Effect of Mo on δ phase precipitation and tensile properties in cast alloy IN625

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Abstract

Cast alloy IN625 is a candidate Ni-based alloy for advanced ultra-supercritical (A-USC) power plants. In order to investigate the influence of Mo on δ phase precipitation, the heat treatment experiments on the Mo series of alloys were carried out at 750 °C (the service temperature of A-USC power plants) up to 17,000 h, and effects of δ phase on the tensile properties were also studied. The results show that lots of δ phase precipitated after long-term aging at 750 °C and the Mo content has a weak effect on the precipitation of the δ phase. With the increasement of δ phase content, the strength of alloys after aging at 750 °C for up to 5,000 h increased nearly linearly with the increasing aging time while the elongation was affected by δ precipitation to a relatively limited extent. It is suggested that the δ phase serves as a strengthening phase in cast alloy IN625.

K e y words: δ phase, element Mo, cast alloy IN625, AUSC

1. Introduction

Pulverized coal combustion (PCC) power plant dominates the power industry and maintains its superiority for the foreseeable future. An increase in the operating efficiency of PCC power plants is necessary for meeting the demand for clean and affordable energy. One of the most effective ways is to increase the steam parameters. Therefore, advanced ultra-supercritical (A-USC) power plants are being developed with a steam temperature of 700 to 760 °C and pressure of 27.6 to 34.5 MPa. Ni-based alloy is selected as a candidate material for such service conditions [1– 3], such as IN625. This is because of its high strength, excellent fabricability and outstanding corrosion resistance [4–9]. Due to the large castings in A-USC power plants [4], cast alloy IN625 is highly needed, but the comparative study is scarce [10]. Meanwhile, the δ phase is easy to precipitate in cast alloy IN625 after long term aging at $700\text{--}760\,^\circ\!\mathrm{C}$ (the service temperature of A-USC power plants) [10]. So, studies on cast alloy IN625, such as the one presented in this paper, are needed to investigate its potential application in A-USC power plants.

Table 1. The nominal compositions of cast alloy IN625 with different Mo content (wt.%)

Alloy	Nb	Mo	\mathbf{Cr}	С	Ni	
8Mo 9Mo 10Mo	3.8 3.8 3.8	8 9 10	$21.5 \\ 21.5 \\ 21.5 \\ 21.5$	$0.05 \\ 0.05 \\ 0.05$	Bal. Bal. Bal.	

δ phase is an important phase in alloy IN625 and IN718. It has a significant effect on mechanical properties. In previous reports, the presence of the δ phase led to an increase in strength and loss in ductility in wrought alloy IN625 [11–15], but it had a beneficial effect on the rupture ductility in wrought alloy IN718 [16–19]. According to our work [10], the δ phase in cast alloy IN625 plays the same role on the mechanical properties as in wrought alloy IN625, and the precipitation of δ phase is strongly affected by Nb content. However, there is no available data on Mo's effect on δ precipitation in cast alloy IN625. Considering the above mentioned, this work aims to determine the effects of Mo on δ phase precipitation in cast alloy

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Alloy	8Mo			9Mo			10Mo						
Element	Nb	Mo	\mathbf{Cr}	Ni	Nb	Mo	\mathbf{Cr}	Ni	Nb	Mo	\mathbf{Cr}	Ni	
$\begin{array}{c} C_{\rm D} \\ C_{\rm I} \\ K \end{array}$	$1.9 \\ 5.7 \\ 3.00$	$7.0 \\ 10.5 \\ 1.50$	$23.2 \\ 20.7 \\ 0.89$	$67.9 \\ 63.1 \\ 0.92$	$1.9 \\ 5.8 \\ 3.05$	$7.5 \\ 10.1 \\ 1.34$	$23.0 \\ 21.0 \\ 0.91$	$67.6 \\ 63.1 \\ 0.93$	$2.0 \\ 6.3 \\ 3.10$	$8.8 \\ 13.7 \\ 1.56$	$22.3 \\ 20.8 \\ 0.93$	$66.9 \\ 59.2 \\ 0.88$	

Table 2. Element segregation ratio in cast alloy IN625 with different Mo content (wt.%)



Fig. 1. SEM micrographs of the δ phase in cast alloy IN625 with different Mo content after aging at 750 °C for up to 10,000 h: (a) 8Mo/1000 h, (b) 9Mo/1000 h, (c) 10Mo/1000 h, (d) 8Mo/5000 h, (e) 9Mo/5000 h, (f) 10Mo/5000 h, (g) 8Mo/10,000 h, (h) 9Mo/10,000 h, (h) 9Mo/10,000 h, and (i) 10Mo/10,000 h.

