CRWMOVNB9-2) steel after long-term ageing at 600 and 650 °C, Archives of Metallurgy and Materials 61 (2016) 2, 753-760
- [7] L.A. Dobrzański, B. Tomiczek, M. Pawlyta, M. Król, Aluminium AlMg1SiCu matrix composite materials reinfor-

- ced with halloysite particles, Archives of Metallurgy and Materials 59 (2014) 1, 335-338
- [8] M.U. Uysal, M. Kremzer: Buckling Behaviour of Short Cylindrical Functionally Gradient Polymeric Materials, Acta Physica Polonica A, 127 (2014), 1355-1357
- [9] T. Dudziak, V. Deodeshmukh, L. Backert, N. Sobczak, M. Witkowska, W. Ratuszek, G. Bruzda: Phase Investigations Under Steam Oxidation Process at 800 °C for 1000 h of Advanced Steels and Ni-Based Alloys. Oxidation of Metals (2016), 1-20
- [10] B. Tomiczek, L.A. Dobrzański, M. Macek, Effect of milling time on microstructure and properties of AA6061/ MWCNTs composites, Archives of Metallurgy and Materials 60 (2015) 4, 3029-3034

Note: The responsible translator for English language is Arkadiusz Mączyński Gliwice, Poland