The Evaluation of MAR-M247 Microstructural Changes after Standard Heat Treatment and Creep Test

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Abstract

The cast nickel-base MAR-M247 superalloy has been widely used for high-temperature components. In this work, the creep and room temperature tensile behavior of MAR-M247 superalloy after dual-stage standard heat treatment is evaluated. The microstructure of heat-treated superalloy consists of a γmatrix with a γ′-eutectic, γ′strengthening cubic precipitates, and particulate, script-like carbides at the grain boundaries (GBs). Under the creep testing conditions in this study, the volume fraction and primary gamma prime size increased; the script-like MC carbide along the GB elongated, and the TCP phase formed in the vicinity of grain boundary carbides. On the other hand, during the creep test, the thin film formed around the gamma prime phase became thicker, causing an increase in the toughness of the superalloy and an expansion of the steady-state stage in the creep curve.

Keywords:MAR-M247 Superalloy, Creep, Microstructure, Heat Treatment.

1. Introduction

MAR-M247 is a typical polycrystalline nickel-based superalloy applied in investment casting[1]. The cast nickel-base polycrystalline superalloy MAR-M247 exhibits excellent resistance against high thermo-mechanics loading. It has been widely used for high-temperature components, such as advanced turbine blades and rotating parts in the aerospace industry, power generation equipment, and gas turbine engines. The balanced composition of superalloy MAR-M247 provides a great combination of good castability and mechanical properties such as strength, superior creep properties, and hot corrosion resistance at elevated temperatures up to 1034 ˚C [2-8]. These excellent mechanical properties result from the ordered intermetallic γ′-Ni_3(Al, Ti) precipitate strengthening that is enhanced by solid solution and grain boundary strengthening [2, 3].A refinement microstructure process, where fine primary carbides and γ′ precipitates in the face-centered cubic (FCC) g matrix can improve the mechanical properties, is a process that can be used for improving the lifetime and reliability of superalloys [9]. The γ′ phase strengthens Mar-M247 precipitated in the matrix [4, 5]. Theγ′ phase can prevent the gliding of dislocations, strengthening the alloy [10]. After heat treatment, MAR-M247 consists of approximately 60% semi-coherent γ′ phase, the main strengthening phase, which is in a γ matrix, a Ni-rich solid solution strengthened by cobalt, molybdenum, tungsten, and chromium [6, 7, 11, 12]. Further improvement in the strength can be achieved through solid solution hardening of the matrix, e.g., by adding refractory elements, such as Re, W, and Ta [13].

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With the addition of 0.15% carbon to the MAR-M247 superalloy, a substantial amount of particulate $M_{23}C_6$ carbide precipitates from it. When well distributed at GBs following aging heat treatment, this carbide plays a crucial role in strengthening the superalloy. It effectively pins up the GB at high temperatures, preventing the gliding of GB and thereby increasing the creep resistance [4, 5, 10].
Because the fine-grain microstructure has advantages such as refined grains, carbides, and precipitates, the fine-grain process was developed to improve the strength, creep, and fatigue life of disc rotors and turbine blades working at intermediate temperatures [14, 15]. However, since the strength and the creep resistance of the MAR-M247 superalloy increased significantly with the temperature, although the elongation and toughness dropped [10], lower elongation is closely related to the character of the carbide. In particular, the morphology of carbide strongly affects the properties of MAR-M247 superalloy. Carbide has a complex effect in a superalloy. Carbide that precipitates at GB is usually believed to be able to pin up the GB, preventing it from gliding and increasing the alloy's creep life and strength [4, 16, 17]. However, script-like MC carbide is the main reason for the low-creep elongation [18]. Since they are very brittle and are the main route of the initiation and propagation of cracks [19], the prevention and delay of the initiation and propagation of the crack are closely related to the mechanical properties of the MAR-M247 superalloy. Also, due to the high percentage of refractory elements (Ta + W + Mo), γ/γ′ eutectic and carbide form in the inter-dendritic regions during solidification [7, 11]. Creep in superalloys is very dependent on several microstructural parameters. The γ′ precipitate volume fraction,

lattice mismatch, and morphology are those of primary importance. It was reported [20] that the creep life shows a roughly linear increase with the γ′ volume fraction. The lattice mismatches between the matrix and γ′ also affects the creep strength [21].
For optimum creep strength, the γ′ particles should be very small, but this might lead to undesirable losses in ductility. The coarse primary γ′ phase particles could be a result of the micro-segregation of alloying elements during the solidification process and were challenging to refine or even dissolve during heat treatment.
Notably, carbides play a pivotal role in creep strengthening, primarily by pinning the grain boundaries and thereby preventing the grain boundaries from sliding and migrating.
In the present study, microstructural changes of MAR-M247 polycrystalline Nickel-based superalloy are evaluated after dual-stage standard heat treatment and after mechanical tests, and differences between microstructures are investigated.

