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## Effect of hot-rolling and cooling rate on microstructure and high-temperature strength in 9CrODS steel

XCH. Wu<sup>a,\*</sup>, S. Ukai<sup>b</sup>, R. Miyata<sup>a</sup>, N. Oono<sup>b</sup>, S. Hayashi<sup>b</sup>, B. Leng<sup>a</sup>, S. Ohtsuka<sup>c</sup>, T. Kaito<sup>c</sup>

<sup>a</sup> Hokkaido University, Graduate School of Engineering, Materials Science and Engineering: N13, W-8, Kita-ku, Sapporo 060 8628, Japan

<sup>b</sup> Hokkaido University, Faculty of Engineering, Materials Science and Engineering: N13, W-8, Kita-ku, Sapporo 060 8628, Japan

<sup>c</sup> Japan Atomic Energy Agency: 4002, Oarai, Ibaraki 311-1393, Japan

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### ABSTRACT

The 9CrODS steel specimens were prepared by different processing with hot-rolling and different cooling rate. The hardness and high-temperature tensile properties were measured. Microstructure was analyzed by means of EBSD inverse pole figure and kernel average misorientation angles. The hot-rolled and then air-cooled specimen has the highest tensile strength. The furnace-cooled specimen also has better tensile strength at 700 °C than air-cooled specimen at normalized condition. The high-temperature strength of 9CrODS steel is significantly improved with increasing grain size that can be induced by hot-rolling or furnace-slow cooling, where the localized grain boundary deformation can be suppressed.

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### 1. Introduction

9CrODS (oxide-dispersion-strengthened) ferritic steel, which has high temperature strength and excellent radiation resistance, is promising candidate material for the advanced fission and fusion blanket system [1]. In former research [2–4], it was confirmed that 9CrODS steel consists of a dual phase; that is a martensite phase and a residual ferrite phase. The formation and strengthening mechanism of the residual ferrite phase were studied in details [5]. We also found that a transform ferrite phase induced by hot-rolling can improve the tensile strength of 9CrODS steels [6]. In this paper, for furthermore study, we investigate effects of hot-rolling at the  $\gamma$ -austenite region and subsequent cooling at different cooling rate that could modify the microstructure and mechanical properties. For those specimens, hardness and high-temperature tensile properties are measured. Inverse pole figure (IPF) and kernel average misorientation (KAM) are analyzed to evaluate the microstructural change and strengthening mechanism.

### 2. Experimental produce

The pure metal powders were mixed together in composition of 9Cr–0.13C–2W–0.2Ti–0.35Y<sub>2</sub>O<sub>3</sub> (wt%); then they were mechanically alloyed by a planetary-type ball mill (Fritsch P-5) for 48 h in argon gas atmosphere. A weight ratio of the powder to ball is 1/10. The rotation speed is 300 rpm. The mechanically alloyed powders were consolidated by spark plasma sintering (SPS) at 1100 °C for 2 h with a loading pressure of 44 MPa. Then, the con-

solidated specimens were hot-rolled in a reduction of 80% at  $\gamma$  austenite region and followed by two type of cooling. One is furnace cooling (FC) by returning the hot-rolled (HR) specimen into a furnace at temperature of 850 °C then it was cooled at a cooling rate of 100 °C/h, which is designated as HR–FC. The other is air cooling (AC) at 10,000 °C/h after hot-rolling; it is designated as HR–AC. As a control specimen, normalizing (N) specimens were prepared at 1050 °C for 1 h, then followed by furnace cooling, designated as N–FC, or air cooling, N–AC.

Microhardness of the specimens were measured by Shimadzu HMV-2 microhardness tester at a loading of 9.8 N and duration of 5 s. The high-temperature tensile test was conducted by using miniaturized specimens at 700 °C and strain rate of  $1.0 \times 10^{-3}$ /s under argon gas atmosphere. The IPF and KAM figures were analyzed by field-emission (FE) type scanning electron microscope (SEM).

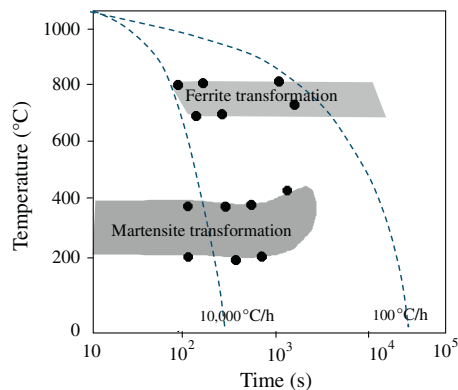
### 3. Results and discussion

#### 3.1. Phase characterization

According to a continuous cooling transformation (CCT) diagram of normalized 9CrODS steels [7], as showed in Fig. 1, the phase compositions of the specimens was analyzed. When the specimens are cooled from the austenite region at the cooling rate of about 10,000 °C/h, the cooling curve almost does not cross the ferrite transformation region, but quickly come across the martensite transformation. Thus, the austenite will transform into martensite. The previous study of 9CrODS steels also reported that the normalized specimen has a residual ferrite phase that cannot be transformed into austenite when it was heated up above  $A_{C3}$

\* Corresponding author. Tel.: +81 11 711 6362; fax: +81 11 711 6355.

