High Temperature Deformation of Laves Phase Precipitates in Alumina-forming Austenitic Stainless Steels

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Abstract

Laves phases with the C14 type structure are present in recently-developed, alumina-forming austenitic (AFA) stainless steels. Here, **for the first time**, we show the Laves phase undergoes plastic deformation when such AFA steels are deformed at 700 °C at strain rate of $5 \times 10^{-4} \text{ s}^{-1}$. The gliding dislocations in the Laves phase leave behind stacking faults that we have analyzed using transmission electron microscopy. The stacking faults are on the basal plane of the Laves phase and are bounded by $1/3 \langle 10\bar{1}0 \rangle$ partial dislocations. **These slip of dislocations are effective plastic deformation and improve the ductility of AFA materials**

Introduction

Laves phases are intermetallic phases that have a composition AB_2 . There are three different classes: cubic $MgCu_2$ (C15), hexagonal $MgZn_2$ (C14), and hexagonal $MgNi_2$ (C36). The latter two classes are unique forms of the hexagonal arrangement, but share the same basic structure [1]. **Laves phases have been shown to be** brittle at ambient temperature due to the difficulty in moving dislocations which occurs by a complex deformation process called synchroshear [2, 3]. Laves phase are often present in many multi-phase alloys, and are generally believed to have a detrimental effect on the overall mechanical properties. However, recently, there have been considerable efforts to develop new austenitic heat-resistant steels strengthened by Laves phases [4, 5].

Fe_2M (M is typically Nb) Laves phases are often present in conventional heat-resistant steels and alloys, and **are considered to** have an unfavorable effect on the mechanical properties. However, in a Fe–20Cr–30Ni–2Nb alloy with 89% grain boundary coverage by Laves phase precipitates a high creep ductility of ~70% at elevated temperatures [6, 7] was observed, indicating that the Fe_2Nb Laves phase on grain boundaries does not reduce the creep ductility. In a Fe–20Cr–30Ni–2Nb–5Al alloy, it was found that even when the grain boundaries had 93% coverage with NiAl and Laves phase precipitates, the alloy still exhibited 19% elongation at room temperature [8]. Furthermore, it was recently found that the Fe_2Nb Laves phase that is in equilibrium with the γ -Fe phase can exhibit plastic strain of around 4% under nanoindentation at room temperature [7, 9]. Thus, the Laves phase on grain boundaries may not negatively affect the ductility of Fe–20Cr–30Ni–2Nb–5Al based steels at ambient temperature.

Binary stoichiometric Fe₂Nb (Fe-33.3 at. % Nb) does not exhibit any planar faults and has a very low dislocation density. A similar situation was also observed in the stoichiometric Fe₂Nb containing 18 at. % Ni and in binary Fe-rich Fe₂Nb (Fe-27.5 at. % Nb) systems. When Ni content increased, many planar faults parallel to the basal plane of the C14 structure were observed in Fe-rich Fe₂Nb where Fe was replaced by Ni. In this case, the fault density increased with increasing Ni content up to 33.1 at. %. [9, 10]

In the present study, we examined the mechanical properties of Fe-20Cr-30Ni-2Nb-5Al, alumina-forming austenitic (AFA) alloys at both room temperature and high temperature. The (Fe,Ni)₂Nb Laves phase present has the C14 type structure in the (face-centered cubic) f.c.c. AFA alloys. Here we show that stacking faults are formed in the Laves phase when the AFA was tensile tested at 700 °C at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ for the first time. Transmission electron microscopy was used to analyze the stacking faults in detail. The understanding of deformation on Laves phase will have influence on the design and development of future AFA alloys.

Experimental

The AFA stainless steel, referred to as DAFA29, was obtained from the Oak Ridge National Laboratory (ORNL). The details of the alloy post processing are described in previous publications [11, 12]. The analyzed chemistry of the AFA stainless steel is shown in Table 1.

The TEM specimen is prepared according to the method used in [11, 12]. The prepared TEM samples were examined using an FEI Tecnai F20 field emission gun (FEG) TEM operated at 200 kV with energy dispersive X-ray spectroscopy (EDS). Backscattered electron (BSE) imaging was performed using an FEI XL-30 field FEG scanning electron microscope (SEM).

Both room temperature and elevated temperature (700°C) tensile tests were performed using an Instron 5690 tensile testing machine. A preload force of 50 N was applied before the tensile tests. The initial strain rate for all tensile tests was $5 \times 10^{-4} \text{ s}^{-1}$. Elongations were measured directly from the gauge of the specimens after the tensile tests. Three tests were performed at each temperature.

Results and discussion

True stress - true strain curves for as-received DAFA29 at both room temperature and 700 °C are shown in Figure 1. At 700°C, the alloy shows a yield strength of $505 \pm 21 \text{ MPa}$, with little subsequent work hardening and fails at a true strain of 27%. The stress strain curve of DAFA29 tested at room temperature is shown for comparison. At room temperature, the yield strength is $560 \pm 16 \text{ MPa}$ after which considerable work hardening occurs reaching an ultimate tensile strength of $1140 \pm 23 \text{ MPa}$ at a true strain of $20 \pm 2\%$.

