THERMAL CYCLING INFLUENCE ON MICROSTRUCTURAL CHARACTERIZATION OF ALLOYS WITH HIGH NICKEL CONTENT

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ABSTRACT

The IV nuclear energy generation systems are aimed at making revolutionary improvements in economics, safety and reliability, and sustainability. To achieve these goals, Generation IV systems will operate at higher temperatures and in higher radiation fields. This paper shows the thermal cycling influences on microstructure and hardness of nickel based alloys: Incoloy 800 HT and Inconel 617. These alloys were meekly at a thermal cycling of 25, 50, 75 and 100 cycles. The temperature range of a cycle was between 400°C and 700°C. Nickel base alloys develop their properties by solid solution and/or precipitation strengthening.

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

Introduction

Six most promising systems identified for next-generation nuclear energy are chosen to be Generation IV.

Two employ a thermal neutron spectrum with coolants and temperatures that enable hydrogen or electricity production with high efficiency (Supercritical Water Reactor—SCWR and Very High Temperature Reactor—VHTR). Three employ a fast neutron spectrum to enable more effective management of actinides through recycling of most components in discharged fuel (Gas-cooled Fast Reactor—GFR, Lead-cooled Fast Reactor—LFR, and Sodium-cooled Fast Reactor—SFR). The Molten Salt Reactor (MSR) employs a circulating liquid fuel mixture that offers considerable flexibility for recycling actinides and may provide an alternative to accelerator-driven systems.

This paper shows opportunities for studying new materials used in Generation IV reactors. The focus of this study has been on microstructural characterization of alloys with high nickel content: Incoloy 800 HT and Inconel 617 after they have been subjected to different thermal cycling treatments with various cycles. In this study, the samples which have been obtained after thermal cycling treatments were analyzed by microstructures, microhardness and type of precipitates and were compared with the microstructures in as-received conditions.

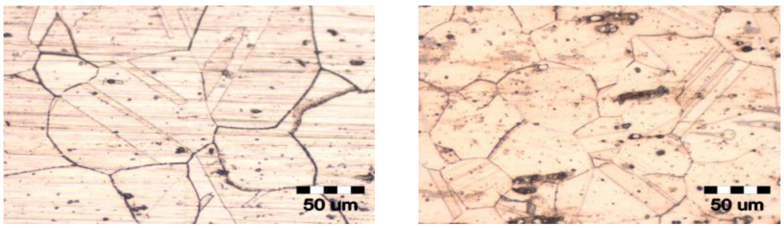
Experimental part

The studding alloys, Incoloy 800 HT (UNS N08811) and Inconel 617 (UNS N 06617), are commercial alloys with a determinate chemical composition listed in [1]. The investigations were conducted on samples of 15x30x2mm size samples.

The parameters which were choosing for cycling treatment were: heating speed 5°C/min, cooling speed 5°C/min, the maximum temperature of a cycle T_{max}= 700°C, the minimum temperature of a cycle T_{min}= 400°C. Before use, the samples were ground down to a series of SiC papers of varying grit sizes until 4000 grit paper (grinder machine BETA Grinder-Polisher, Buehler) and cleaned through ultrasonic waves in acetone. The influence of thermal cycling treatment has been analyzed by microhardness tests and grain size (metallographic microscope OLYMPUS GX 71). The thermal treatment has been realized using a Guss oven.

