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Precipitate characteristics and their effects on the high-temperature creep resistance of alumina-forming austenitic stainless steels

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ABSTRACT

In this paper, the dynamic evolution of precipitates and its influence on the high-temperature mechanical properties of newly developed AFA steels were systematically investigated. At 1023 K or above, three main types of precipitates, i.e., the B2-NiAl, Laves-Fe_2Nb, and δ/σ phases, were formed in the base steel, and the major strengthening medium is Laves-Fe_2Nb, which coarsened quickly, leading to undesirable creep properties. Phase competition between the most effective strengthening NbC nanosized precipitates and the Laves-Fe_2Nb phase was analyzed, and it was found that adjusting the Nb/C ratio in the steels could enable the precipitation of highly stable, fine NbC particles. In addition, the formation of detrimental σ phases could be suppressed by lowering the Mo and Si content in the alloy. Eventually, a new type of AFA steel consisting of a high density of nanosized NbC particles homogeneously dispersed in the austenitic matrix was successfully developed, and significant enhancement in the creep resistance was achieved due to the effective strengthening resulting from the tiny secondary NbC particles.

1. Introduction

The energy crisis and global warming demand the development of high-performance structural materials to improve energy efficiency. For example, to increase the efficiency of energy conversion systems, such as boiler/steam turbine power plants, the most feasible method is to increase the steam temperature and pressure [1]. As such, next-generation structural materials are required that simultaneously possessing higher creep strength and larger oxidation-resistance at elevated temperatures than those currently used. Alumina-forming austenitic stainless steels (AFA) are a relatively new class of dispersion-strengthened austenitic steels [2–6] and exhibit superior oxidation-resistance to conventional stainless steels due to the formation of an Al_2O_3-based protective scale at high temperatures. Recently, research in this field has focused on high-temperature oxidation mechanisms [7–9], whereas little attention has been paid to the mechanical performance of these steels, particularly the high-temperature creep behavior, which is critical for their engineering applications.

It is well known that most of the high-temperature structural materials, such as conventional heat-resistance austenitic steels, Ni-base superalloys, oxides-dispersion strengthened alloys and particulate reinforced metal matrix composites, are strengthened via the so-called precipitation hardening. Naturally, the morphology, distribution and thermal stability of precipitates have a significant impact on the high-temperature mechanical properties of these metallic materials. During long-term service at high temperatures, precipitates could be coarsened and/or dissolved into an alloy matrix due to their low thermal stability. In some extreme cases, harmful precipitates could be formed, which greatly degraded the structural materials and severely shortened their life time. Therefore, knowledge regarding the precipitate formation kinetics and stability under stress at elevated temperatures is vital for developing high-temperature structural materials. In fact, the precipitation behavior of different reinforcing phases in austenitic steels has been widely investigated. Takeyama et al. studied the Laves-Fe_2Nb and δ-Ni_3Nb precipitates in austenitic steels at 1073 K [10]. Maziasz found that the addition of Si could promote the formation of the Laves phase in heat-resisting austenitic steels [11], while McGurty et al. reported a class of precipitation-hardening austenitic alloys strengthened by B2-NiAl [12]. For the newly developed AFA steels, however, the

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precipitation behavior and its influences on the mechanical performance remain to be elucidated systematically.

In this paper, the alloying and annealing effects on the formation and stability of precipitates in recently developed AFA steels will be reported. In addition, the relationship between the characteristics of the different precipitates and the mechanical properties will be explored, and the alloy design principle for optimizing high-temperature mechanical performance will also be summarized.

2. Experimental

The chemical composition of the AFA steels that were investigated is presented in Table 1; the actual composition measured by chemical titration analysis is also included for comparison. The alloy ingots were prepared by arc melting with a non-consumable tungsten electrode under argon atmosphere using commercially pure elements as the starting materials. The ingots were remelted at least eight times and then drop-cast into a 15 mm × 15 mm × 100 mm copper mold. The as-cast bars were sealed in silica tubes under Ar atmosphere, homogenized at 1523 K for 24 h, and then cold-rolled 10–40%. The cold-rolled specimens were then sealed in tubes under Ar atmosphere and recrystallized at 1523 K for approximately 4 h to control the grain size, followed by water quenching.

