Validation of Alternative Process to Simulate Irradiation Effect on Stainless Steel

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1. Introduction

Stainless steel is widely used as an internal structural material in nuclear power plants due to its good mechanical properties and corrosion resistance. However, in the primary water environments of pressurized water reactors, where stainless steel is exposed to radiation, the integrity of the internal structure may be compromised. Therefore, it is crucial to consider the effects of radiation to accurately assess the structural integrity.

The best way to investigate the effects of radiation damage on materials is through the use of neutron-irradiated materials. However, handling neutron radiation on a laboratory scale is almost impossible due to safety and cost issues. Therefore, there are several ways to simulate the effects of radiation on materials from a mechanical and chemical perspective.

There are several methods for simulating neutron irradiation. One approach is to use alternative irradiation sources such as protons, helium, and heavy ions. Another method is to use sensitizing heat treatment, while a third approach involves mechanical working. Although these alternative processes have some limitations, they can produce similar results to neutron irradiation.

This study involved conducting a sensitizing heat treatment to simulate the radiation-induced segregation effect along the grain boundaries, as well as a mechanical rolling process in a warm environment to create defects in the material and increase its hardness without generating martensite phase. To investigate the effects of this alternative process for simulating radiation damage, mechanical tests such as tensile and hardness tests were performed, and the material's microstructure was observed using scanning electron microscopy. The chemical composition was measured using energy-dispersive X-ray spectroscopy.

2. Experimental & Result

This section will provide basic information about the material used in this study, as well as explain the important criteria of this research, such as the target value of radiation damage and various approaches from both chemical and mechanical perspectives used to simulate the effect of radiation damage. Experimental validation is currently underway to determine whether the alternative process can simulate the effects of neutron irradiation.

2.1 Material

The mock-up sample used in this study is made of 304H stainless steel, which contains high carbon content. The figure below and the table show the 304H stainless steel block used and its chemical composition, respectively.

Fig. 1. 304H stainless steel mock-up sample with dimensions of 300 mm in length, 200 mm in width, and 22.2 mm in thickness

Table I: Chemical composition of 304H stainless steel mock-up sample used in this study

Fe	**C**	**Si**	**Mn**	**P**
Bal.	0.0542	0.429	1.069	0.0158
S	**Cr**	**Ni**	**Cu**	**Mo**
0.002	18.25	8.055	0.027	0.004
Ti	**Nb**	**Al**	**Co**	**N [ppm]**
0.0018	0.003	0.003	0.022	396

2.2 Target radiation damage level

The internal structure of nuclear power plants is exposed to radiation during operation. Previous research has shown that when materials are exposed to radiation damage up to 10 dpa, significant changes in grain boundary composition, strength, and ductility occur. Other studies have found that the radiation hardening effect reaches its maximum value at 5 dpa and saturates beyond that point [1]. Therefore, the target radiation damage level for this study was set at 5 dpa.

2.3 Sensitization heat treatment

To generate chromium depletion at the grain boundaries similar to that caused by neutron irradiation, sensitizing heat treatment is necessary. Chromium typically forms carbides at grain boundaries, which can lead to depletion of chromium in those areas. Therefore, to determine the appropriate heat treatment conditions for sensitization, thermodynamic calculations must be performed.

We used JMatPro v11.2 software to perform thermodynamic calculations [2], which revealed that the 304H stainless steel we used can exhibit five distinct phases at different temperatures. The following graph and list provide essential information about the phase properties of 304H stainless steel.

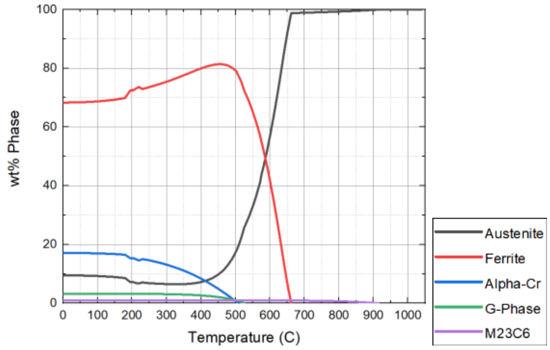

Fig. 2. Phase diagram of 304H stainless steel used in this study, calculated using JMatPro v11.2 software

1. 304H stainless steel can exist in the following phases: Austenite, Ferrite, Alpha-Cr, G-phase, and M23C6.
2. 304H stainless steel can only form carbides in the M23C6 phase.
3. The austenization temperature for 304H stainless steel is approximately 915 ℃.

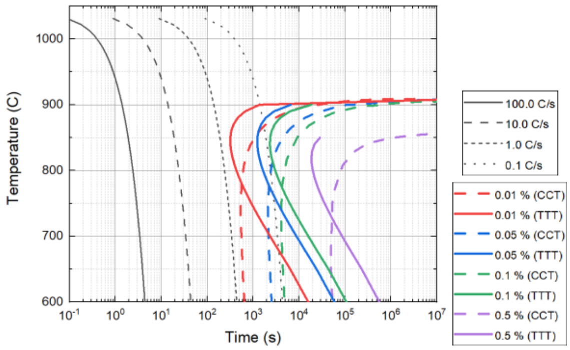

Fig. 3. Curve representing a combination of the continuous cooling transformation curve and the time-temperature transformation curve. The black and gray lines indicate cooling rates, while the solid-colored line represents the time-temperature transformation curve, and the dashed-colored line represents the continuous cooling transformation curve, both starting from the austenization temperature of 1,040 ℃. Each color represents the amount of carbide formation.