2. Materials and Methods

The MAR-M247 superalloys used in this investigation were prepared by vacuum induction melting (VIM) followed by remelting in a vacuum furnace and then casting into test bars. The pouring and mold temperatures were 1480 and 1100 ˚C, respectively. The main major elemental compositions were detected by X-ray fluorescence (XRF).
Table. 1. denoted the compositions of the parent alloys. The test bar was subjected to regular heat treatment under vacuum heat treatment. Solution treatment was performed at 1200 ˚C for 2 h, and then the system was cooled to the ambient temperature in argon.
Then, the test bars were aged at 870 ˚C for 24 h and cooled to room temperature in a furnace. Standard constant load uniaxial tensile creep tests were carried out at the applied stress ranging from 230 to 300 N/mm^2 and at the testing temperatures of 975 ˚C according to ASTME139. The creep elongations were measured using a linear variable differential transducer and were continuously recorded digitally and computer-processed.
A creep test was run to the final fracture of the specimen SATEC M3 creep testers. Cylindrical creep specimens with a gauge of 28.35 mm in length and 6 mm in diameter were used in this study. Tensile tests were carried out at room temperature using an Instron mechanical testing machine according to ASTME8M.
The specimen for microscopic analysis was prepared by polishing and etching (etching solution 10ml HNO_3+ 5ml HCl). The longitudinal cross-section of the specimen was observed by scanning electron microscopy (SEM) (LYRA, Tescan) equipped with EDS microanalysis.

3. Results and Discussion

Fig. 1. shows the microstructure of the MAR-M247 superalloy after regular solutionizing heat treatment.
The MAR-M247 superalloy consisted of coarse dendritic grains microstructure (Fig. 1a). In addition, particulate MC carbides and script-like $M_{23}C_6$ formed at GBs with different sizes and morphologies are observed in the inter-dendritic regions, which also show γ/γ′ eutectic pools and thin γ′ plates (Fig. 1b). Gamma prime strengthening cubic precipitates were dispersed in the gamma matrix.
The microstructure of the analyzed MAR-M247 superalloy exhibited about 60 vol.% of the gamma prime particles in the gamma matrix (Figs. 1c and 1d).
Alloying by Mo, Co, Ti, W, and other additives leads to further solid solution and precipitation strengthening of the matrix and, thus, to an increased strengthening effect in the gamma phase. Furthermore, creep behavior could be influenced by carbides of the type $M_{23}C_6$, MC, and M_6C [22].
Due to operating temperature and applied stress, the carbides may change their size and morphology. These changes could influence the creep properties of the alloys during long-term high-temperature exposure [23]. The EDS analysis of the MC carbide phase identified the main metallic elements present as follows (in wt.%): 15.0 W, 61.5 Ti, and 9.5 C. Borides were not detected, possibly because of their very small amount and size

Table. 1. Chemical Composition of cast MAR-M247 superalloy.

Element	Ni	Cr	Co	Mo	Ti	Al	W	C	Zr	Fe	Nb	Sn	P	S
Content (wt.%)	Bal.	9.20	10.10	0.16	2.10	4.80	11.40	0.13	0.05	0.06	1.10	0.01	0.014	0.010