IN625 and the associated tensile properties are also discussed.

2. Materials and methods

Table 1 lists the nominal compositions of the ex-

perimental alloys used in this paper. The experimental alloys were prepared by vacuum induction. The alloys were solution-treated at $1200 \,^{\circ}$ for 1 hour, followed by water cooling and then aged at $750 \,^{\circ}$ for up to 17,000 hours.

Metallographic samples were prepared for observation in a JSM-6301F Scanning Electron Microscope

(SEM). Tensile tests at 700 °C were carried out on a Shimadzu AG-250KNE test machine. The cylindrical, threaded tensile test rods with a gauge length of ϕ 5 mm \times 25 mm machined from the thermally exposed bars were prepared as Standard GB/T 4338-2006 [20]. Each data are an average of at least two values of the tensile test.

3. Results and discussion

Table 2 gives the EDS-measured compositions (wt.%) at different positions and partition coefficients of the Mo series of as-cast alloys. The segregation level of alloying elements in as-cast structures can be expressed by the partition coefficient between the interdendritic region and the dendrite core: $K = C_D/C_I$, where C_I and C_D are the concentrations of the element in the interdendritic region and the dendrite core, respectively. Unlike in our previous work on the Nb series of alloys, the content of Mo has little impact on the element segregation level. Both series of alloys show the same equiaxed structure before aging treatment, and no δ phase precipitation is observed [10, 21].

The micrographs in Fig. 1 show the microstructures of the 8Mo, 9Mo, and 10Mo alloys after aging at $750 \,^{\circ}$ C for up to 10,000 h. Following aging for 1000 h, lots of needle-like δ phases precipitated in the Mo series of alloys, while few $\gamma^{\prime\prime}$ phase precipitates were observed. The amount of δ phase precipitation does not seem to be affected by the increasing Mo content. Identification of the δ phase is based on our previous work [10, 21]. Prolonged aging of the experimental alloys at 750 °C up to 10,000 h resulted in a significant increase in volume fraction of the δ phase precipitates for all the alloys. The increasing fraction of the δ phase precipitates corresponds to an increase in the aging time. However, the volume fractions of the δ phase precipitates for alloys aged 10,000 h and 17,000 h appear to be the same.

Few γ'' phase precipitates were observed after aging at 750 °C for up to 17,000 h. It is also noticed that Mo has a slight effect on δ phase precipitation, while the δ phase precipitation is strongly affected by Nb segregation [22]. The volume fraction of the δ phase was measured from the scanning electron microscope micrographs using *Image-Pro Plus 6.0 software* (Media Cybernetics Inc.). The results are shown in Fig. 2. It was visualized that the volume fraction of δ precipitation after aging at 750 °C increased with the aging time.

The values of the precipitation kinetics parameters were calculated to describe the effect of Mo on δ phase precipitation more intuitively, as shown in Fig. 2. At a given aging temperature, the relationship between the weight percentage of δ phase and aging time can

40
30
20
10
1000 3000 5000
10000
10000
1000
1000
1000
1000
1000
1000
1000
1000

Fig. 2. δ phase volume fraction evolution as a function of aging time and Mo content in cast alloy IN625 with different Mo content after aging at 750 °C for up to 17,000 h.



Fig. 3. Relationship between $\ln[-\ln(1 - V_{\delta}/V_{s})]$ and $\ln(t)$ in cast alloy IN625 with different Mo content after aging at 750 °C.

be described by the Avrami equation, Eq. (1):

$$W_{\delta} = W_{\rm s} \left[1 - \exp(-\alpha t^n) \right], \tag{1}$$

where n is a constant that depends on the nucleation and growth of δ phase, α is the rate of δ precipitation, and $W_{\rm s}$ is the saturation level for the δ precipitation [23]. In this work, W_{δ} and $W_{\rm s}$ are replaced by V_{δ} (the volume percentage of δ phase) and $V_{\rm s}$ (the saturation level for the δ precipitation), respectively. The values of the precipitation kinetic parameters obtained by fitting the experimental data (Fig. 3) to Eq. (1) are listed in Table 3. The effect of Mo on δ phase precipitation can be approximately described by the values of n and α . It is observed that as the content of Mo increases, the value of α increases while the value of n decreases. Here, it is worth mentioning that the values of n and α change slightly when compared to those in

T a ble 3. Parameters for precipitation kinetics of δ phase in cast alloy IN625 with different Mo content after aging at 750 $^{\circ}\mathrm{C}$

А	8Mo	9Mo	10Mo	
lpha n	$1.362E-6 \\ 0.845$	$3.51E-6 \\ 0.792$	3.667E-6 0.798	



Fig. 4. The tensile results at 700 °C of cast alloy IN625 with different Mo content after aging at 750 °C for different time: (a) yield strength, (b) ultimate tensile strength, (c) elongation, and (d) area reduction.