Results and comments

Optical microscopic analysis and grain size of as received materials.
Alloys present austenitic macles grain structures and precipitates distributed at grain boundaries and in matrix (**Figure 1**).



a) b)
Figure 1 *Alloy microstructure of as received x300:*
a) Incoloy 800HT b) Inconel 617

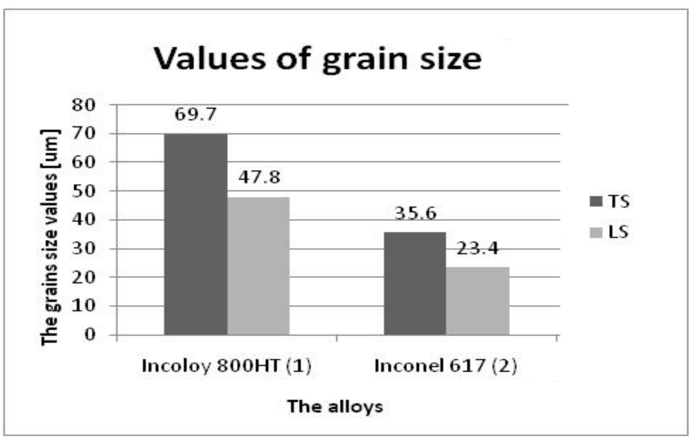


Figure 2 *Values of grain size - As received*

Figure 2 presents the values of grain sizes of as received materials. It can be seen that in longitudinal section (LS) grain size varies from 23.4 to 47.81 μm and in cross section (TS) between 69.7 and 35.6 μm. In both sections, Incoloy 800HT has the largest grain size, followed by Inconel 617 [1].

Microhardness of as-received alloys with high nickel content

Vickers microhardness (**Figure 3**) was determined after an electrolytic attack of as received conditions with Microhardness OPL.

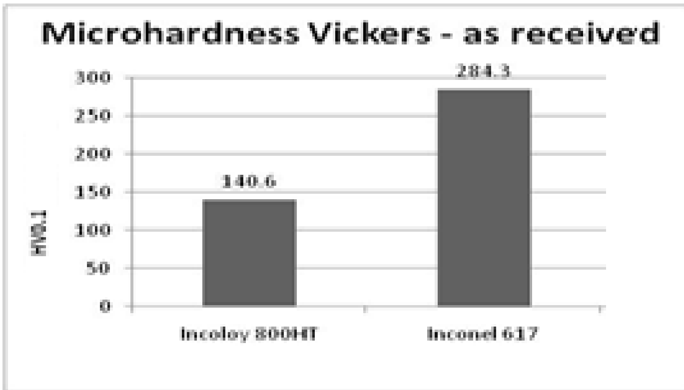
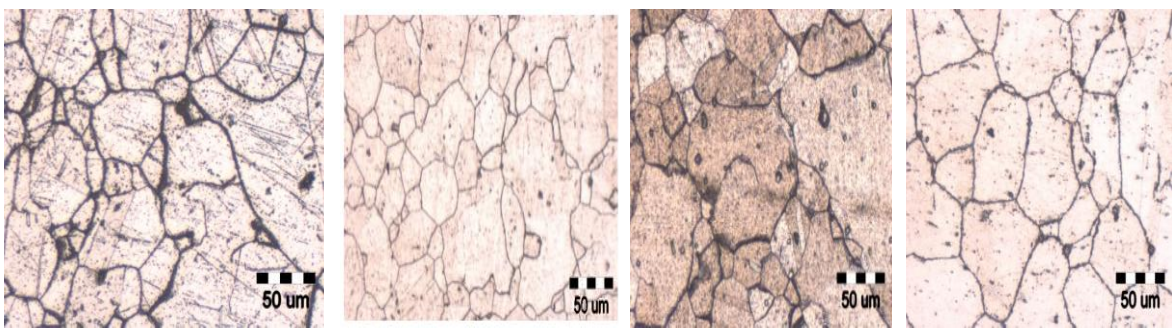


Figure 3 *Vickers microhardness of as received alloys with high nickel content*

As received Inconel 617 has the highest microhardness and Incoloy 800HT has the largest grain size and the lowest microhardness.