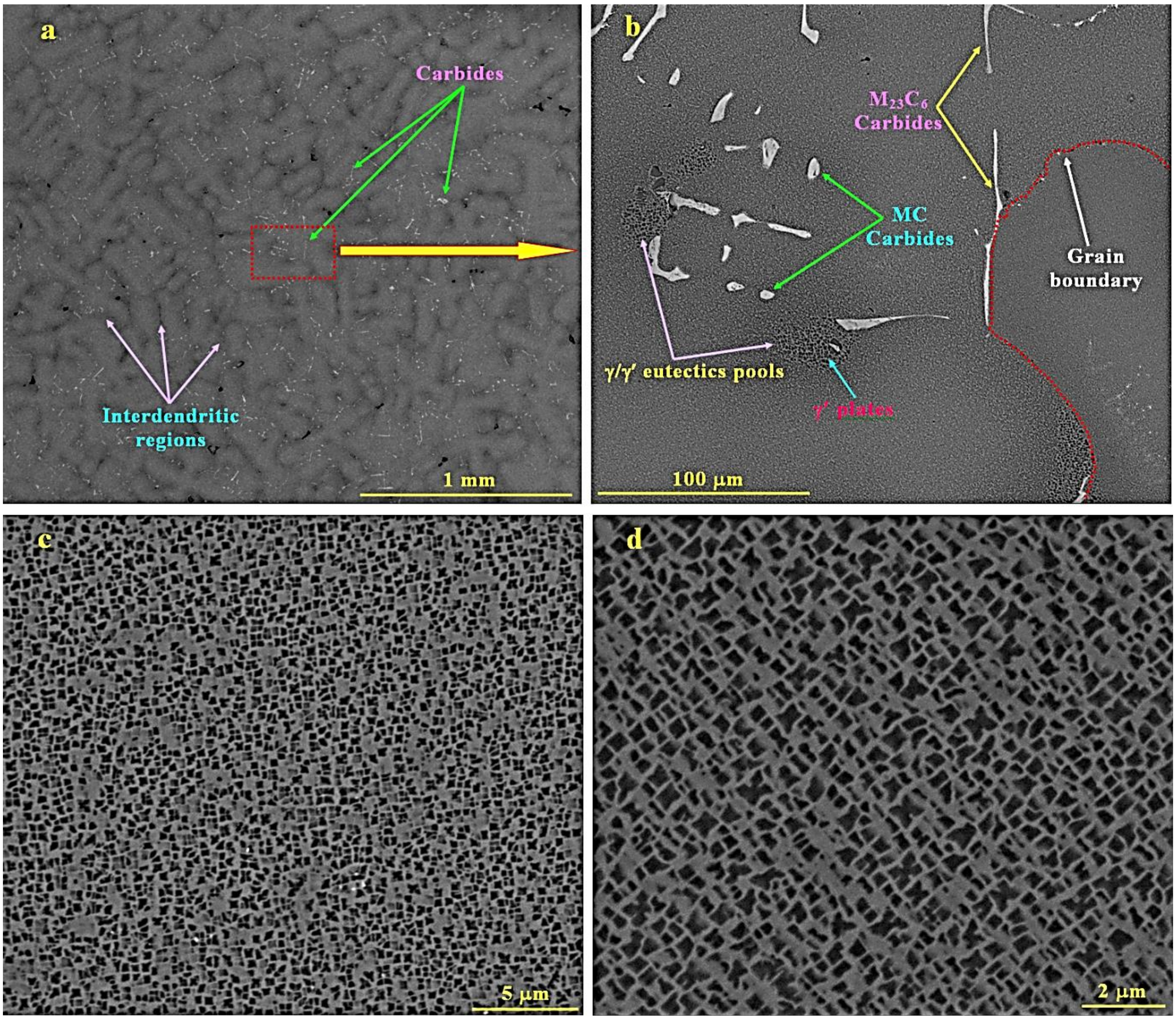

Fig. 1. Microstructure of MAR-M247 after Solutionizing: a)Coarse dendritic grain, b) γ/γ′ eutectic pools and thin γ′ plates, c, d) The gamma prime particles in the gamma matrix.

Fig. 2. shows the microstructure of the MAR-M247 superalloy after the solution and first aging heat treatment. The carbides of MAR-M247 are almost similar to those after solutionizing heat treatment in size and distribution. Furthermore, many small carbides are distributed in the matrix in clusters (Fig. 2.a). The carbides are distributed at GB in small particles. The GB can be fixed to prevent GB gliding, strengthening the GB at a high temperature.

If the script-like MC carbide is separated at GB or in the grain (Fig. 2.a), then the binding capacity of the interface between the hard and brittle carbide and the matrix is slightly weak. During the tensile and creep test, the interface of the MC carbide cannot be accommodated by gliding while the carbide is not deformed. Therefore, too many dislocations pile up around the carbide. When the strain at the interface reaches a certain value, cracks appear on the interface and propagate continuously, resulting in a slightly low elongation of MAR-M247 superalloy [4, 19]. Also, It can be seen that all of the γ-γ′ eutectic phases did not dissolve during aging (Fig. 2a).

The gamma prime precipitates were observed in dual size modification: the primary γ′ has an average size of 1 µm, and the secondary γ′ has an average size of lower than 0.5 µm. Both can effectively hinder the dislocation slip to enhance the creep-rupture life. Based on the microstructure observation in [24], the best creep strength and ductility can be achieved when the size of γ′ ranges from 0.1 to 0.5 µm. When the size of γ′is extremely small, the dislocations can easily cut or bypass these small particles. On the other hand, when the size of g¢is large, the dislocation could be accumulated in the interface of γ′and γ, where cracks can nucleate. In addition, fine secondary γ′ particles are also formed during the aging stage among coarse primary γ′ particles with an average size lower than 0.2 µm (Fig. 2d).

