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Precipitation of sigma and chi phases in δ-ferrite of Type 316FR weld metals

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ARTICLE DATA ABSTRACT

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The decomposition behavior and kinetics of δ -ferrite are examined using aging treatments between 873 and 1073 K for Type 316FR stainless steel weld metals with different solidification modes (316FR AF, 316FR FA). The dominant precipitates are sigma, chi, and secondary austenite nucleated at δ-ferrite/austenite interfaces or in the interior of the ferrite grains. These precipitates consume all the ferrite during isothermal aging in both 316FR AF and FA weld metals. Differences in the precipitation behavior (precipitation initiation time and precipitation speed) between weld metals can be explained by і) the degree of Cr and Mo microsegregation within δ-ferrite or austenite near ferrite and іі) the nucleation sites induced due to the solidification mode (AF or FA), such as the ferrite amount. For both weld materials, a Johnson–Mehl-type equation can express the precipitation behavior of the sigma + chi phases and quantitatively predict the behavior at the service-exposure temperatures of a fast breed reactor.

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1. Introduction

Compared to common austenitic stainless steels, an advanced Type 316FR austenitic stainless steel has an improved creep rupture resistance and is a key component in the nextgeneration of commercialized fast breed reactors (FBRs) [\[1](#page--1-0)–6]. Because a FBR typically operates for about 60 years at high temperatures (773–823 K) due to its outstanding ability to generate heat compared to other types of nuclear reactors (ex. light water-cooled reactor), aging of the structural materials is an inevitable issue. Thus, before FBRs can be commercialized, the aging behavior and the appropriate repair processes must be clarified [\[7\].](#page--1-0) One of the most important factors to determine the aged degree of components made of austenitic stainless steels is the inferior microstructural stability of the weld metal at elevated temperatures. Weld metals of austenitic stainless steel generally involve some amount of δ-ferrite, which is used to prevent solidification cracking during welding. Weld metals are typically composed of different solidification modes, such as primary austenite (γ) with a secondary phase δ -ferrite solidification mode (AF mode) or primary δ-ferrite with a secondary phase γ solidification mode (FA mode) according to the Cr_{eq}/Ni_{eq} variation. However, δ-ferrite may decompose into intermetallic phases (sigma or chi) in a high temperature environment, negatively impacting the mechanical and chemical properties of a weld joint [\[8\].](#page--1-0)

Hot cracking is a common problem while welding austenitic stainless steel in the AF mode [\[9\]](#page--1-0). Because changing the solidification mode or optimizing the δ-ferrite content during solidification can control the hot cracking susceptibility [\[9\]](#page--1-0), the

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chemical composition of the consumable welding components can be adjusted in the FA mode. However excessively increasing the amount of ferrite intensifies the embrittlement of the weld metal because a large amount of intermetallics is transformed while in actual service.

Although numerous studies have investigated the aging behavior (precipitation) in austenitic and duplex stainless steels [10–[38\],](#page--1-0) information about the kinetics for the weld metal is insufficient compared to that for the base metal [\[32](#page--1-0)–38]. Most studies have approximated the kinetics for δ-ferrite decomposition without employing a detailed approach to evaluate individual precipitates [32–[38\]](#page--1-0). Moreover, comparison studies on the precipitation kinetics in weld metals between the AF and FA modes with similar composition ranges are insufficient, despite the possibility that the kinetics may depend on microsegregation according to solidification mode variations.

The present study aims to clarify the δ-ferrite decomposition behaviors in Type 316FR stainless steel weld metals utilizing welding consumables with two different solidification modes (AF and FA modes). The decomposition behaviors of ferrite are quantified by accurately measuring the amount of each precipitate. Based upon quantitative analyses, kinetic approaches are pursued and different precipitation behaviors between welding consumables are discussed.

2. Materials and Experimental Procedure

The materials used are within the compositional range of Type 316FR stainless steel. Table 1 lists their chemical compositions. The designed steel with the AF mode is referred to as 316FR AF, while that with the FA mode is referred to as 316FR FA. The base metal plates of these steels, which measured 120 mm \times 40 mm \times 3 mm, were manufactured via hot-rolling. To prepare the weld metal, bead-on-plate welding was performed using gas tungsten arc welding (GTAW) on the manufactured plates with an arc current of 110 A, arc voltage of 14 V, and a welding speed of 1.67 mm/s. Welded specimens were aged at various conditions (Table 2) and subsequently quenched with water.

The as-welded microstructures were observed by electron backscatter diffraction (EBSD) after electrolytic etching with a 10% aqueous solution of KOH. To investigate the elemental distribution in the as-welded state, an electron probe X-ray micro analyzer (EMPA) measured the concentration profiles of the elements. Precipitates were identified by transmission electron microscopy (TEM) under an acceleration voltage of 200 kV after jet polishing with a solution of perchloric acid (5%) and acetic acid (95%) using an applied voltage of 50 V. To clarify the microstructural changes due to the aging treatment, specimens were observed by scanning electron

microscopy (SEM) and EBSD using both electrolytic etching and mechanical polishing with colloidal silica. Image processing at a 1000-time magnification on the basis of the SEM micrographs quantitatively measured the area fraction of the precipitates.

3. Characterization of Weld Microstructures and Solidification Modes

[Fig. 1,](#page--1-0) which shows the as-welded microstructure of the 316FR AF weld metals, confirms the cellular morphology associated with the solidification behavior. All the δ-ferrite is located at the cell boundaries or triple points of γ with an elongated or globular shape. The average volume fraction of ferrite is 2%. These characteristics are consistent with the AF mode [\[39\]](#page--1-0).

[Fig. 2](#page--1-0) shows the microstructure of 316FR FA after GTAW bead-on-plate. The average volume fraction of δ-ferrite is 5%. The shape associated with the bands of vermicular δ-ferrite enclosed by γ at the center of a dendritic cell is consistent with the FA mode [\[40\].](#page--1-0)

[Fig. 1](#page--1-0) also shows the corresponding pole figures to examine the crystallographic orientation relationship between γ and δ-ferrite. The preferred orientation relationship between γ and δ-ferrite is (111) _γ// (110) _δ, $[111]$ _γ// $[110]$ _δ (Kurdjumov–Sachs crystallographic orientation relationship, which is hereafter referred to as the K–S relationship) together with a virtually parallel relationship ((100)_γ//(100)_δ: preferred growth direction) with a small deviation. Similar relationships are confirmed at other locations of the weld beads.

The crystallographic orientation relationship between vermicular δ -ferrite and γ was also examined. The pole figure in [Fig. 2](#page--1-0) shows a perfect parallel relationship $[100]$ _v// $[100]_{\delta}$ with a nearly K–S relationship and a small angle deviation. These observations confirm different orientation relationships between γ and δ-ferrite in the 316FR AF weld metals. These results are included in the previous results for the crystallographic orientation relationship between γ and δ-ferrite in austenitic stainless steel weld metals performed by Inoue et al. [\[39,40\]](#page--1-0).

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