Research and Development of Stainless Steel with Hydrogen Embrittlement Resistance

Analysis of crystalline phase causing hydrogen embrittlement

Achievements

- Demonstration of the existence of a phase causing **hydrogen embrittlement**** by observing the crystalline microstructure of **stainless steel*** at room temperature
- Contribution to the research and development of a stainless steel resistant to hydrogen embrittlement and its applications

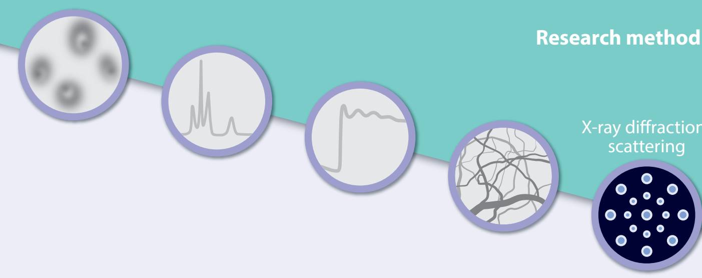
R&D facility: NIPPON STEEL Stainless Steel Corporation and Osaka Prefecture University

***Stainless steel:** Stainless steel is an iron alloy containing a minimum of 11% Cr. About ~ 50 million tons of stainless steel is produced worldwide per year. SUS304 (18-8 stainless steel) is a typical stainless steel that contains 18% Cr and 8% Ni and has high strength, ductility, and corrosion resistance. Therefore, it has been widely used as a material in built-in kitchens, elevators, and chemical plants and tanks.

****Hydrogen embrittlement:** Hydrogen embrittlement is a phenomenon that occurs in metals. When an external force is applied to metals into which hydrogen penetrates, their ductility and toughness decrease and they become brittle. Metals suffering from hydrogen embrittlement may break upon the application of force.

*****Lorentz transmission electron microscope (TEM):** A Lorentz transmission electron microscope is used to observe the magnetic domains of magnetic materials. Electrons that enter a ferromagnetic substance change their direction of travel upon the application of a Lorentz force via a magnetic field (deflection). Electrons are subjected to different deflections from neighboring magnetic domains. This enables a contrast to be observed at the neighboring magnetic domains in images, revealing the boundary of the phases.

Research method



Role of SPring-8

Background

In addition to the use of renewable energy as an alternative to fossil fuels, hydrogen has been attracting much attention as a source of energy. Stainless steel is a promising material for containers used for the storage and transport of hydrogen because of its excellent mechanical properties such as high strength and ductility as well as corrosion resistance. However, it may undergo hydrogen embrittlement.

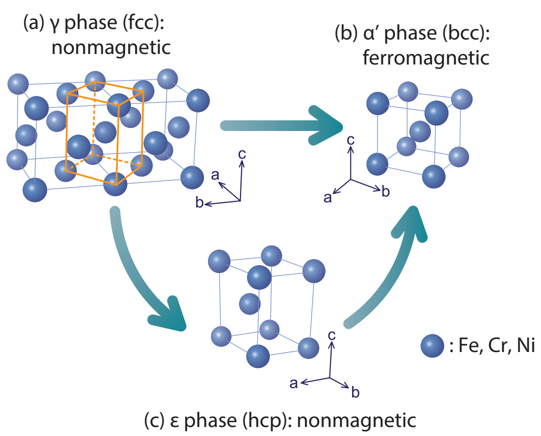
To develop stainless steel with hydrogen embrittlement resistance, the mechanism underlying hydrogen embrittlement should be clarified. However, the theory of hydrogen embrittlement has not been explained, and it has been difficult to observe the change in the crystalline microstructure during the processing of stainless steel at room temperature at an atomic scale.

Results

In situ X-ray diffraction observation of SUS304 specimens with and without prestrain was carried out at SPring-8 while the specimens were strained using a tensile testing machine. The results indicate that the ε phase, which is considered to be the cause of hydrogen embrittlement, was observed when the crystalline microstructure of the specimen transformed from the nonmagnetic γ phase to the ferromagnetic α′ phase. In addition, the site where the crystalline phase was formed was observed using a **Lorentz transmission electron microscope*****. The ε phase was observed at the twin boundary between the nonmagnetic γ phase or ε phase and the ferromagnetic α′ phase.

