E14-2013-111

I. Ionita [1], M. Balasoiu [2,3,*], D. Soloviev [2],
A. M. Balasoiu-Gaina [2], M. Fulger [1]

SMALL-ANGLE NEUTRON SCATTERING INVESTIGATION OF HEAT-TREATED INCOLOY 800HT SAMPLES: PRELIMINARY RESULTS

[1] Institute of Nuclear Research, Pitesti, Romania
[2] Joint Institute for Nuclear Research, Dubna, Russia
[3] Horia Hulubei National Institute of Physics and Nuclear Engineering, Bucharest, Romania
[*] E-mail: balas@jinr.ru; balasoiumaria@yahoo.com

Ионита И. и др. E14-2013-111

Исследование методом малоуглового рассеяния
нейтронов образцов INCOLOY 800HT,
подвергнутых термической обработке. Предварительные результаты

Методом малоуглового рассеяния нейтронов были исследованы образцы INCOLOY 800HT, подвергнутые термической обработке в течение 60 сут при различных температурах отжига, для изучения их микроструктурной эволюции.

Работа выполнена в Лаборатории нейтронной физики им. И. М. Франка ОИЯИ.

Сообщение Объединенного института ядерных исследований. Дубна, 2013

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Small-Angle Neutron Scattering Investigation of Heat-Treated
INCOLOY 800HT Samples: Preliminary Results

INCOLOY 800HT samples subjected for 60 days to different annealing temperatures have been investigated by small-angle neutron scattering (SANS) with the aim to study their microstructural evolution.

The investigation has been performed at the Frank Laboratory of Neutron Physics, JINR.

Communication of the Joint Institute for Nuclear Research. Dubna, 2013

INTRODUCTION

The INCOLOY® 800 series of alloys are the result of years of monitoring and maintaining the ultimate chemical properties for high-temperature strength and resistance to oxidation, carburization and other types of high-temperature corrosion.

Each one a refinement of the one before, these alloys have set the industry standard in high-temperature applications requiring optimum creep and rupture properties.

INCOLOY nickel–iron–chromium alloy 800 was introduced to the steel market in the 1950s to fill the need for a heat- and corrosion-resistant alloy with relatively low nickel content since nickel was, at the time, designated a «strategic» metal. Over the past 60 years it has been widely used for its strength at high temperatures and its ability to resist oxidation, carburization, and other types of high-temperature corrosion. Applications include furnace components and equipment, petrochemical furnace cracker tubes, pigtails and headers, and sheathing for electrical heating elements.

INCOLOY 800, 800H, and 800HT nickel–iron–chromium alloys are identical except for the higher level of carbon in alloy 800H, and the addition of up to 1.2% aluminum and titanium in alloy 800HT. INCOLOY 800 was the first of these alloys and it was slightly modified into INCOLOY 800H. This modification was to control carbon (0.05–0.10%) and grain size to optimize stress rupture properties. INCOLOY 800HT has further modifications to the combined titanium and aluminum levels (0.85–1.20%) to ensure optimum high-temperature properties.

INCOLOY 800HT is a nickel-based alloy with excellent corrosion resistance in water at high temperatures. The good mechanical properties of INCOLOY 800, combined with its resistance to high-temperature corrosion, make this alloy exceptionally useful for many applications involving long-term exposure to elevated temperature and corrosive atmosphere. The 800HT alloy is widely used in various types of heat-treating furnaces, in power generation for steam generators tubing and high-temperature heat exchangers in gas-cooled nuclear reactors. Recently, this alloy has started to be studied as candidate material for fuel cladding in GEN IV reactors like supercritical water reactors (SCWR). In connection with this research, samples of INCOLOY 800HT subjected for 60 days to different annealing temperatures have been investigated by small-angle neutron scattering (SANS) with the aim to study precipitates phase's microstructural evolution. In the paper are presented preliminary results obtained at the IBR-2 reactor.

EXPERIMENTAL

SANS measurements were carried out using the YUMO [1, 2] instrument installed at the IBR-2 high pulsed reactor (JINR, Dubna, Russia), equipped with a two-detector system. Processing of the measured spectrum, calculation of the spectrometer resolution, carrying out the spectrum normalization were performed using SANS software package [3]. The coverage interval of scattering vector Q was $0.02\ \text{Å}^{-1} < Q < 0.4\ \text{Å}^{-1}$, where the magnitude $Q = \left|\vec{Q}\right| = \left|\vec{k} - \vec{k}_0\right|$, with $\vec{k}$ and $\vec{k}_0$ the wave vectors of incident and scattered neutrons, has the expression

$$Q = (4\pi/\lambda)\sin(\theta/2),$$

where λ is the incident neutron wavelength and θ is the scattering angle.