The recrystallized specimens were annealed either isothermally at a fixed temperature of 1023 K for a different time span, i.e., 0, 24, 48, 116, 164, 450 and 1000 h, or for a fixed time span of 15 h at varied temperatures, i.e., 973, 1003, 1023 and 1073 K. Dog-bone shaped tensile specimens, with a thickness of 1–2 mm, a gauge length of 12 mm and a width of 4 mm, were prepared by electro-discharged machining and then polished using 2000-grit SiC papers. Tensile tests were conducted in air using the strain-rate-increase method suggested by Mecking [13]. Creep tests were performed in air at 1023 K under a constant load of 100 MPa. Thermodynamic calculations were performed using the Thermo-Calc software package [14] and the same thermodynamic database as applied in our previous study [15].Strain-rate jump tests were conducted, and six strain rates (i.e., 6.4×10^{-7}, 2.6×10^{-6}, 6.4×10^{-6}, 3.2×10^{-5}, 3.2×10^{-4} and $3.2\times10^{-3}\ s^{-1}$) were successively applied on each specimen to analyze the aging effects on the steady state flow behavior of AFA alloys. Microstructures and phase identification of the annealed specimens were examined using a scanning electron microscope (SEM, Zeiss Supra 55, 15 kW) equipped with an energy dispersive spectrometer (EDS), a transmission electron microscope (TEM, Tecnai G2 F30) operated with a voltage of 200 kV, and an X-ray diffraction (XRD, Rigaku DMAX-RB-12KW) instrument using Cu-Kα radiation at a scan rate of 10°/min and a step size of 0.02°. The TEM samples were first mechanically ground to a 50-μm thick plate and then twin-jet electropolished using a solution mixed in the ratio of $HClO_4$: CH_3CH_2OH = 1:19.

3. Results

3.1. Formation and stability of precipitates in the base AFA steel

Backscattered electron SEM images and the corresponding XRD patterns of the base AFA steel aged at 1023 K (the targeted service temperature) for different aging time periods are shown in Figs. 1 and 2, respectively. As shown in Fig. 1a, only bright spherical particles of micrometer size are observed in the as-solutionized specimen with no aging. Based on the XRD results in Fig. 2, these particles can be identified as NbC, and the matrix is confirmed to be austenite. Note that these large NbC particulates were formed during solidification (termed as primary NbC) and could not be dissolved during the high-temperature homogenization process. The total volume fraction of the primary NbC phase is estimated to be ~1.7%.

After being aged at 1023 K for 24 h, the steel exhibited three extra precipitates in addition to the large primary NbC particles, i.e., the gray, dark and bright phases at the grain boundaries, as shown in Fig. 1b. Based on the EDS results in Fig. 1g and h and the XRD results in Fig. 2, three phases were formed: the B2-NiAl, δ-phase and Laves-Fe_2Nb. The gray particles at the grain boundaries are confirmed to be of δ-phase, while the dark ones are B2-NiAl. In reference to the TEM data in Fig. 3, the fine bright particles can be confirmed to be Laves-Fe_2Nb. When the aging time reached 48 h, the specimen exhibited a similar XRD pattern as that of the specimen aged for 24 h. When the aging time exceeds 116 h, the σ-phase started to form, as shown in Fig. 2b, which is a magnified view of Fig. 2a at the 2θ angle from 40° to 55°. In addition, the B2-NiAl and Laves-Fe_2Nb phases were still present, although their sizes were gradually increased (Fig. 1d). Nevertheless, the types of the precipitates remain the same, even as the aging time increased up to 450 h, as confirmed by the results in Figs. 1e and 2.

As shown in Figs. 1b and 2, the Laves-Fe_2Nb precipitates appeared after 24 h aging at 1023 K and quickly grew to a size in-between 300 and 400 nm in the specimens aged for over 48 h, as illustrated in Fig. 1c–e. To further demonstrate the growth tendency of different phases, Fig. 3 shows the AFA steel aged for 15 h at different temperatures: 973, 1003, 1023 and 1073 K. As shown in Fig. 3a and b, the fine NbC particles (hereafter termed as secondary NbC phase) with a size of approximately 40 nm are the only precipitate at 973 K. As the annealing temperature is

Table 1
Nominal and actual chemical composition of the AFA steels (wt%) in this study.

	Base		1.5Nb0.15C		1.0Nb0.1C		0Mo		0Si		0.5Nb0.08C	
	Nominal	Actual	Nominal	Actual	Nominal	Actual	Nominal	Actual	Nominal	Actual	Nominal	Actual
Cr	18	17.90	18	17.91	18	17.93	18	17.99	18	17.84	18	17.94
Ni	25	25.21	25	25.08	25	24.95	25	24.96	25	24.69	25	25.08
Al	3	3.01	3	3.02	3	2.99	3	2.95	3	2.97	3	2.97
Si	0.15	0.15	0.15	0.18	0.15	0.18	0.15	0.15	–	–	0.08	0.13
Nb	1.5	1.50	1.5	1.57	1.0	1.02	1.5	1.49	1.5	1.50	0.5	0.51
Mo	1.5	1.44	1.5	1.53	1.5	1.51	–	–	1.5	1.50	0.8	0.83
C	0.08	0.07	0.15	0.16	0.1	0.1	0.08	0.083	0.08	0.086	0.08	0.085
B	0.01	0.008	0.01	0.005	0.01	0.007	0.01	0.006	0.01	0.005	0.01	0.007
P	0.04	0.030	0.04	0.039	0.04	0.036	0.04	0.073	0.04	0.039	0.04	0.035
Hf	0.15	0.014	0.15	0.14	–	–	0.15	0.14	0.15	0.14	–	–
Y	0.01	0.008	0.01	0.005	0.1	0.04	0.01	0.005	0.01	0.005	0.1	0.07
Ti	–	–	–	–	–				–	–	0.1	0.08
Fe	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.