The Influence of the Thermal Transient Regime on Grain Size at Nickel–based Alloys

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The paper shows the influence of the thermal transient treatment on the grain sizes of nickel-based alloys: Incoloy 800 HT, Inconel 617, Inconel 230 and Inconel 718. These alloys present interest in nuclear technology. Five different treatments of thermal transient regime were performed at 1200°C for each one of the alloys: the first was only heated and cooled without maintaining the alloy on a temperature plateau and the other four were maintained different periods of time on the plateau for 1, 2, 3 and 4 h. The characterization of the samples was realised on the longitudinal and transversal sections. As received, the greatest grain size was for Incoloy 800 HT, followed by Inconel 230, 617 and 718. After the thermal transient without maintaining at 1200°C, the grain sizes of all alloys were smaller compared to as received alloys. For the thermal transient treatment with a plateau at 1200°C for 1, 2, 3 and 4 h, the only alloy that increases its grain size was Inconel 718, whereas all the other decreased their values compared to as received alloy.

Keywords: nickel alloys, Incoloy, Inconel, thermal transient, grain size, hardness

Generation IV reactors are promising advanced nuclear systems due to their high thermal efficiency and plant simplification. One of the major issues is to propose performance materials for the fuel claddings and the core components. Because the materials will be used under high pressure and temperatures, the requirements for cladding materials are: good mechanical properties, corrosion resistance, resistance to neutron irradiation, low susceptibility to stress corrosion cracking and micro structural stability [1]. Also, they must have high temperature microstructural stability. To utilize their maximum potential, precipitation strengtheners, superalloys are used in the turbine at intermediate temperature where phase transformations and grain growth kinetics are rather sluggish [2]. As candidate alloy systems that could be considered for high temperature application, nickel-based alloys were selected as promising material based on experimental results [3]. Although thermal stability of Inconel 718 is extensively characterized [4–7], the literature lacks comparative stability data on other Ni-based superalloys. Several elements are added in small concentrations for control of grain size and of mechanical properties that have a strong influence on the grain boundaries. For these alloys, the literature shows the influence of various elements on the alloys microstructure. The carbon present in small quantities may be trapped by the carbide-forming elements Ti and Nb in the form of carbides MC - (Ti, Nb) C. These carbides mainly precipitate at the grain boundaries of the matrix γ, and they help to improve the resistance to intergranular creep. The nominal carbon content must be low, otherwise it reduces the levels of Ti and Nb, necessary for the formation of phases γ' and γ". Minor additions of C tend to result in the formation of carbides, often located at the grain boundaries. Nickel gives the material its good resistance to hot oxidation. Although face centred cubic (FCC) nickel is the major alloy constituent, Inconel 617, Inconel 230 and Inconel 718 contain a combination of five to ten other elements, below 40 %wt content. Chromium provides, in addition, the resistance to oxidation. Molybdenum increases the mechanical strength of the matrix (solid solution hardening of austenitic structure). Niobium is involved in the formation of hardening precipitates γ", therefore the hardness and strength increase almost linear by the concentration of niobium. Titanium and aluminium lead to the formation of precipitates γ whose role is also hardening, but with a lesser degree than γ" precipitation. These elements are important for control of damage accumulation at grain boundaries in service [8]. Carbon exhibits a high affinity for elements such as Ti, Nb, Mo, V and Cr and tends to form primary carbides directly from the liquid during solidification of Ni-based superalloys. Because these carbides occupy a significant volume along the grain boundaries and are often interconnected, particularly if they are formed with scrip morphology, potential degradation of the mechanical properties may occur. Although carbides may serve as crack-initiation sites during fatigue (in the absence of other more major types of defects) [9], the presence of discrete carbides at the grain boundaries inhibits sliding and damage accumulation during high-temperature creep [8]. Niobium substitutes for aluminium in γ phase (Ni,Al) as titanium does. Niobium also forms γ phase (Ni,Nb), the body centred tetragonal strengthened phase, in Inconel 718 [10]. In broad terms, the elements additions in nickel based alloys can be categorized as being: a) γ formers (elements that tend to partition to the γ matrix), b) γ formers (elements that partition to the γ precipitate); c) carbide formers; and d) elements that segregate to the grain boundaries [11-13].