The SEM figures show that the thin film around the carbides at GB became thicker (Fig. 2c). A thick film is present around the script-like MC carbide within the grain interior, but the g¢-phase becomes coarse (comparison between Fig. 1.c and Fig. 2.e). MAR-M247 is a nickel-based superalloy that has very high oversaturation. During solid solution heat treatment at 1200˚C/2 h, the primary γ′-phase and the γ-γ′ eutectic structure in the grain dissolved into the matrix somewhat, forming an oversaturated solid solution. After 24 h of the first aging treatment at 870 ˚C, many $M_{23}C_6$ carbides are precipitated at GB. At the same time, the γ′-phase thickened more at the interface between the carbide $M_{23}C_6$ and the matrix (Figs 2b to 2d). After aging treatment, the thin film surrounding the $M_{23}C_6$ carbide becomes thick. The $M_{23}C_6$ carbide precipitated at GB is $Cr_{23}C_6$, which consists of a large amount of Cr. Much of the $Cr_{23}C_6$ precipitated at GB would deplete most of the Cr, and GB would become a depletion zone in Cr. The diffusion rate at GB exceeds that inside the grain because the alloy has high Al and Ti contents, so the percentage of Al and Ti at GB increases. This higher Al content leads to a larger local volume fraction of γ′ and higher diffusivity, which may explain the large γ′ particles observed in those regions [25].

The thick film around the carbides is tough, increasing the binding strength of the interface. It very efficiently delays the propagation of cracks and toughens the interface and the GB, improving its mechanical properties at high temperatures. Much of the oversaturated γ′-phase in the MAR-M247 got enough driving force and time to grow and thicken during the first aging treatment. As the γ′-phase is thickened, the yield strength and the tensile strength of the MAR-M247 superalloy decline, but the elongation is increased because the film around the carbide delays the growth of the cracks [26].