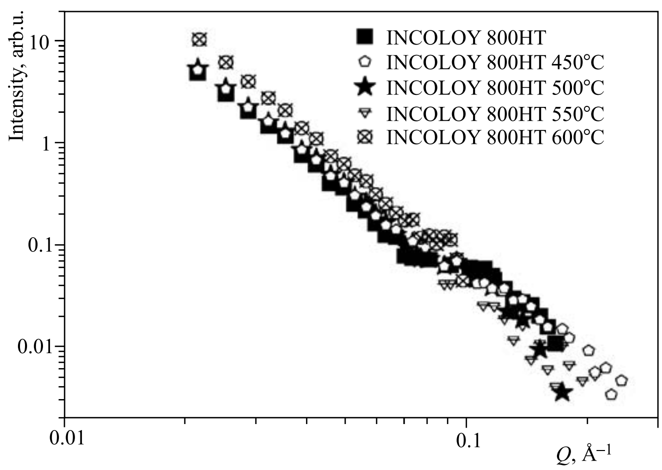

Fig. 1. SANS patterns from INCOLOY 800HT without heat treatment, with heat treatment (annealing time 60 days) at $450°\text{C}$, $500°\text{C}$, $550°\text{C}$, and $600°\text{C}$

In Figs. 1–6 are presented the SANS patterns from INCOLOY 800HT without heat treatment, with heat treatment (annealing time 60 days) at $450°C$, $500°C$, $550°C$, and $600°C$, and the fitting curves.

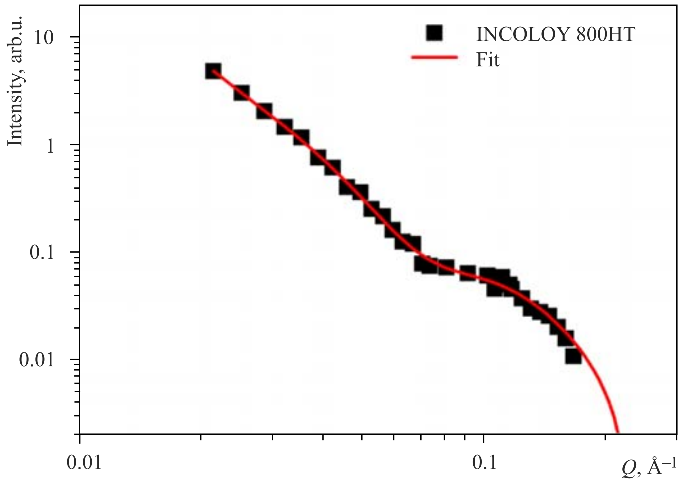

Fig. 2. SANS curve and fit for INCOLOY 800HT without heat treatment

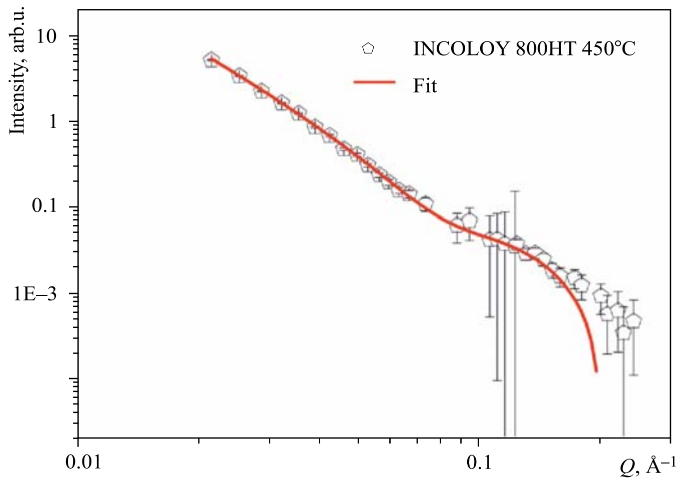

Fig. 3. SANS curve and fit for INCOLOY 800HT with heat treatment (annealing time 60 days) at $450°C$

