Short Communication

The characteristics of $\mu$ phase precipitated during 720 °C long-term aging in alloy 617B

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ABSTRACT

In this work, the precipitation of $\mu$ phase is detected in alloy 617B during 720 °C aging for 10,000 h. The lattice parameters of $\mu$ phase is determined as $a=0.4720-0.4760$ nm and $c=2.5394-2.5656$ nm in this work. The $\mu$ phase in alloy 617B is mainly composed of Mo and Cr, in accompany with Ni and Co. The chemical formula of $\mu$ phase in samples aged for 5000 h and 10000 h is $(Ni_{0.031}Co_{0.884}Cr_{0.885})_{2.34}Mo$ and $(Ni_{0.030}Co_{0.991}Cr_{0.879})_{2.3}Mo$, respectively. The precipitation of $\mu$ phase competes with $M_23C_6$ carbide for Cr and Mo elements.

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1. Introduction

Alloy 617B is a typical nickel-base superalloy candidate for advanced ultra-supercritical (A-USC) power plants. It is expected to operate for 100,000 h without apparent microstructure and property degeneration. Hence, the microstructure stability is of vital importance. Large amount of alloying elements are added in alloy 617B to get excellent high temperature property, which also makes the microstructure evolution during thermal aging complicated.

There have been a number of reports about phase evolution of alloy 617B during thermal aging and the major phases are $\gamma'$ phase, MC, $M_6C$ and $M_23C_6$ type carbide [1,2]. No topologically close-packed (TCP) phases, such as $\mu$, $\sigma$ and $\chi$ phase, are reported in alloy 617B [3,4]. However, Mo content in alloy 617B is as high as 9%, which is beneficial for precipitation of $\mu$ phase. Furthermore, $\mu$ phase has been detected in other nickel-base superalloy with comparable Mo content. The precipitation behavior of $\mu$ phase is closely related with chemical composition of alloy [5] and the effect on mechanical properties is complicated [5-7].

In the authors’ previous work some phase that is not a carbide is detected on grain boundary in alloy 617B during 10,000 h aging [8]. In this work, this phase is confirmed to be $\mu$ phase by micro-chemical phase analysis. Each phase in alloy 617B is chemically extracted separately and the weight and chemical composition are determined precisely. The precipitation reason of $\mu$ phase is explained based on element redistribution. The result of this work can give some references for material selection in A-USC candidates.

2. Experimental material and procedures

Alloy 617B used in this work is gotten from finished pipe in solution annealing state. The chemical composition is listed in Table 1. Long-term aging was carried out at 720 °C. Samples
for microstructure observation were electrolitically polished with a solution of 20% CH₃OH and 80% H₂SO₄, and electrolitically etched with a solution of 15 g CrO₃ + 10 ml H₂SO₄ + 170 ml H₃PO₄. Thermo-dynamic calculation was carried out by Thermo-calc software (a commercial thermodynamic computer software) with Ni-Data v5 database.

The quantitative weight and element concentration analysis for precipitates were carried out by micro-chemical phase analysis. The γ precipitates were electrolitically extracted by an aqueous solution 1% (NH₄)₂SO₄ + 1% C₆H₅O₂. The carbides were electrolitically extracted in a methanol solution containing 5% HCl, 5% C₆H₅O₂ and 1% C₆H₅O₂. The extracted precipitates were washed by alcohol solution of 1% C₆H₅O₂, aqueous solution of 1% C₆H₅O₂ and distilled water in a sequence and then dried. The crystallographic structure of precipitates was determined by X'Pert MPD X-ray diffractometer equipped with Cu Kα radiation. The amount of μ phase is very limited. Hence, sample was electrolitically extracted by methanol solution containing 5% HCl, 5% C₆H₅O₂ and 1% C₆H₅O₂ in a large scale and then boiled by 80 ml C₆H₅OH + 20 ml HCl solution to dissolve parts of M₇C₃ carbide. The remained part is used to identify μ phase. Chemical compositions of precipitates were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES).

### Table 1 - Chemical composition (wt%) of Alloy 617B.

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>C</th>
<th>Zr</th>
<th>Nb</th>
<th>B</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>22.13</td>
<td>12.14</td>
<td>9.05</td>
<td>1.44</td>
<td>0.44</td>
<td>0.047</td>
<td>0.08</td>
<td>0.08</td>
<td>0.004</td>
<td>Bal</td>
</tr>
</tbody>
</table>

#### 3. Results and discussion

Fig. 1 is morphology evolution of grain boundary phases in alloy 617B during 720 °C aging. The white phase on grain boundary is M₇C₃ carbide, and it coarsens with the increase of aging time. When aging time increases to 4000 h, some gray phases in the secondary electron image can be seen on grain boundaries. More such gray phases can be detected with increasing time. Energy dispersive spectrometer (EDS) analysis indicates that it is mainly composed of Cr, Co, Ni and Mo elements. The typical composition of this phase corresponds with thermo-dynamic calculation result of μ phase as compared in Fig. 1.

The XRD result for detection of μ phase by micro-chemical phase analysis method introduced above is shown in Fig. 2 and μ phase can be detected, further confirming the gray phase on grain boundary in Fig. 1 is μ phase. The Miller indices of μ phase are also marked in the XRD result. Though precipitation of μ phase is calculated by equilibrium phase diagram [9], however, it has not been detected in previous work [3,4]. This is because the amount of μ phase is relatively low and it begins to precipitate after long-term thermal aging, which makes the detection difficult. However, μ phase is easy to precipitate in...
superalloy with high content of Mo. The Mo concentration in alloy 617B is up to 0.05%, which makes precipitation of μ phase possible.