E-mail address: [chaoxiaowu\\_008@163.com](mailto:chaoxiaowu_008@163.com) (XCH. Wu).



**Fig. 1.** Continuous cooling transformation (CCT) diagram of normalized 9CrODS steels; black dots show the boundaries measured by thermal expansion method during the various cooling rate.

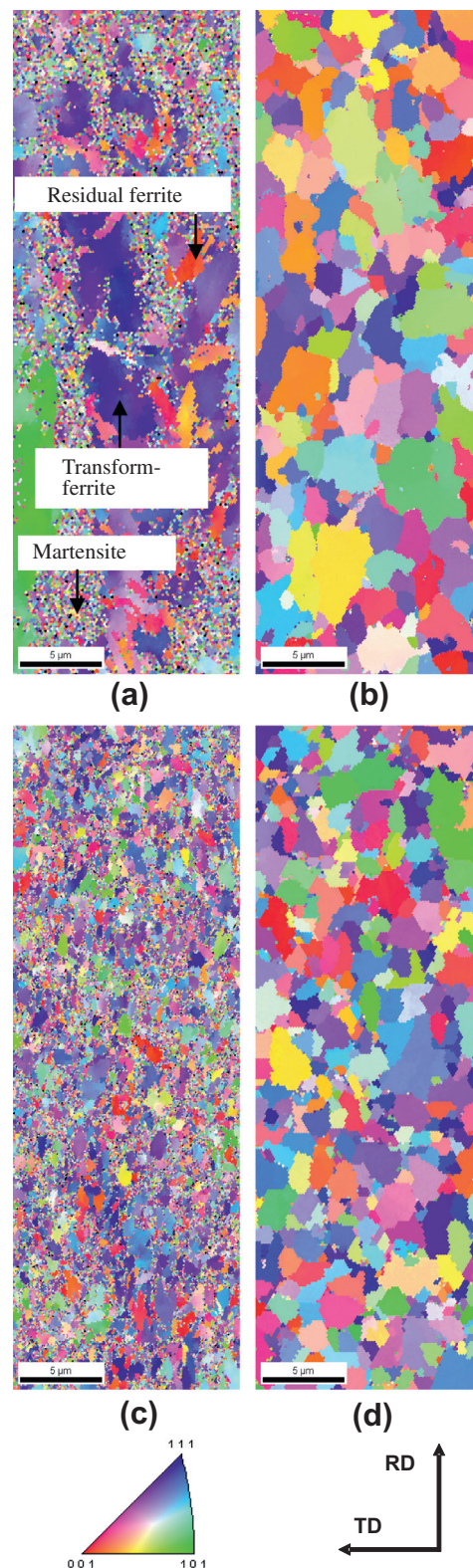
temperature [5]. Based on above information, the N-AC specimen consists of martensite and residual ferrite. However, the situation of HR specimen is different, because hot-rolling can shift the ferrite transformation region toward the left side of the diagram. That means, when HR specimen is cooled down at the same cooling rate as AC (10,000 °C/h) condition, it will cross the ferrite transformation region. We designated this ferrite as transform-ferrite [6]. Thus, HR-AC specimen contains three different phases; residual ferrite, transform-ferrite and martensite, where the residual ferrite and transform-ferrite have the same crystalline structure. However, microstructure and hardness in both ferrites could be different.

The cooling rate of FC specimen is 100 °C/h. For both N specimen and HR specimen, cooling curve cannot cross the martensite transformation region when they cooled from austenite phase, suggesting that they are composed of both residual ferrite and transform-ferrite. These phase characterization is summarized in Table 1.

### 3.2. IPF analyses

The microstructures of specimens analyzed by IPF are shown in Fig. 2. These data are characterized on the basis of CCT diagram mentioned in Section 3.1. The HR-AC specimen shown in Fig. 2a consists of some irregular shape small grains and a few coarse grains. The formation mechanism of the coarse grains was extensively evaluated in the previous study, and these coarse grains were proven to be transform-ferrite [6]. The process of the ferrite-transformation and resultant coarse grain formation in the conventional steels induced by HR-AC is reviewed by Furuhashi et al. [8]. Some areas of the figure contain full of noise, which correspond to martensite having a lot of strains. Other small red color grains are also identified as the residual ferrite.

The HR-FC specimen was composed of nearly equi-axed grains, and most of them are in a similar size as shown in Fig. 2b. Although there are also some small grains, they are quite uniform microstructure in round shape. As previously mentioned, HR-FC specimen is composed of the residual ferrite and transform-ferrite, but



**Fig. 2.** The microstructures of specimens analyzed by IPF: (a) hot-rolling and air-cooling (HR-AC), (b) hot-rolling and furnace cooling (HR-FC), (c) normalizing and air-cooling (N-AC) and (d) normalizing and furnace-cooling (N-FC).

it is difficult to distinguish them from the IPFmap. From Fig. 3a and b showing the size distribution measured in these two specimens, the grain size of HR-FC specimen ranges from 0.2 μm to 5 μm, and it distributes more uniformly as compared with HR-AC specimen.

**Table 1**

Phase composition of the specimens.

Specimens	Phase composition
HR-AC	Residual ferrite + transformed ferrite + martensite
HR-FC	Residual ferrite + transformed ferrite
N-AC	Residual ferrite + martensite
N-FC	Residual ferrite + transformed ferrite

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