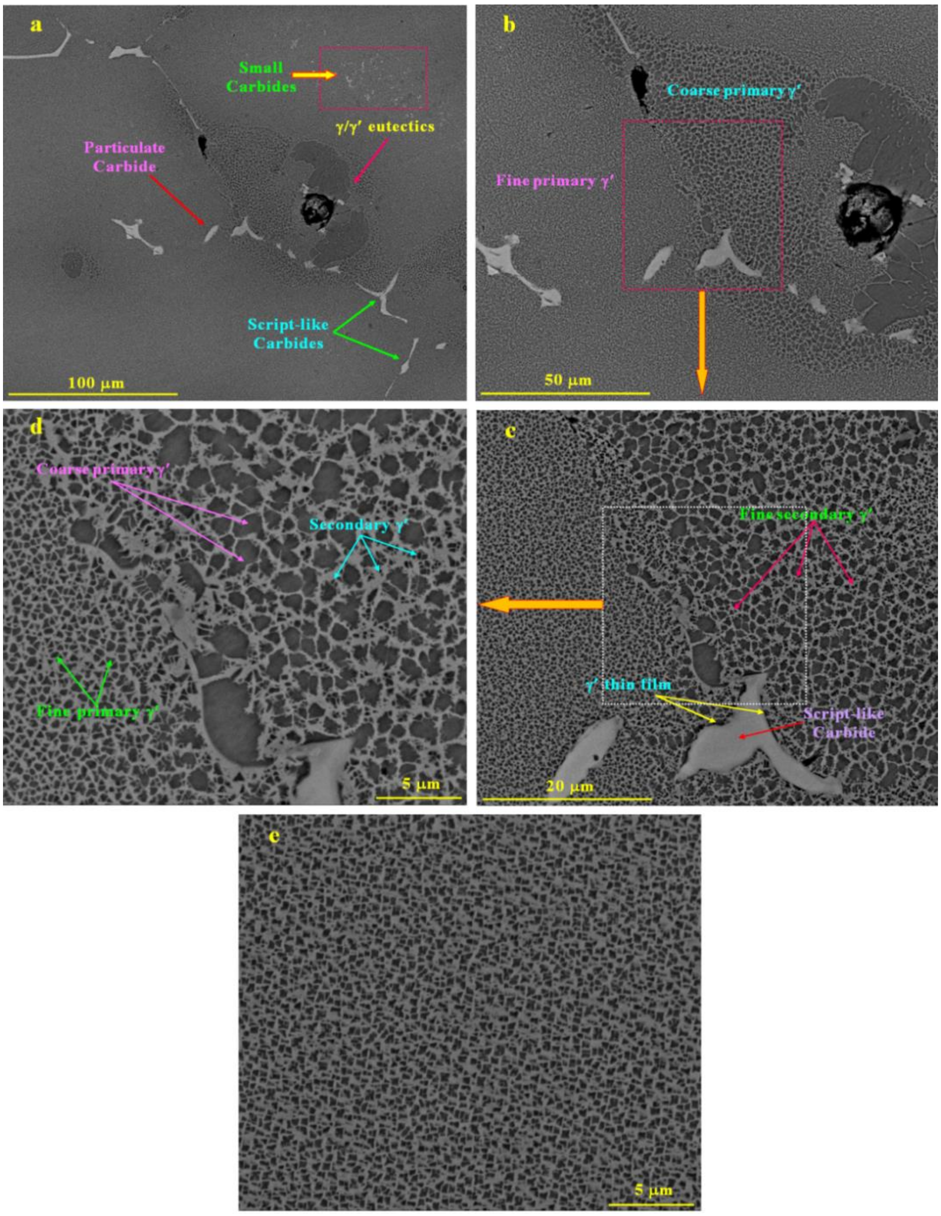

Fig. 2. a) The microstructure of different carbide types and γ-γ′ eutectic structurein MAR-M247 after aging, b) Different primary γ′ microstructure, c) The specified area in Fig. b, d) The specified area in Fig. c.

Table. 2. The results of creep and tensile test

Tensile test	**Proof Strength (MPa)**	**Tensile Strength (MPa)**	**Elongation (%)**	**Reduction in Area (%)**
	800	965	7.0	3.1
Creep test	**Applied Stress (N/mm^2)**	**Holding Time (hh.mm.ss)**	**Elongation (%)**	
	230	40:00:00	5.48	
	300	11:56:56		

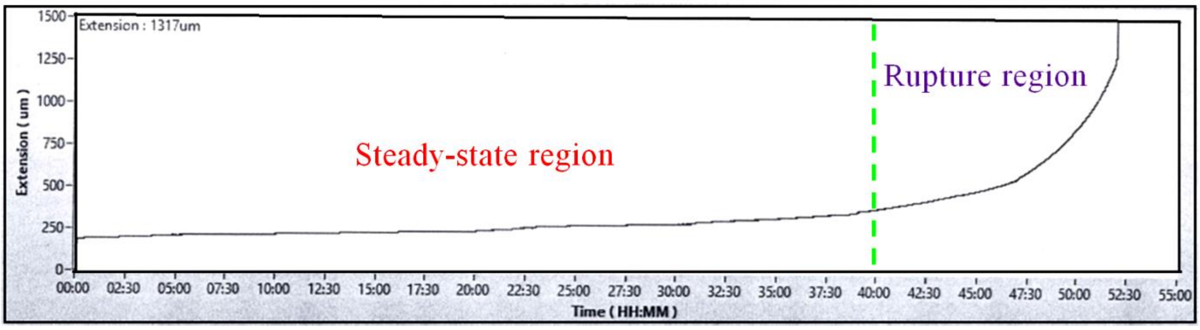

Fig. 3. Creep curves of MAR-M247 after creep test.

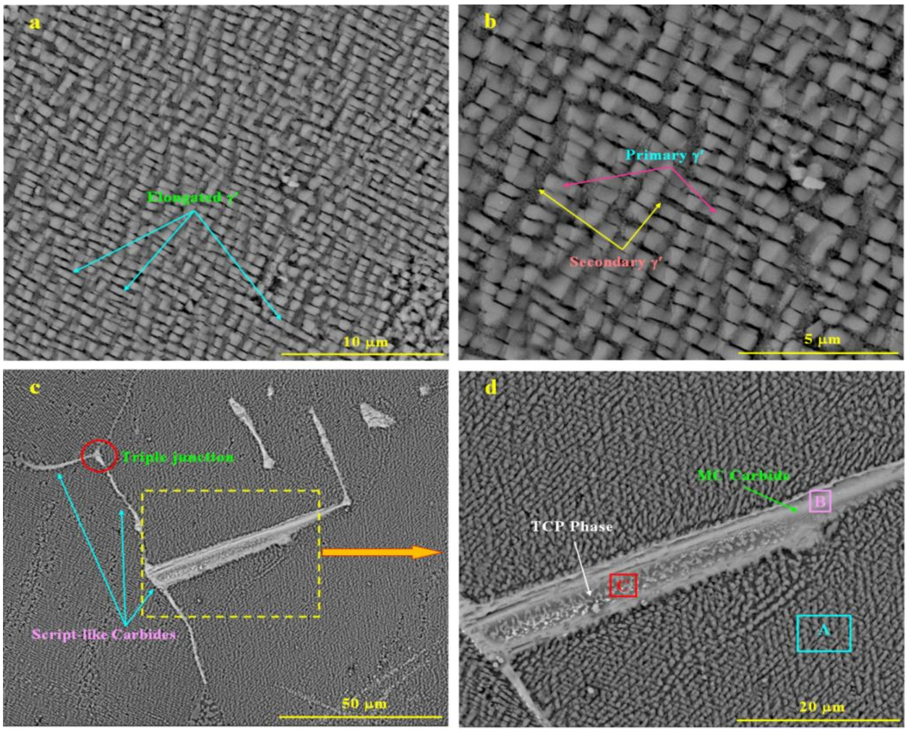

Fig. 4. Microstructure of MAR-M247 after creep test: a) The elongated primary γ′, b) The secondary γ′, c) The script-like carbide morphology, d) The specified area in Fig. c.