Figure 2a is a BSE image of a section along the tensile axis of DAFA29 after the room temperature tensile test. The bright contrast precipitates in Figure 2c and d are the large Laves phase precipitates [11, 13]. Many of these precipitates fractured during the tensile test at room temperature. The black lines inside the precipitates are cracks formed during the tensile tests. All the cracks are perpendicular to the tensile test direction as shown by the white arrow. Figure 2b

is a BSE image from a longitudinal section of DAFA29 after tensile testing at 700 °C. There are no cracks in the Laves phase. Some voids and small cracks are observed inside the f.c.c. matrix. The precipitate distributions after testing were unchanged from those before testing.

Multiple slip features were observed in the grain boundary Laves phase precipitates after the 700 °C tensile tests. These slip features are likely to have formed during the high temperature tensile tests. The slip bands are typically ~100 nm wide, see Figure 3a. All the slip features are aligned parallel to $\langle 10\bar{1}0 \rangle$. Softening of the Laves phase, possibly due to solid solution softening by Ni, may be why slip occurs in these particular Laves phase particles. Figure 3b is an x-ray spectrum showing the Laves phase and contains all of the transition metals present in the alloy.

The inset in Figure 3a shows a select area diffraction (SAD) pattern from this precipitate. The zone axis is $[11\bar{2}0]$. These slip features are expected to be on the basal planes [14]. In order to confirm this, the Laves phase precipitate was characterized in different diffraction conditions.

Figure 3c, d and e show bright field (BF) TEM images of the Laves phase precipitate observed in Figure 3a at different zone axes. When viewed along both $[11\bar{2}0]$ and $[1\bar{1}00]$ the slip features appear as lines, see Figure 3c and d. Viewing along the $[10\bar{1}1]$ (Figure 3e), those features became slip bands. By orienting these features corresponding to the $[11\bar{2}0]$ zone axis based on SAD patterns, we confirmed that these stacking faults are on the basal planes of the Laves phase. The fringes in both Figure 3c and d are the edge on images of these parallel planar features. The white arrows in Figure 3e shows one of slipping bands with a partial dislocation at the end of one or multiple stacking faults. This is similar to previous observations of stacking faults and bounding dislocations in other C14 structures such as Cr-Ta and Cr-Nb system [14, 15], Cr-Ti and Cr-Hf system [14, 16].

Recently, the partial dislocations associated with a basal stacking fault in the C14 structure have been directly observed. This verified the possibility of plastic deformation occurring by synchroshear [2, 3, 17]. Synchroshear requires two Shockley dislocations bounding a planar fault to glide on two different levels inside the triple layer with different Burgers vectors, e.g.: $1/3 [10\bar{1}0]$ and $1/3 [\bar{1}010]$ [3, 9]. The passage of these dislocations can produce slip along $\langle 10\bar{1}0 \rangle$ type directions on a basal plane in the Laves phase [9].

Conclusions

C14 type of Laves phases are present in f.c.c. AFA alloys. In this study, we characterized the deformation of Laves phases $((\text{Fe,Ni})_2\text{Nb})$ in AFA alloys under both room temperature and high temperature tensile tests for the first time. It was found:

1. In room temperature tensile tests, cracks formed in the Laves phases. In 700 °C tensile tests, no cracks were observed which might be due to the softening of Laves phase.
2. The softening of Laves phase is mainly contributed from solid solution softening by Ni and several other transition metals that are present.
3. Multiple stacking faults are formed on the basal plane of Laves phase under tensile stress.

3. The main deformation mechanism in the $(\text{Fe,Ni})_2\text{Nb}$ C14 Laves phase is basal slip. All the slipping features in the Laves phase show $\langle 10\bar{1}0 \rangle$ slip directions and $1/3 \langle 10\bar{1}0 \rangle$ partial dislocations are shown to bound the stacking faults

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Figure

Figure 1 Comparison of true stress - true strain curves for DAFA29 at 700 °C (black curve) and room temperature (dashed red curve)

Figure 2 BSE images of longitudinal sectional region (Note the cracks in the Laves phase after room temperature testing) in DAFA29 after tensile tests at a) room temperature, and b) 700 °C, c) and d) are the EDS results of the bright contrast precipitates in a) and b), respectively.

Figure 3 Slip features in a Laves phase precipitate after deformed at 700 °C. (a) TEM images of the Laves phase precipitate (inset in a is the SAD of the circled region), (b) EDS from the circled region of the Laves phase precipitate, (c, d, e) are BF TEM images of the slip bands in the Laves phase precipitate obtained from three different zone axis, insets in (a, b, c) are the corresponding SAD of the precipitate.

Table 1 Analyzed chemistry (wt.%) of as-received DAFA29.

Elements	wt. %
Fe	45
Cr	14
Mn	0.1
Ni	32
Cu	0.1
Al	3
Si	0.2
Nb	2.9
V	<0.01
Ti	2
Mo	0.1
W	<0.01
Zr	0.3
C	0.1
B	<0.001
P	<0.005
N	<0.0001