Incoloy 800HT microstructure

Incoloy 800HT is a nickel and iron based alloy with austenitic matrix and precipitates located at grain boundaries and in the matrix. **Figure 4** presents Incoloy 800HT microstructures obtained after thermal cycling.



a) b) c) d)

Figure 4 *Incoloy 800 HT microstructure evolution in function of number of cycles: a) 25 (x300), b) 50 (x300), c) 75 (x300) and d) 100 (x300) cycles*

By image analysis, of as received alloy was determined 36% $Fe_{0,65}Ni_{0,35}$ and after 100 cycles this proportion increases to 72%. In Cr carbide ($Cr_{23}C_6$), thermal cycling caused the replacement of about 7.4% Cr with Fe and formed $Cr_{15,64}Fe_{7,36}C_6$. Mixes of compounds crystallize in cubic system of as received and after 100 cycles, but with completely different network parameters.
Changing elemental composition of the identified compounds is due to a dynamic redistribution of chromium and the appearance of $Cr_{15,64}Fe_{7,36}C_6$. Structural changes lead to modifications in compounds proportions with large elementary cell volume and small volume, with an increase from 36% to 72% of the small cell volume compounds (**Table 1**).

Table 1 *Incoloy 800HT compounds*

Thermal treatments	Elements	Chemical formula	Estimated percentage [%]	Crystallization system	Matrix parameters (a, b, c)[Å]	Elemental cell volume [$10^{-6}pm^{-3}$]	Reference
As received	Fe, Ni	$Fe_{0,65}Ni_{0,35}$	36	cubic	3.5730	45.61	[2]
	Cr,C	$Cr_{23}C_6$	64	cubic	10.9030	1296.10	[3]
Thermal cycling 100 cycles	Fe, Ni	$Fe_{0,65}Ni_{0,35}$	72	cubic	3.5730	45.61	[2]
	Cr, Fe, C	$Cr_{15,64}Fe_{7,36}C_6$	28	cubic	10.5966	1189.87	[4]

Incoloy 800HT grain size variation

Figure 5 presents grain sizes variations versus cycles for Incoloy 800HT.

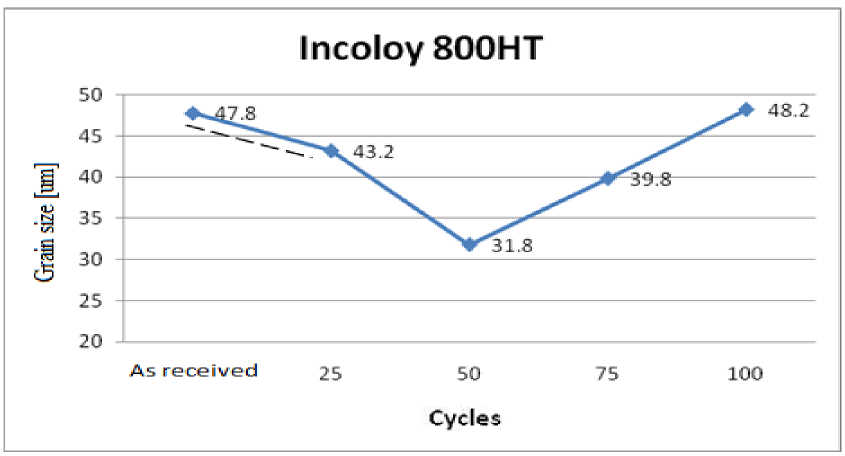


Figure 5 *Incoloy 800 HT grain size of as received condition and after 25, 50, 75 and 100 cycles*

After 25 cycles, grain size decreases slightly. Doubling the number of cycles the grain size decreases but if the number of cycles increases an increase almost constant of grain size can be observed.

Incoloy 800HT microhardness

Figure 6 presents microhardness variations versus number of cycles for Incoloy 800HT.
Microhardness after 25, 50 and 100 cycles present values that are above the values obtained of as received alloy. For 75 cycles appears a microhardness decrease. Between 25 -50 cycles the hardness decrease and is correlated with the variation of grain size (**Figure 5, 6**). Hardness

increase to more than 75 cycles may be associated with an increased proportion of hard precipitates in alloy microstructure.