Fig. 5. The tensile results at 700 °C of cast alloy IN625 with different Mo content as a function of δ phase: (a) yield strength, (b) ultimate tensile strength, and (c) elongation.

Nb series of alloys. This may be due to the weak influence of Mo on the degree of Nb segregation. Hence, the experimental results show that the precipitation of the δ phase is not affected by Mo content.

Figure 4 presents the tensile properties at $700 \,^{\circ}{\rm C}$ of the Mo series of alloys as-heat treated and after aging at $750 \,^{\circ}{\rm C}$ for up to 5000 h. It is observed that the yield strength (YS), ultimate tensile strength (UTS), elon-

gation (EL), and area reduction (AR) after solution treatment appear to be insensitive to the varied additions of Mo. However, both YS and UTS increased considerably with aging time. This is similar to that in the Nb series of alloys after aging at 700 and 750 °C [10]. As opposed to the increase in strength, the values of EL and AR decreased with the increasing aging time. In general, aging for longer duration results in an increase in strength and loss in ductility.

As presented in Fig. 5, YS, UTS, EL, and AR values are plotted as a function of the δ phase volume fraction. It can be observed that both YS and UTS presented a similar behavior. They increased almost linearly with the δ phase volume fraction for all alloys. As for EL and AR, the values decreased with the increasing amount of δ phase. It is noticed that the majority of the elongation values are between 10 and 20 %, except for those obtained at the as-heat treated condition with no δ precipitates. All the above results are found to be in accordance with our previous work. This verifies our former conclusion that δ phases serve as a strong phase in cast alloy IN625 [21].

4. Conclusions

The effects of Mo on δ phase precipitation and the tensile properties in cast IN625 alloy were investigated in this paper. It is found that the increasing Mo content has a weak effect on the precipitation of the δ phase due to its little effect on Nb segregation. It was observed that the yield strength, ultimate tensile strength, elongation, and area reduction after solution treatment appeared to be insensitive to the varied additions of Mo. Both yield strength and ultimate tensile strength increased nearly linearly with aging time, which is primarily associated with the precipitation of the δ phase. The elongation was affected by δ precipitation to a relatively limited extent. δ precipitation is believed to serve as a strengthening phase in the Mo series of cast alloy IN625.

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References

 R. Zhou, L. Zhu, Y. Liu, Z. Lu, L. Chen, X. Ma, Microstructural evolution and the effect on hardness of Sanicro 25 welded joint base metal after creep at 973 K, J. Mater. Sci. 52(2017) 6161–6172. doi:10.1007/s10853-017-0758-6

