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The effect of elemental vaporization and redistribution during sub-solvus recrystallization of a single crystal superalloy

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ABSTRACT

The elemental vaporization and redistribution effect during sub-solvus recrystallization (1100 °C) of a second-generation single crystal superalloy DD6 containing 2 wt% Re has been investigated in this work. For fine polished specimens free from selective chemical erosion effect, accurate elemental energy dispersive spectroscopy (EDS) analysis was taken from the surface to the inner original matrix and across the boundary of the recrystallization reaction front. The results revealed that the vaporization induced elemental depletion of Ni and Al had occurred in the top surface layer. This would lead to the destabilization of the γ phase in the recrystallized aggregates, facilitating the final precipitation of refractory-rich P phase.

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1. Introduction

Nickel-based superalloys are developed as structural materials to withstand extreme conditions of temperature and loading, of which the microstructural stability during service is always a long-lasting issue [1,2]. Until now, a comprehensive understanding of related aspects of recrystallization in nickel-based superalloys has already been gained, including the effects of strain [3], deformation temperature [4,5], deformation orientation [6], annealing conditions [7,8], and the interaction with secondary phases [9,10]. Recently, the traditional view that, during high-temperature solution heat treatment (1360 °C) of superalloys, the thermodynamic system is closed for mass balance considerations has been demonstrated to be incorrect [11]. On the other hand, significant higher levels of refractory elements are added to most modern single crystal superalloys [1], which causes an potential promotion of phase instabilities that lead to the formation of topologically close-packed (TCP) phases during exposure to elevated temperatures. These TCP phases can serve as damage accumulation sites that initiate or accelerate crack propagation, which ultimately cause the creep strength of the material to decrease [12]. Accordingly, for more reliable manufacturing capability, the present study is aimed at providing more detailed insights with respect to better understanding of elemental vaporization and redistribution induced P phase formation during sub-solvus recrystallization of the single crystal superalloys.

2. Material and methods

The second-generation nickel-based single crystal superalloy DD6 [13] with tensile and creep rupture properties comparable to René N5 and CMSX-4, was used in this study. Single crystal plate of the alloy, 160 mm in length, 24 mm in width and 12 mm in thickness was directionally solidified in Bridgman furnaces. Details of DD6 preparation have been described previously [13]. Longitudinal orientation of the plate is within 15 °deviating from [001] direction. In order to avoid additional surface deformation, electric discharge machine (EDM) was used to cut the plates into small pieces (11 mm × 11 mm × 8 mm), with the 11 mm × 11 mm plane parallel to the [001] direction. The single crystal pieces were grit blasted at a pressure of 0.25 MPa on the 11 mm × 11 mm plane for 30 s. SiO_2 spheres with a radius of 75 μm were used. The grit blasted pieces were tubed in vacuum silica glass to avoid oxidation, and then subjected to heat treatment at 1100 °C for 16 h, followed by air cooling. For elemental energy dispersive spectroscopy (EDS) analysis on the specimens free from chemical etching, the metallographic specimens were carefully grinded with fine metallographic grinding papers (1000–5000 grit) and then polished using the diamond paste with diameters ranging from 3 μm to 0.2 μm. The EDS line-scans and area mapping were obtained on a Carl-Zeiss Auriga field

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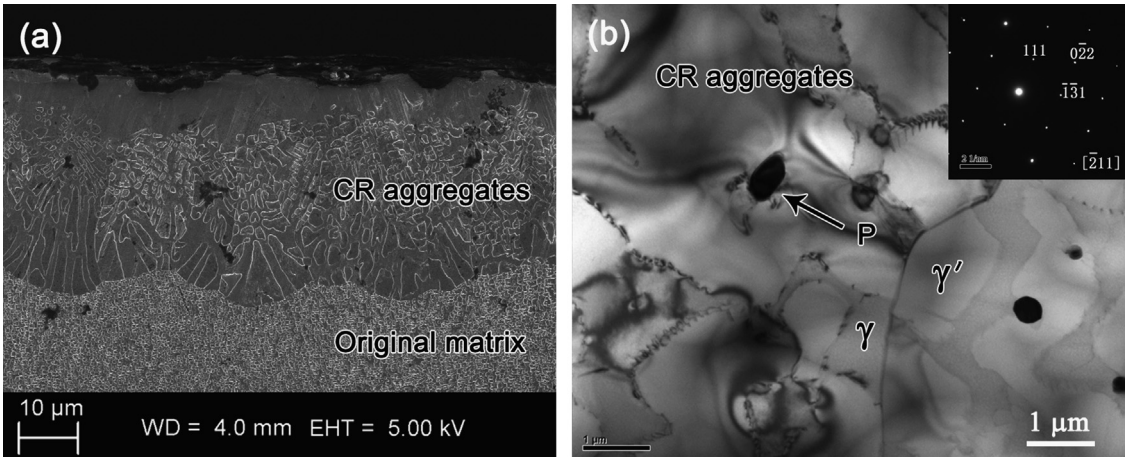

Fig. 1. Typical secondary electron SEM image (a) and bright field TEM image (b) with an inserted selected area electron diffraction pattern of P phase (the inset) of the chemical etched specimens after annealed at 1100 °C for 16 h.