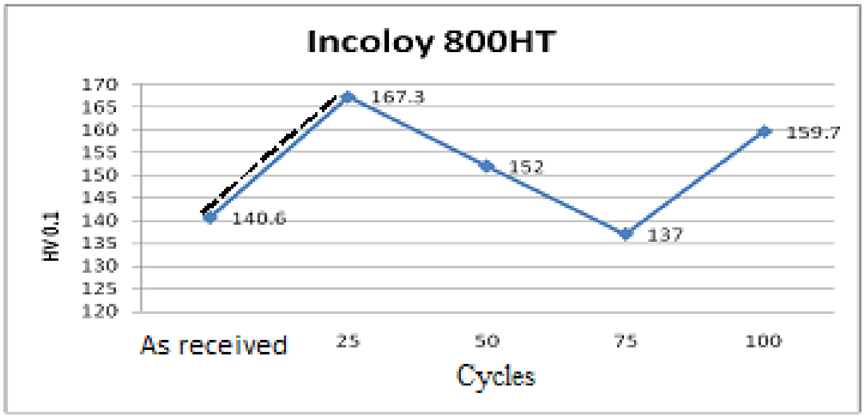


Figure 6 *Incoloy 800HT microhardness of as received and after thermal cycling*

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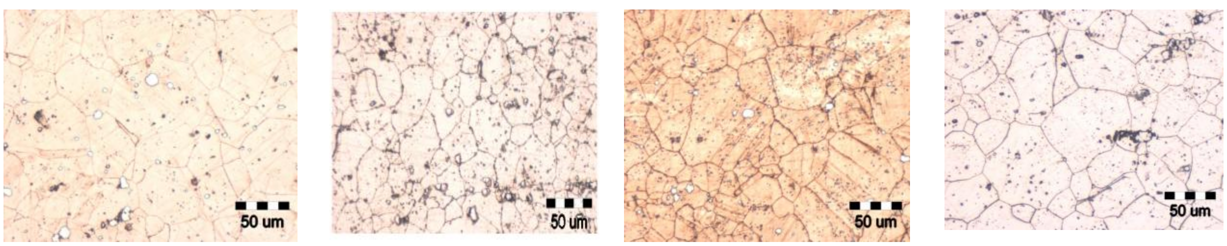
nconel 617 microstructure

Inconel 617 presents an austenitic matrix with fine carbide present in matrix and at grain boundaries. **Figure 7** presents Inconel 617 microstructures obtained after thermal cycling.



a) b) c) d)

Figure 7 *Inconel 617 microstructure evolution in function of the number of cycles: a) 25 (x300), b) 50 (x300), c) 75 (x300) and d) 100 (x300) cycles*

By X diffraction performed on Inconel 617 it can be observed both of as received and after 100 cycles a single type of compound $Cr_{0,5}Ni_{0,5}$.

Table 2 *Inconel 617 compounds*

Thermal treatments	Elements	Chemical formula	Estimated percentage [%]	Crystallization system	Matrix parameters (a, b, c)[Å]	Elemental cell volume [$10^{-6}pm^{-3}$]	Reference
As receive	Cr, Ni	$Cr_{0,5}Ni_{0,5}$	100	cubic	3,5910	46.31	[5]
Thermal cycling 100 cycles	Cr, Ni	$Cr_{0,5}Ni_{0,5}$	100	cubic	3,5910	46.31	[5]

Inconel 617 grain size variation

Figure 8 presents grain size variations versus number of cycles for Inconel 617.