- [2] X. Song, L. Tang, Z. Chen, R. Zhou, Micro-mechanism during long-term creep of a precipitation-strengthened Ni-based superalloy, J. Mater. Sci. 52 (2017) 4587– 4598. doi:10.1007/s10853-016-0703-0
- [3] S. J. Patel, J. J. Debarbadillo, B. A. Baker, R. D. Gollihue, Nickel base superalloys for next generation coal fired AUSC power plants, Procedia Engineering 55 (2013) 246–252. <u>doi:10.1016/j.proeng.2013.03.250</u>
- [4] P. D. Jablonski, J. A. Hawk, C. J. Cowen, P. J. Maziasz, Processing of advanced cast alloys for A-USC steam turbine applications, J. Miner. Met. Mater. Soc. 64 (2012) 271–279. <u>doi:10.1007/s11837-012-0241-4</u>
- [5] A. Nair, K. Somasundaram, Newer materials for supercritical power plant components – A manufacturability study, International Conference on Advances in Production and Industrial Engineering, NIT Trichy (2015), pp. 326–332.
- [6] F. Abe, Research and development of heat-resistant materials for advanced USC power plants with steam temperatures of 700 °C and above, Engineering 1 (2015) 211–224. <u>doi:10.15302/J-ENG-2015031</u>
- [7] N. Lückemeyer, H. Kirchner, H. Almstedt, Challenges in A-USC steam turbine design for 1300 °C F/700 °C, ASME Turbo Expo 2012: Turbine Technical Conference and Exposition, Copenhagen (2012), pp. 685– 693. doi:10.1115/GT2012-69822
- [8] J. A. Siefert, J. P. Shingledecker, J. N. DuPont, S. A. David, Weldability and weld performance of candidate nickel based superalloys for advanced ultrasupercritical fossil power plants, Part II: Weldability and cross-weld creep performance, Sci. Technol. Weld. 21 (2016) 397–427. doi:10.1080/13621718.2016.1143708
- [9] D. Castello, B. Rolli, A. Kruse, L. Fiori, Supercritical water gasification of biomass in a ceramic reactor: Long-time batch experiments, Energies 10 (2017) 1734–1750. <u>doi:10.3390/en10111734</u>
- [10] Y. Q. Mu, C. S. Wang, W. L. Zhou, L. Z. Zhou, Tensile properties of cast alloy IN625 in relation to δ phase precipitation, Acta Metall. Sin.-Engl. 32 (2018) 535–540. doi:10.1007/s40195-018-0772-y
- [11] F. Xu, Y. Lv, Y. Liu, B. Xu, P. He, Effect of heat treatment on microstructure and mechanical properties of Inconel 625 alloy fabricated by Pulsed Plasma Arc Deposition, Phys. Procedia 50 (2013) 48–54. doi:10.1016/j.phpro.2013.11.010
- [12] Y. Gao, D. Zhang, M. Cao, R. Chen, Z. Feng, R. Poprawe, J. H. Schleifenbaum, S. Ziegler, Effect of δ phase on high temperature mechanical performances of Inconel 718 fabricated with SLM process, Mat. Sci. Eng. A 767 (2019) 138327. doi:10.1016/j.msea.2019.138327
- [13] A. Chatterjee, G. Sharma, R. Tewari, J. K. Chakravartty, Investigation of the dynamic strain aging and mechanical properties in Alloy-625 with different microstructures, Metall. Mater. Trans. A 46 (2015) 1097– 1107. <u>doi:10.1007/s11661-014-2717-z</u>

- [14] V. Shankar, K. B. S. Rao, S. L. Mannan, Microstructure and mechanical properties of Inconel 625 superalloy, J. Nucl. Mater. 288 (2001) 222–232. doi:10.1016/S0022-3115(00)00723-6
- [15] Y. F. Wang, X. Z. Chen, C. C. Su, Microstructure and mechanical properties of Inconel 625 fabricated by wire-arc additive manufacturing, Surf. Coat. Technol. 374 (2019) 116–123. doi:10.1016/j.surfcoat.2019.05.079
- [16] N. Ye, M. Cheng, S. Zhang, H. Song, H. Zhou, P. Wang, Effect of δ phase on mechanical properties of GH4169 alloy at room temperature, J. Iron Steel Res. Int. 22 (2015) 752–756. doi:10.1016/S1006-706X(15)30068-6
- [17] G. A. Rao, M. Kumar, M. Srinivas, D. S. Sarma, Effect of standard heat treatment on the microstructure and mechanical properties of hot isostatically pressed superalloy inconel 718, Mat. Sci. Eng. A 355 (2013) 114–125. doi:10.1016/S0921-5093(03)00079-0
- [18] L. Chang, W. Sun, Y. Cui, F. Zhang, R. Yang, Effect of heat treatment on microstructure and mechanical properties of the hot-isostatic-pressed Inconel 718 powder compact, J. Alloys Compd. 590 (2014) 227–232. doi:10.1016/j.jallcom.2013.12.107
- [19] L. C. M. Valle, L. S. Araújo, S. B. Gabriel, J. Dille, L. H. Almeida, The effect of δ phase on the mechanical properties of an Inconel 718 superalloy, J. Mater. Eng. Perform. 22 (2013) 1512–1518. doi:10.1007/s11665-012-0433-7
- [20] International Organization for Standardization. Metallic materials – Tensile testing at elevated temperature (ISO 783: 1999, MOD); GB/T4338-2006; International Organization for Standardization, Geneva, Switzerland 1999.
- [21] Y. Q. Mu, C. S. Wang, W. L. Zhou, L. Z. Zhou, Effect of Nb on δ phase precipitation and the tensile properties in cast alloy IN625, Metals 8 (2018) 86–94. doi:10.3390/met8020086
- [22] J. B. Singh, M. Sundararaman, P. Mukhopadhyay, N. Prabhu, Effect of ternary Nb additions on the stability of the D022 structure of the Ni3V phase, Scr. Mater. 48 (2003) 261–267. doi:10.1016/S1359-6462(02)00363-9
- [23] W. C. Liu, M. Yao, Z. L. Chen, Effect of cold rolling on the precipitation behavior of δ phase in IN-CONEL 718, Metall. Mater. Trans. A 30 (1999) 31–40. doi:10.1007/s11661-999-0193-7