The γ′-film surrounds the carbide of the MAR-M247 superalloy; it can toughen the interface between the carbide and the matrix and delay the initiation and propagation of cracks during tensile and creep tests. However, the mode of fracture is on the basis of the initiation and propagation of cracks along the script-like MC carbide within the grain interior or at GB. The interface of the brittle script-like MC carbide is the main reason for the brittleness of the MAR-M247 superalloy at high temperatures and its low-creep life and elongation [26].

According to Fig. 2.e, not only does the γ′-phase become coarse, but also the γ′-phase grows, and its volume fraction increases up to 65%.

The results of the tensile and creep tests, as shown in Fig. 3. and Table. 2., respectively.

The creep curve, for instance, reveals two distinct stages: a steady-state region lasting up to 40 minutes and a rupture region extending up to 12 minutes.

The microstructure of γ′phases and carbide phases after the creep test, depicted in Fig. 4., presents an area of significant findings.

The elongation and growth of primary γ′ in the tensile direction, as well as the increase in their volume fraction (Fig. 4.a and Fig. 4.b), highlight the potential impact of this research on the field of materials science and engineering.
Elongation can be efficiently improved in this high-temperature tensile and creep test. The steady-state creep rate of a superalloy is strongly related to the γ′-phase's volume fraction and size.
A higher volume fraction and smaller particle size of γ′-phase correspond to a lower steady-state creep rate. In this experiment, the γ′-phase becomes thick after the solution and first aging heat treatment, seemingly increasing the steady-state creep rate [27].Since the ordered structure of the γ′-phase includes powerful atomic bonding forces, when dislocation passes the γ′-phase, the dislocation must overcome the bonding of the adjacent atom, generating an anti-phase boundary, which more powerfully prevents the gliding of dislocations than the matrix.
Although the strength of the thick film is higher than the matrix, a dislocation can still glide on its slip plane. Hence, some plasticity is evident, and the γ′-phase does not pile up dislocations like the script-like MC carbide, which can make the alloy brittle.
The script-like carbides became thicker and elongated (Fig. 4c).
According to EDS results (Fig. 5.) of the specified zone in Fig. 4.d, The needle-like precipitates adjacent to the MC carbides could be TCP (topological closed packed phase), which has a deleterious effect on creep ductility as a place for creep damage nucleation [28]. During creep, the stress concentration of dislocations generated on the script-like MC carbide interface can be relaxed by altering the carbide thick-film's plasticity, toughening the MAR-M247 superalloy. γ′ is invariably deformed by fine slip in superalloy, revealing that when the γ′-phase generates a film around carbide by fine slip, it can not only effectively accommodate the stress state but also effectively delay the initiation and propagation of the crack.
The thick film around carbide can facilitate crack blunting, influencing the crack propagation rate during creep and improving ductility.