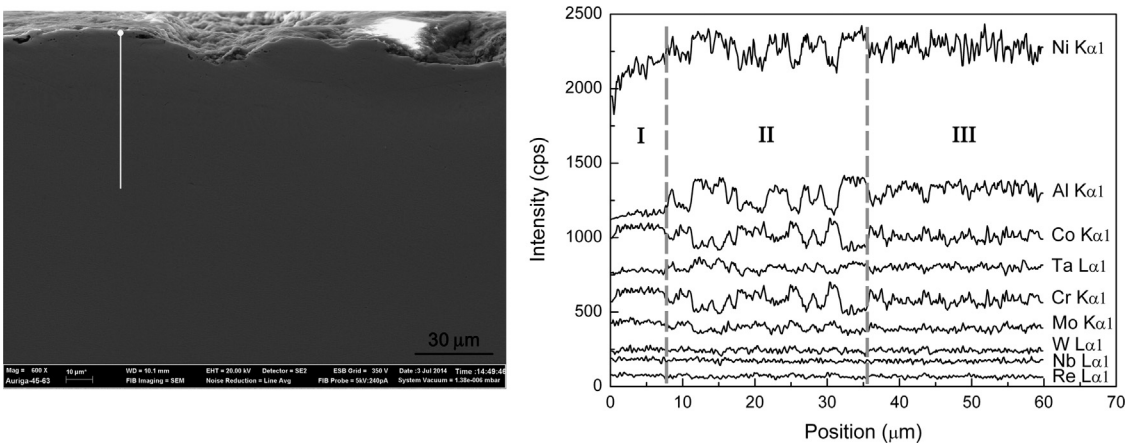

Fig. 2. EDS line-scan taken from the surface into the inner OM region of the polished specimen.

emission scanning electron microscope (SEM) instrument equipped with an Oxford X-Max^N 50 EDS. In addition, typical secondary electron SEM image of the specimen after polishing and chemical etching in the reagent consisting of 20 g $CuSO_4$, 100 ml HCl, and 100 ml H_2O, and bright field transmission electron microscopy (TEM) image of the specimen after electropolishing with 8% perchloric acid solution in ethanol at 25 V and −25 °C, were obtained respectively on the Carl-Zeiss Auriga field emission SEM and a FEI G^2 Tecnai 20 TEM.

3. Results and discussion

Typical secondary electron SEM image of the specimen after chemical etching and bright field TEM image of the specimen after electropolishing are shown in Fig. 1. As shown in Fig. 1(a), the growth of the cellular recrystallization (CR) aggregates is demarcated by the boundary ~35.5 μm away from the surface. Clear cellular precipitation with a phase mixture of γ, γ′ and the refractory-rich precipitates P ($(WMoReTaNb)_{43.7}Cr_{12.7}(NiCo)_{49.6}$) [14] (Fig. 1(b)) occurs within the surface layer due to the thermal–mechanical prerequisite (i.e. sub-solvus heat treatment at 1100 °C for 16 h after surface grit-blasting), which is markedly absent in the inner original matrix (OM) remote from the surface. More detailed information can be seen in Refs. [14,15].

For accurate elemental EDS analysis free from selective chemical erosion, the metallographic specimens were carefully grinded with fine metallographic grinding papers (1000–5000 grit) and then polished using the diamond paste with diameters ranging from 3 μm to 0.2 μm. As shown in Fig. 2, the EDS line-scan traversing from the surface into the inner OM region confirms that dominant elemental vaporization (~7.5 μm thick with depletion of Ni and Al) has occurred within the top surface layer (District I), leading to relative enrichment of Co, Cr and Mo. It is notable that compared with the inner OM region > 35.5 μm away (District III), the more obvious elemental fluctuation and fluctuation intervals within the region from 7.5 μm to 35.5 μm (District II) correspond to the elemental difference between the coarsened γ and γ′ in the CR aggregates. In addition, similar to the thickness of the CR region (District II), the thickness of the top surface layer (District I) also strongly depended on annealing time (Fig. S1).

The fact is further reinforced by the EDS area mapping taken in the vicinity of the surface of the polished specimen. The mapping images of elements Ni, Al, Co, Cr, Mo, Ta, W and Re are shown in Fig. 3. Variations can be observed in the image contrast. It exhibits in the top surface layer with a thickness of ~7.5 μm that, strong depletion of Ni and Al, together with relative enrichment of Co, Cr, Mo and Re, has occurred. It should also be noted that the elements Ta and W distribute evenly across the mapping area, indicating no obvious partitioning preference.

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