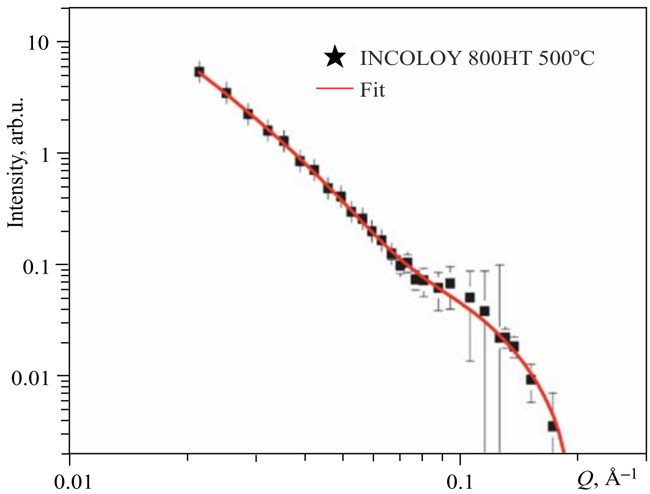

Fig. 4. SANS curve and fit for INCOLOY 800HT with heat treatment (annealing time 60 days) at 500°C

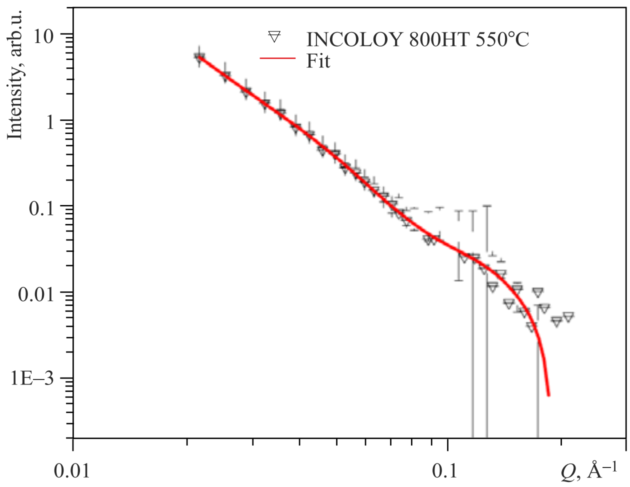

Fig. 5. SANS curve and fit for INCOLOY 800HT with heat treatment (annealing time 60 days) at 550°C

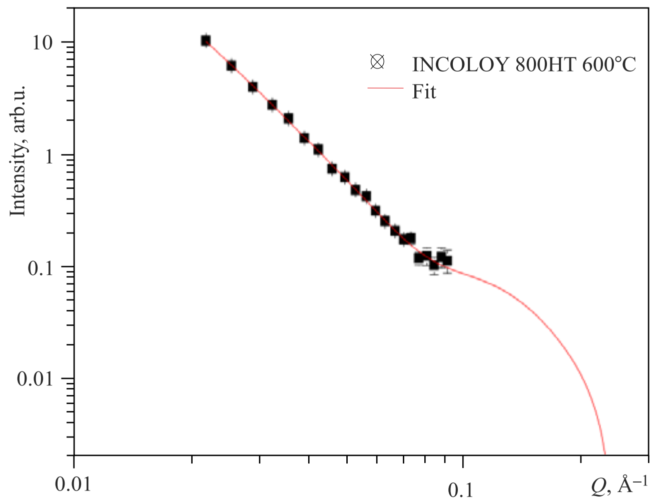

Fig. 6. SANS curve and fit for INCOLOY 800HT with heat treatment (annealing time 60 days) at 600°C

RESULTS AND DISCUSSION

For best fit the following expression, which combines 2 Guinier and one power-law dependences, was found:

$$I(Q) = I_1 \cdot \exp\left(\frac{Q^2 R_{g1}^2}{3}\right) + I_2 \cdot \exp\left(\frac{Q^2 R_{g2}^2}{3}\right) + A \cdot Q^{-\alpha} + B.$$

The 2 Guinier dependences point to the presence of two grain types or two phase type domains detected in the used SANS Q-range [4]. The radii of gyration of corresponding grains or domains are obtained from the best fit (see table and Fig. 7).

Fitting parameters R_{g1}, R_{g2}, α of SANS curves from INCOLOY 800HT without and with heat treatment

No.	Sample	Heat treatment, °C	R_{g1}, Å	R_{g2}, Å	Fractal dimension α
1	INCOLOY 800HT	—	29.9 ± 1.6	96.2 ± 6.9	2.45 ± 0.10
2	INCOLOY 800HT	450	26.4 ± 3.1	62.9 ± 4.9	2.58 ± 0.16
3	INCOLOY 800HT	500	31.2 ± 2.1	15.1 ± 2.8	2.48 ± 0.12
4	INCOLOY 800HT	550	27.08 ± 2.3	84.9 ± 9.9	2.89 ± 0.14
5	INCOLOY 800HT	600	60.4 ± 7.8	27.3 ± 2.4	2.75 ± 0.15

It looks like the first grain or phase sample compound type is a little susceptible to size changes with heat treatment excepting the 500°C and 600°C temperatures. The second grain or phase sample compound type suffers more drastic changes, especially also at the 500°C and 600°C temperatures.