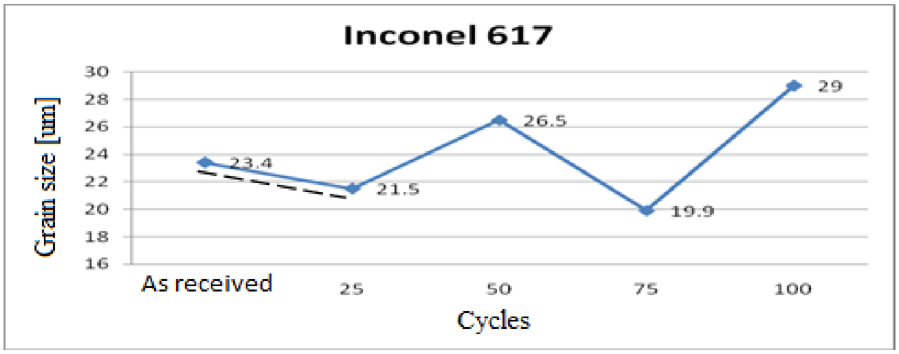


Figure 8 *Grain size for Inconel 617 of as received condition and after 25, 50, 75 and 100 cycles*

Inconel 617 grain size presents a minimum at 25 and 75 cycles and a maximum after 100 cycles.

Inconel 617 microhardness

Figure 9 presents microhardness variations versus number of cycles for Inconel 617.

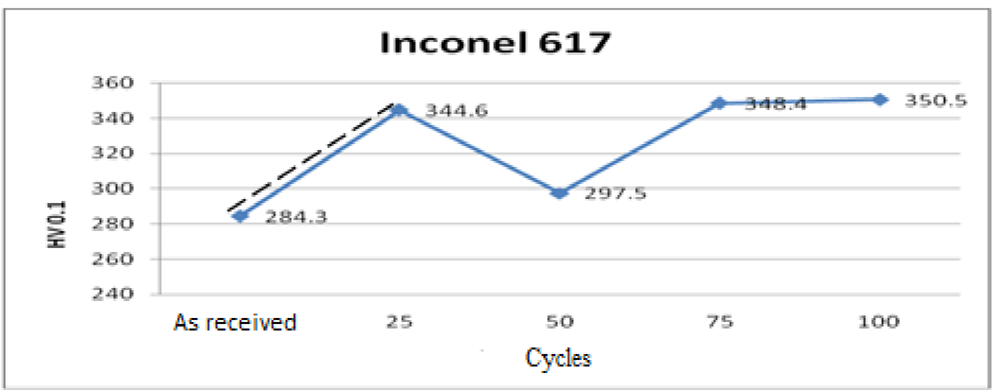


Figure 9 *Inconel 617 microhardness of as received alloys and after thermal cycling*

Between 25-50 cycles, microhardness decrease with the increasing of grain size. It could be observed that microhardness values can be correlated with grain size only for a small number of cycles (50 cycles). For treatments with large number of cycles, the hardness values may be associated with precipitating phases.

Conclusions

Taking into consideration the chemical composition of alloys with high nickel content, a first set of conclusions can be drawn by groups of alloys.

Incoloy 800HT

- A correlation cannot be made between grain size and microhardness variation, because during thermal cycling, significant changes occur in precipitated compounds.

Inconel 617

- For as received material, the austenitic grain size of Inconel 617 could be correlated with the determined values obtained for microhardness.
- Inconel 617 microhardness remains constant for 75 to 100 of cycles.

A general review of the studied alloys on microhardness variation versus number of thermal cycles is presented in **Table 3.**

Table 3 *Alloys microstructures according to the number of cycles*

Alloy	**Microhardness HV**				
	As received	**25 cycles**	**50 cycles**	**75 cycles**	**100 cycles**
Incoloy 800HT	140.6	167.3	152	137	159.7
Inconel 617	284.3	334.6	297.5	348.4	350.5

- Alloys show an increase in hardness for 25 cycles; this phenomenon can be correlated with the decrease of grain size compared with as received.
- At 100 cycles, all alloys lead to higher hardness values than as received ones.
- The spectacular increase of microhardness is performed for Inconel 617 followed in descending order by Incoloy 800HT.

As a conclusion, our experimental data have shown that during thermal cycling the studied alloys with high nickel content (Incoloy, Inconel) experience molecular reorganization with a various modification of grain size and microhardness.

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