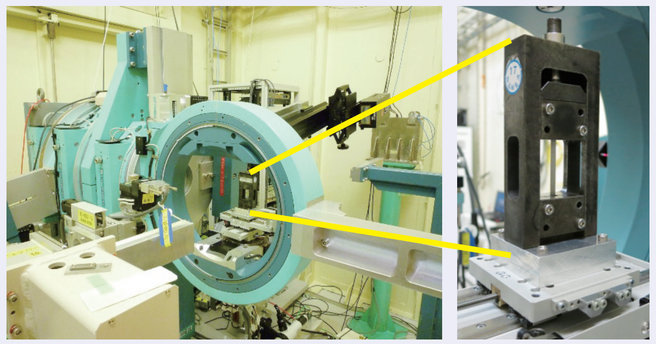
The above results indicate the existence of the ε phase, which is considered to cause the hydrogen embrittlement of stainless steel at room temperature, but it has not been observed thus far. It is expected that a new stainless steel with hydrogen embrittlement resistance will be developed and that new applications of this stainless steel will be proposed in the future.

Change in crystalline microstructure of SUS304



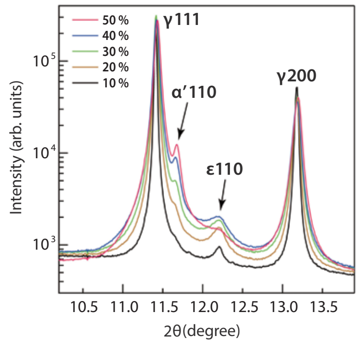
SUS304 crystals are nonmagnetic and have the face-centered-cubic crystalline structure (γ phase). When a SUS304 specimen is strained at room temperature, the γ phase is transformed to the body-centered-cubic crystalline microstructure (α′ phase) and the specimen becomes ferromagnetic (deformation-induced martensite transformation). This transformation improves the strength and ductility of SUS304. In this study, the generation of the ε phase, which is considered to cause hydrogen embrittlement, was confirmed during the transformation process from the γ phase to the α′ phase.

X-ray diffraction experiment at SPring-8

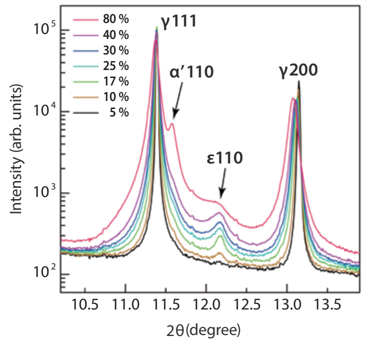


A multiaxis diffractometer, which enables easy control of the incident angle of X-rays and the detection angle of diffracted X-rays, and a tensile testing machine were installed in the station of BL02B1. The *in situ* observation of stretched SUS304 specimens with and without prestrain was carried out.

Observation of X-ray diffraction profiles using SUS304 specimens with and without prestrain

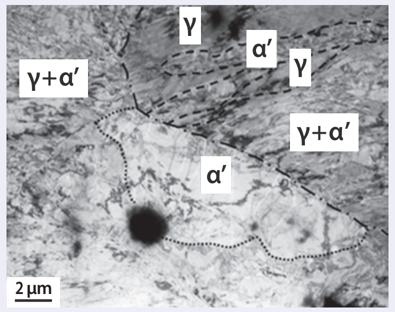


With increasing amount of tensile strain (10%, 20%,...), the area of the peak of the ε phase in the profile increases up to a tensile strain of 40%, after which it decreases. From this result, it is found that the ε phase starts to transform to the ferromagnetic α′ phase at a tensile strain of ~ 30%.

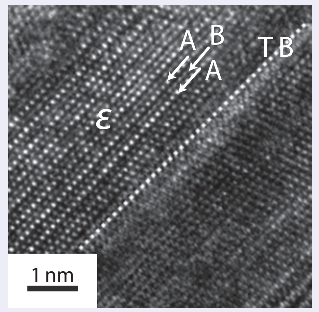


To observe the transformation process from the ε phase to the α′ phase, *in situ* observation of strained SUS304 specimens without tensile strain was carried out. With increasing amount of strain, the amount of the ε phase increases, which is followed by the appearance of the α′ phase.

Observation of magnetic phase: Lorentz TEM image of fracture specimen



The boundary between black and white magnetic phases was observed in the area of the α′ phase in a Lorentz TEM image of a fractured specimen.



An array of crystals unique to the ε phase ("ABAB") was observed in the high-resolution TEM image of a SUS304 specimen subjected to 20% tensile strain (compared with "ABC" in the general γ phase).