Experimental part

The studding alloys, Incoloy 800 HT (UNS N08811), Inconel 617 (UNS N 06617), Inconel 230 (UNS N 06230) and Inconel 718 (UNS 07718) are commercial alloys with a determinated chemical composition listed in table 1.

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The effective composition of the alloys has been determined by fluorescence analysis ARL ADVANTAX IntelliPower Series. Before use, the samples were ground down to a series of SiC papers of varying grit sizes until 4000 grit (using grinder machine BETA Grinder-Polisher, Buehler) and cleaned through ultrasonic waves in acetone.

The influence of thermal transient treatment has been analysed by measuring the microhardness and the oxide layer and grain size (using metallographic microscope OLYMPUS GX 71). All thermal treatments have been performed at 1200°C in flowing steam with Argon.

The difference between the five thermal transient regime experiments applied for each alloy was the maintaining time at 1200°C: 0, 1, 2, 3 and 4 h. The used argon has same impurities (<2ppm oxygen, <3ppm nitrogen and <3ppm water). The treatments have been done using a thermo analyser SETSYS EVOLUTION 24.

Results and discussions

The alloys, as received, have been characterized in transversal (TS) and longitudinal section (LS) by microhardness and grain size. Figure 1 shows the values of grain size and microhardness. It can be observed that the grain size of the alloys varied for LS at 11 to 47.81 μm and for TS at 18.9 to 69.7 μm. In both sections, Incoloy 800 HT has the highest grain size, followed by Inconel 230, Inconel 617 and Inconel 718.

The mass variation in function of the maintaining time at 1200°C is represented in figure 2. It can be observed that at the beginning of the treatment an apparent growing of the mass, due to the Archimede force generated by the argon gas dilated, is recorded, at the end of the tests a decrease of the mass has been observed due to the inert gas compression. The mass variations of the samples after the treatment have different values table 2.

In function of the isothermal maintaining times we observe:
- an expansion of the quasi-linear austenitic matrix γ between 150-300°C;
- a slight contraction between 150-300°C, bound at the end of the precipitation of phase γ′ Ni₃(Ti, Al) structure CFC;
- in the temperature range 200-400°C a first expansion phenomenon occurs representing the beginning of the dissolution of precipitates γ′;
- in the temperature range 1200-1400°C a second phenomenon was the dissolution of phase γ′′;
- from 1200 to 1400°C a contraction began: this represents the dissolution of phase β

The oxygen impurity in the argon gas, determines the oxide layer formation at the surface of the samples. It is known that the corrosion resistance depends on the types of oxide layer. The presence of oxide layers is illustrated by figure 3. A slight difference between the four alloys could

<table>
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<th>Alloy</th>
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<th>Ni</th>
<th>Cr</th>
<th>Ti</th>
<th>Mo</th>
<th>Co</th>
<th>Nb</th>
<th>W</th>
<th>Al</th>
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<th>V</th>
<th>P</th>
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<td>31.22</td>
<td>20.06</td>
<td>0.52</td>
<td>0.61</td>
<td>0.51</td>
<td>0.34</td>
<td>0.077</td>
<td>0.41</td>
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<td>20.05</td>
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<td>0.348</td>
<td>0.508</td>
<td>0.0211</td>
<td>0.597</td>
<td>0.456</td>
<td>0.08</td>
<td>0.718</td>
<td>0.428</td>
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<td>52.98</td>
<td>22.99</td>
<td>0.22</td>
<td>9.70</td>
<td>11.19</td>
<td>1.17</td>
<td>0.06</td>
<td>0.09</td>
<td>0.07</td>
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Table 1
CHEMICAL COMPOSITION OF STUDIED ALLOYS IN wt%

Table 2
MASS VARIATIONS OF THE SAMPLES AFTER THE TREATMENT
be observed. The oxide formed on Inconel 617 and Inconel 230 is riched in Cr$_2$O$_3$ [18-20]. The oxide layer formed on Incoloy 800 HT was found to be compact and adherent. The oxide layer on Inconel 718 samples is mainly formed of a rhombohedral phase Cr$_{2-x}$M$_x$O$_3$ which has a high content in Cr [21]. The modification of the grain size and the microhardness appear after the thermal treatment and show also a different behavior. After the thermal transient treatment, a little difference appears between the values of grain size on the two sections TS and LS (fig. 4).