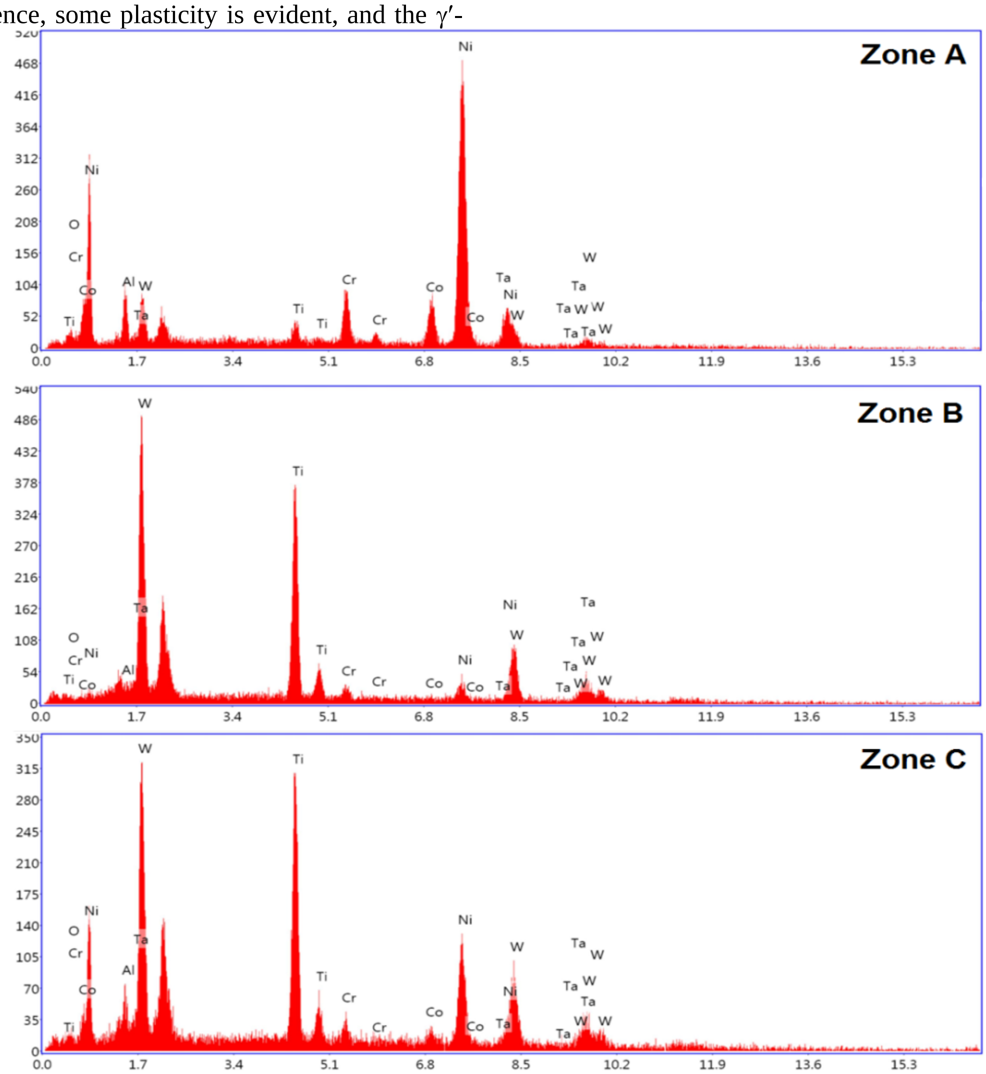

Fig. 5. EDS results of the specified zones in Fig. 4d.

4. Conclusion

In the present study, mAR-M247 polycrystalline Nickel-based superalloy microstructural changes are evaluated after dual-stage standard heat treatment and mechanical tests, and differences between microstructures are investigated. The results are as follows:

1. The microstructure of heat-treated superalloy consists of a γ matrix with a γ-γ′ eutectic, γ′strengthening cubic precipitates, particulate, and script-like carbides at the GBs.
2. During the creep test, the volume fraction and primary gamma prime size increased; the script-like MC carbide along the GB elongated, and the TCP phase formed near GB carbides.
3. During the creep test, the thin film formed around the gamma prime phase became thicker, causing an increase in the ductility of the superalloy and an expansion of the steady-state stage in the creep curve.