Before beginning the sensitization heat treatment, the material must undergo an austenization heat treatment. After this process, the material must be rapidly cooled to prevent the formation of carbides, which can be achieved by using a continuous cooling transformation curve. The material should then be held at a high temperature for a specific amount of time to allow carbide formation to reach the target value, as indicated by the time-temperature transformation curve. The graph above illustrates the combination of the continuous cooling transformation and time-temperature transformation curves, starting from the austenization temperature of 1,040 ℃ for the 304H stainless steel used in this study.

The heat treatment process was established by following the combined graph in Fig. 3. The material was rapidly cooled in air with a cooling rate of approximately 1.0 ℃/s until it reached a temperature of 800 ℃ from the austenization temperature 1,040 ℃. It was then held at this temperature for 20 hours to allow for carbide formation exceeding 0.5 wt%. Finally, the material underwent water quenching to prevent any unwanted phase transformation. The graph below provides a brief overview of the heat treatment process performed in this study.

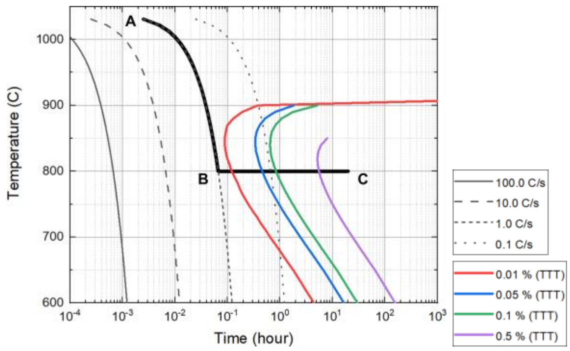

Fig. 4. Heat treatment process taking into account the austenization temperature, continuous cooling transformation curve, and time-temperature transformation curve. The process starts from point A, where the material underwent austenization, then proceeds to point B, where the temperature is held for sensitization, and finally reaches point C, indicating the final state of carbide formation.

2.4 Mechanical work in warm environment

Radiation can make a material harder by forming defects, such as dislocations. As a result, both yield stress and tensile stress increase when the material is irradiated. According to previous research, these mechanical properties eventually reach identical values once the radiation damage exceeds 5 dpa. Although there was some scatter in the data from previous research, the average value of yield stress for austenitic stainless steel with radiation damage of 5 dpa is approximately 850 MPa [1]. Therefore, the target yield stress for hardening by mechanical work is set at around 850 MPa.

There are several ways to mechanically deform a material, and one of them is rolling. Rolling can be divided into three categories based on temperature range: cold, warm, and hot rolling. Each category has its own advantages and disadvantages. Cold rolling is easier to perform than the other methods because it does not require heating the material before rolling. However, this process might form a martensite phase in the material, which can reduce its ductility and generate high residual stress. Hot rolling is a better choice for reducing the area without forming residual stress and martensite phase, but it might change the basic phase distribution of the material. Additionally, high temperatures during hot rolling might cause carbide formation.

In the case of warm rolling, the temperature range is intermediate between that of cold and hot rolling, which can effectively reduce residual stress and martensite phase transformation without changing the basic phase distribution [3]. Furthermore, the warm temperature is not enough to form carbides, making this process suitable for the purpose of this research. According to Fig. 2, the temperature range between 150 to 250 ℃ does not significantly affect the phase distribution of the material, and as noted in previous research, this temperature range is also sufficient to prevent martensite formation. In conclusion, warm rolling with the temperature range from 150 to 250 ℃ is adapted for this research.

As mentioned above, the target yield stress for mechanical hardening is approximately 850 MPa. To achieve this value, we will consider various reduction rates for the warm rolling process.

2.5 Specimen preparation

To investigate defects formation and chromium depletion at the grain boundary, a coupon specimen with dimensions of 20 mm in length, 10 mm in width, and 3 mm in thickness was cut from the warm-rolled block. Each sample was mechanically polished using emery paper with grit sizes of 400, 600, and 800, followed by diamond suspensions with particle sizes of 6, 3, and 1 μm, respectively.

To measure the mechanical properties such as yield stress and tensile stress, tensile specimens were cut from the warm-rolled block with dimensions following ASTM E8/E8M standards [4]. The tensile specimens did not undergo any surface treatment.

2.6 Test method

Basic microstructural observations will be conducted using a scanning electron microscope to determine the difference in grain boundary density, and detailed microstructural observations will be carried out using a transmission electron microscope to measure the defect density.

We will measure chemical compositional changes, such as chromium depletion caused by sensitization heat treatment, using energy dispersive X-ray spectroscopy. To compare differences between rare material and thermally treated material, we will also investigate bare 304H stainless steel.

Tensile tests will be conducted following the testing procedures suggested by ASTM E8/E8M standards [4]. The mechanical testing facility used for this study is the INSTRON 8801 model with an extensometer capable of measuring the full range of sample elongation.

3. Conclusion

The purpose of this study was to replicate the effects of neutron radiation on 304H stainless steel using alternative methods such as sensitization heat treatment and warm rolling. Sensitization heat treatment was used to induce chromium depletion at the grain boundary, while warm rolling was used to increase mechanical properties without generating residual stress and martensite phase as well. Specimens were prepared for both microstructural observation and mechanical tests. The microstructural observation was carried out using a scanning electron microscope and a transmission electron microscope, while the chemical composition was measured using energy dispersive X-ray spectroscopy. Mechanical properties were tested using an INSTRON 8801 model combined with an extensometer, following ASTM standards.

ACKNOWLEDGEMENT

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