Micro-chemical phase analysis confirms that μ phase is mainly made up of Cr, Mo accompanied by Ni, Co (Table 2), and Cr accounts for half of the weight of μ phase. μ phase is detected by micro-chemical phase analysis in sample aged for 5000 h, a little later than microstructure observation. This is because enough amount is needed for detection. μ phase has a hexagonal close-packed structure. The lattice parameter is determined as $a = 0.4720–0.4760$ nm and $c = 2.5394–2.5656$ nm in this work. The chemical formula in samples aged for 5000 h and 10000 h is $(Ni_{0.031}Co_{0.084}Cr_{0.885})_{2.34}Mo$ and $(Ni_{0.030}Co_{0.091}Cr_{0.879})_{2.30}Mo$, respectively. Atom fraction of Cr increases slightly with increasing time, and the existence of Ni is beneficial for the stability of μ phase [5].

More accurate information of phase evolution got by micro-chemical phase analysis is shown in Fig. 3. The amount of carbides increases in the first 500 h and then stays stable with some fluctuations. The total amount of MC carbide is around 0.03%, and it is about 0.8% for $M_{23}C_6$ carbide. Furthermore, μ phase can be detected after 5000 h aging with a weight fraction of 0.133%. The weight fraction of μ phase increases to 0.265% after 10000 h aging. Chemical compositions of phases are not unchangeable during aging. The $M_{23}C_6$ carbide in alloy 617B is mainly composed of Cr, Mo and C elements, and the amount of elements increases during coarsening (Fig. 3(b)). Coarsening of phase is an element diffusion controlled process. In the coarsening of $M_{23}C_6$ carbide, Cr and Mo elements diffuse to the surrounding of $M_{23}C_6$ carbide and then transfer into it. However, it is interesting to find that there is an apparent decrease in the amount of carbides and carbide formation elements after 5000 h aging, and precipitation of μ phase is detected at the same time. This can be attributed to the fact that Mo and Cr elements are major elements in μ phase. Previous work has confirmed that μ phase prefer to precipitate at existing Mo-rich carbide on grain boundaries [5]. The coarsening rate of $M_{23}C_6$ carbide slows down gradually with prolonging time, so the demanded amount of Cr and Mo decreases. As a result, abundant Cr and Mo enriches around $M_{23}C_6$ carbide. When the concentration is high enough, μ phase begins to precipitate at the interface of $M_{23}C_6$ carbide.

<table>
<thead>
<tr>
<th>Time/h</th>
<th>Ni</th>
<th>Co</th>
<th>Cr</th>
<th>Mo</th>
<th>Σ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight fraction of elements in phase account for alloy total weight</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5000</td>
<td>0.0026</td>
<td>0.0070</td>
<td>0.065</td>
<td>0.058</td>
<td>0.133</td>
</tr>
<tr>
<td>10000</td>
<td>0.0050</td>
<td>0.015</td>
<td>0.128</td>
<td>0.117</td>
<td>0.265</td>
</tr>
<tr>
<td>Atom fraction of elements in phase account for phase total weight</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5000</td>
<td>2.18</td>
<td>5.90</td>
<td>61.97</td>
<td>29.94</td>
<td>99.99</td>
</tr>
<tr>
<td>10000</td>
<td>2.11</td>
<td>6.32</td>
<td>61.24</td>
<td>30.32</td>
<td>99.99</td>
</tr>
</tbody>
</table>

Fig. 2 – XRD result for detection of μ phase.

Fig. 3 – (a) Weight fraction variation of phases, and (b) weight fraction variation of elements in $M_{23}C_6$ carbide accounts for total weight of alloy.
The electron micro probe analyzer (EPMA) result further confirms that there is apparent enrichment of Cr and Mo on grain boundary (Fig. 4), which facilitates the precipitation of $\mu$ phase. The white particle in the backscattered electron (BSE) image is $\mu$ phase and it precipitates just besides $M_{23}C_6$ carbide. The precipitation of $\mu$ phase competes with $M_{23}C_6$ carbide for Cr and Mo. Hence, the amount of $M_{23}C_6$ carbide decreases when $\mu$ phase appears. Afterwards, supplementary precipitation of $M_{23}C_6$ carbide takes place benefitting from the abundant element supply from matrix and weight fraction increases again. Meanwhile, $\mu$ phase proceeds to precipitate and weight fraction increases.

In addition, it should be pointed out that alloy 617B is a solution strengthened alloy. The precipitation of $\mu$ phase derives Cr, Mo and Co elements from the matrix, which will certainly degenerate the solution strengthen effect. Furthermore, $\mu$ phase decorates between $M_{23}C_6$ carbides and the precipitate on grain boundary is more continuous. The continuous precipitation is not benefit for mechanical properties, such as fatigue crack propagation, impact toughness and so on [10]. The $\mu$ phase keeps precipitating and coarsening with increasing time and more $\mu$ phase will show up if alloy 617 serves for 100 000 h. As a result, enough attention should be paid on the precipitation of $\mu$ phase during long term aging in alloy 617B.

4. Conclusion

The precipitation of $\mu$ phase in alloy 617B during 720 °C aging is detected by microstructure observation at 4000 h and confirmed by micro-chemical phase analysis at 5000 h. It is mainly composed of Cr, Mo in accompany with Ni, Co. The lattice parameter is $a = 0.4770–0.4760$ nm and $c = 2.5394–2.5656$ nm. The precipitation of $\mu$ phase competes with $M_{23}C_6$ carbide for Cr and Mo elements, and may influence the solution strengthened effect and mechanical properties.

**Conflicts of interest**

The authors declare no conflicts of interest.

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