References

[1] Bor HY, Wei CN, Jeng RR, Ko PY, Elucidating the Effects of Solution and Double Ageing Treatment on the Mechanical Properties and Toughness of MAR-M247 Superalloy at High Temperature. Mater. Chem. Phys.2008; 109(2-3):334–341.
[2] Nathal MV, Maier RD, Ebert LJ, The influence of cobalt on the tensile and stress-rupture properties of the nickel-base superalloy mar-m247. Metall. Trans. A 1982; 13:1767–1774.
[3] Nathal MV, Maier RD, Ebert LJ, The Influence of Cobalt on the Microstructure of the Nickel-Base Superalloy MAR-M247. Metall. Trans. A 1982; 13:1775–1783.
[4] Bor HY, Chao CG, Ma CY, The effects of Mg microaddition on the mechanical behavior and fracture mechanism of MAR-M247 superalloy at elevated temperatures. Metall. Trans. A 1999; 30:551–561.
[5] Bor HY, Chao CG, Ma CY, The influence of Mg on creep properties and fracture behaviors of Mar-M247 superalloy under 1255 K/200 MPa. Metall. Trans. A 2000; 31:1365–1373.
[6] Kattus JR, MAR M 247—Aerospace Structural Metals Handbook. West Lafayette: Purdue Research Foundation; 1999.
[7] Szczotok A, Szala J, Cwajna J, Hetmanczyk M, Selection of Etching Methods of Primary Carbides in MAR-M247 Nickel-Base Superalloy for Computer-Aided Quantitative Metallography. Mater. Charact. 2006; 56(4-5):348–354.
[8] Bor HY, Chao CG, Ma CY, The Influence of Magnesium on Carbide Characteristics and Creep Behavior of the MAR-M247 Superalloy. Scr. Mater. 1998; 38(2):329–335.
[9] Murakurrno T, Kobayashi T, Koizumi Y, Harada H, Creep Behavior of Ni-base Single Crystal Superalloy with Various γ′Volume Fraction. Acta Mater. 2004; 52(12):3737–3744.
[10] Allied-Signal Aerospace Company: Engine Materials Specification 55447, Castings, Investment, MAR-M247, 1988.
[11] Bhaumik SK, BhaskaranTA, Rangaraju R, Wenkataswamy MA, Parameswara MA, Krishnan RV, Failure of Turbine Rotor Blisk of an Aircraft Engine. Eng. Fail. Anal. 2002; 9(3):287–301.
[12] Zeisler-Mashl KL, Pletka BJ, Segregation During Solidification in the MAR-M247 system, International Symposium on Superalloys, Warrendale, PA, 1992; 175–184.
[13] Ruusing J, Wanderka N, Czubayko U, Naundorf V, Mukherji D, Rosler J, Rhenium distribution in the matrix and near the particle–matrix interface in a model Ni–Al–Ta–Re superalloy. Scr. Meter. 2002; 46(3):235–240.
[14] Wei CN, Bor HY, Chang L, Effect of Hot Isostatic Pressing on Microstructure and Mechanical Properties of CM-681LC Nickel-Base Superalloy Using Microcast. Mater. Trans. 2008; 49:193–201.
[15] Huang HE, Koo CH, Effect of Zirconium on Microstructure and Mechanical Properties of Cast Fine-Grain CM 247 LC Superalloy. Mater. Trans. 2004; 45(2):554–561.
[16] Liu LR, Jin T, Zhao NR, Sun XF, Guan HR, Hu ZQ, Formation of carbides and their effects on stress rupture of a Ni-base single crystal superalloy. Mater. Sci. Eng. A 2003; 361(1-2):191–197.
[17] Liu LR, Jin T, Zhao NR, Wang ZH, Sun XF, Guan HR, ZQ Hu, Effect of carbon addition on the creep properties in a Ni-based single crystal superalloy. Mater. Sci. Eng. A 2004; 385(1-2):105–112.
[18] Kotval PS, Venables JD, Calder RW, The role of hafnium in modifying the microstructure of cast nickel-base superalloys. Metall. Trans. 1972; 3:453–458.
[19] Gell M, Kortovich CS, Bricknell RH, Kent WB, Radavich JF (Eds.), Proceedings of Superalloys, AIME, Warrendale, PA, 1984; 43–52.
[20] Floreen S, Superalloys Mechanical behaviour, John Wiley & Sons, New York, 1987; 241–262.
[21] Coakley J, Lass EA, Ma D, Frost M, Stone HJ, Seidman DN, Dunand DC, Lattice parameter misfit evolution during creep of a cobalt-based superalloy single crystal with cuboidal and rafted gamma-prime microstructures. Acta Mater. 2017; 136:118-125.
[22] Chen J, Lee JH, Jo CY, Choe SJ, Lee YT, MC carbide formation in directionally solidified MAR-M247 LC superalloy. Mater. Sci. Eng. A, 1998; 247(1-2):113–125.
[23] Tsai YL, Wang SF, Bor HY, Hsu YF, Effects of alloy elements on microstructure and creep properties of fine-grained nickel-based superalloys at moderate temperatures. Mat. Sci.Eng. A 2013; 571:155–160.

[24] Sims CT, Stoloff NS, Hagel WC, Superalloys II, John Wiley & Sons, New York, 1987; 255–256.
[25] Baldan R, Rocha RLP, Tomasiello RB, Nunes CA, Silva Costa AM, Barboza MJR, Coelho GC, Rosenthal R, Solutioning and Aging of MAR-M247 Nickel-Based Superalloy, J. Mater. Eng. Perform. 2013;22:2574–2579.
[26] Bor HY, Wei CN, Jeng RR, Ko PY, Elucidating the effects of solution and double aging treatment on the mechanical properties and toughness of MAR-M247 superalloy at high temperature. Mater. Chem. Phys. 2008; 109(2-3):334–341.
[27] Koul AK, Wallace W, A note on the microstructural dependence of creep strength in Inconel 700. Met. Trans. A 1982; 13:673–675.
[28]Liao JH, Bor HY, Wei CN, Chao CG, Liu TF, Influence of microstructure and its evolution on the mechanical behavior of modified MAR-M247 fine-grain superalloys at 871 °C. Mater. Sci. Eng. A 2012; 539:93–100.