![Fig. 2. Mass variation in function of the maintaining time at 1200°C for 1 h. The numbers represents: 1 - Incoloy 800 HT; 2 - Inconel 617; 3- Inconel 230; 4 - Inconel 718. Also the same behaviour of the mass variation appers for the samples without maintaining: 2 h, 3 h, 4 h on a plateau at 1200°C](image)

![Fig. 3. Oxide layers formed in argon (<5 ppm O$_2$) at 1200°C, during 4 h: a) Incoloy 800 HT, b) Inconel 617, c) Inconel 230, d) Inconel 718](image)

![Fig. 4. Grain size (a) and microhardness (b) obtained after thermal transient regime for Incoloy 800 HT](image)

![Fig. 5. Grain size (a) and microhardness (b) obtained after thermal transient regime for Inconel 617](image)
For the sample without maintaining at 1200°C, the grain size in LS has grown during the isothermal maintaining times up to 3 h and decreased at 4 h. The microhardness has the lowest value for the samples with 2 and 3 h plateau. Figure 5 (a) presents the values of the grain sizes and figure 5 (b) the microhardness of the Inconel 617 in function of the time when the alloy was maintained on the temperature plateau.

The values of the grain size in LS section are smaller than in TS section. The lowest grain size in longitudinal and transversal section has been determined for the samples without maintaining the alloy at 1200°C. The smallest value of the microhardness appeared for the sample which was only heated up to 1200°C, without plateau. In figure 6 are presented the values of the grain sizes and the microhardness, in function of the maintaining times for Inconel 230.

Inconel 230 has the smallest grain size values for both sections comparing with the samples from all alloys. The biggest value of the microhardness Vickers was measured for the samples which were maintained 1 hour on the plateau. In figure 7 are presented the values of the grain sizes (a) and the microhardness (b) of the Inconel 718 in function of the isothermal maintaining time.

The difference that appears between the TS and LS sections is that on TS the grain sizes are bigger than on the LS. It has been observed that for the samples maintained 1 and 2 h the microhardness is the same. Analysing the values of grain size after the thermal transient without isothermal maintaining at 1200°C it was found that the grain sizes of all alloys decrease compared with the grain size of as-receive alloy. The only alloy that increases its grain size after maintaining on a plateau at 1200°C for 1, 2, 3 and 4 h is Inconel 718; all the other alloys decrease their values. These changes of the grain sizes and the microhardness appear because of the transformations that took place during the thermal transient regime. The microstructure of Incoloy 800 HT consists of an austenitic matrix with precipitations of titanium carbide and titanium nitride (or carbonitride) along the grain boundaries and in the matrix [22, 23]. During thermal treatment, some metallurgical reactions such as formation of secondary carbides and phase transformation of titanium carbide into Cr,C, + Ni, Ti, Si appear. Inconel 617 is a solid solution additionally strengthened by intergranular M(C, N) carbonitrides and homogeneously distributed M₂₃C₆ carbides, which tend to grow along the grain to form carbide film at the grain boundaries at high temperature [24]. The main phases precipitated from the solid solution of Inconel 230 are all carbides.

The structure of Inconel 718 consists in a γ matrix, intergranular β precipitates and (Nb, Ti)C carbides; moreover, γ and γ' phases have precipitated in the matrix.
Conclusions

The influence of thermal transience treatment at 1200°C on the alloys with high nickel content revealed specific modifications of the microstructure composition, average grain size and microhardness. These modifications are the result of the coalescence effect on grain size leading to the precipitations apparition inside and at grain boundary.

To conclude on the mechanical properties, more tests are necessary to be carried out. The temperature influence on the mechanical and microstructural characteristics is very significant. Continued improvements in the properties of these materials can be possible through close control of chemistry and microstructure and the introduction of advanced processing approaches.

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References

8. *** ASTM B409 ISS 96A UNS N08811
9. *** SAEMS 5889B; ASME SB 168 UNS N 06617
10. *** ASME SB435; AMS878B UNS NO6230
11. *** ASTM B 670 ISS 02 UNS N0718

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