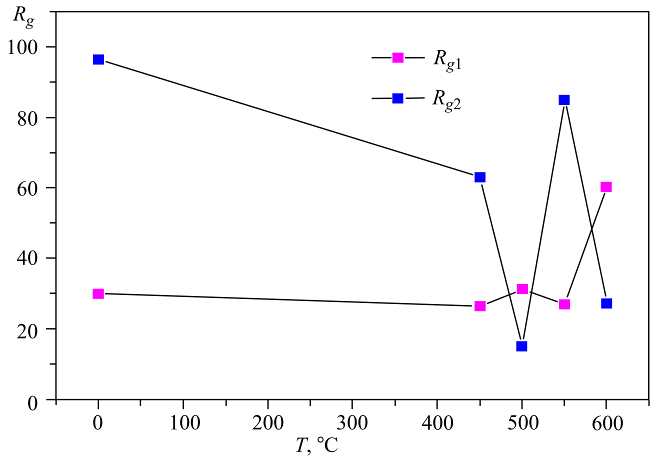

Fig. 7. R_{g1} and R_{g2} variation with heat-treatment temperature in INCOLOY 800HT samples

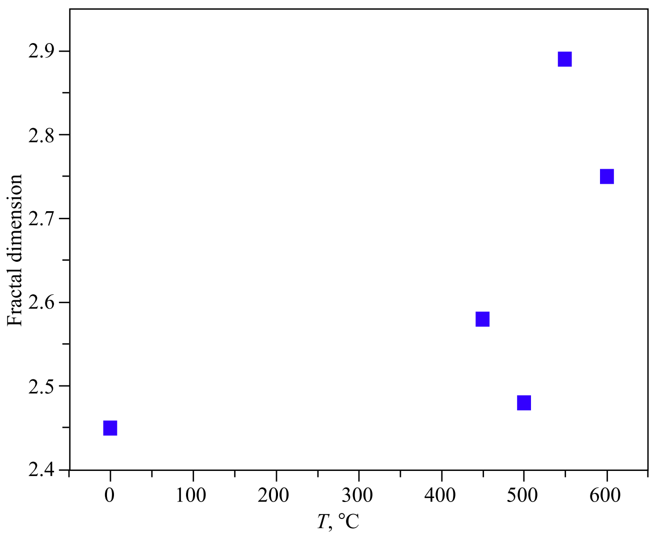

Fig. 8. Fractal dimension variation with heat-treatment temperature of INCOLOY 800HT alloy samples

The results in the case of heat treatment at $600°C$ can reflect the influence of an increased martensitic phase [5]. Further separate investigations of nuclear and magnetic scattering intensity of the samples are needed.

To understand the scattering curves and their evolution with progressive heat treatment (Fig. 1), we had also to consider the scattering from fractals. A scattering from mass fractals was detected. These mass fractals can characterize the interphase (interdomain) spaces. The fractal dimension variation with heat treatment shows the kinetics of interphase (interdomain) spaces transformation (Fig. 8).

For understanding the process, samples heated at lower and higher temperature values than in the present research should be investigated, and electron microscopy analysis is also required.

CONCLUSIONS

Preliminary information on the thermal treatment effects on nanoscale level has been obtained, in particular concerning precipitate size and fractal dimension variation. The results contribute to the microstructural analysis of the INCOLOY 800HT for industrial applicability in the considered sector.

Acknowledgements. The work was accomplished in the framework of the JINR–Romanian Cooperation Programme Project No. 81/18.02.2013 (item 15) and No. 82/18.02.2013 (item 2).

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Received on October 28, 2013.

Редактор *Е. И. Кравченко*

Подписано в печать 14.01.2014.
Формат 60 × 90/16. Бумага офсетная. Печать офсетная.
Усл. печ. л. 0,62. Уч.-изд. л. 0,84. Тираж 230 экз. Заказ № 58160.

Издательский отдел Объединенного института ядерных исследований
141980, г. Дубна, Московская обл., ул. Жолио-Кюри, 6.
E-mail: publish@jinr.ru
www.jinr.ru/publish/