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Correlative characterization on microstructure evolution of Ni-based K403 alloy during thermal exposure

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ABSTRACT

The microstructure evolution of the K403 Ni-based superalloy as a function of thermal exposure temperature and time was investigated using correlative characterization, including a combination of scanning electron microscopy, electron backscatter diffraction, transmission electron microscopy, high-angle angular dark-field scanning transmission electron microscopy imaging and electron energy loss spectroscopy. The as-cast microstructure of the K403 alloy shows a typical dendritic structure and consists of a γ solid solution, γ' phase, $(\gamma + \gamma')$ eutectic phase and a metal carbide (MC) phase. The solutes of Ni and Ti are enriched in the γ solid solution, while, the solutes of Cr and Co are enriched in the γ' phase. The morphology of the γ' phase is nearly cubic. After thermal exposure at 800 °C or 950 °C for up to 200 h, the typical dendritic structure and a similar solute segregation behavior were still observed. However, the MC carbides were partially decomposed and further transformed to the M_6C phase and $M_{23}C_6$ phase. Furthermore, the size of γ' was increased from 302.88 ± 20.49 nm to 374.75 ± 29.76 nm (800 °C, 50 h) and 751.73 ± 123.14 nm (950 °C, 50 h), respectively. The morphology of γ' was changed from cubic to triangular or round. Clearly, there is a significant coarsening of γ' during thermal exposure. A topologically close-packed (TCP) σ phase was observed after thermal exposure at 800 °C for 100 h or 950 °C for 50 h. More interestingly, an in-situ phase transformation of the σ phase to other TCP phase (i.e. P phase) was also observed after thermal exposure at 950 °C for 50 h. The formation and transformation of carbide (i.e. M_6C phase, $M_{23}C_6$ phase) and TCP phases (i.e. σ phase, P phase) is proposed to be a diffusion-controlled process and can be attributed to the solute diffusion during thermal exposure. The present investigation provides a better understanding on the high temperature performance of the K403 Ni-based superalloy, which is essential to predict the failure and thereby enhance the reliability and service life of the K403 Ni-based superalloy.

1. Introduction

The K403 alloy is a Ni-Cr based cast superalloy: alloying with Co, W, Mo, Al, and Ti [1]. Compared with other Ni based cast superalloys (e.g. CSMX4 [2]), the K403 alloy does not contain expensive Ta and Re. The concentration of Co (5.41 wt%) is also less than that (9.0 wt%) of CSMX4 [2]. Although the concentrations of Cr (11.32 wt %) and Mo (4.24 wt%) are higher than that (6.50 wt% for Cr and

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0.60 wt% for Co) of CSMX4, the K403 alloy is believed to be a cheaper alternative available without sacrificing important properties. More importantly, the K403 alloy has an excellent castability and can be cast using different casting technologies, i.e. investment casting under vacuum. The K403 alloy has therefore been widely used in various fields, including as gas turbine guide vanes with working temperatures below 1000 °C, turbine rotor blades with working temperatures below 900 °C and isothermal forging forming dies to deform otherwise hard-to-deform materials such as Ti alloys. Practical application of the K403 alloy generally occurs under very harsh working conditions at high temperatures and under heavy load bearings. Under these conditions, the high temperature thermal exposure is one of the most important factors affecting the microstructure evolution. During high temperature thermal exposure, there may be significant structural changes including the aggregation and the growth of strengthening phases (γ' precipitates) [3–14], precipitation and transformation of carbides [15,16], and precipitation of topologically close-packed (TCP) phases [2,16].

In terms of the strengthening phases (γ' precipitates), it is generally accepted that the coarsening of γ' precipitates is a dominant factor affecting the alloy's properties [3–14]. The coarsening rate of the γ' precipitates has been predicted by the well-known Lifshitz, Slyozov and Wagner (LSW) theory [4–7]. However, there is still a lack of detailed investigations on the microstructure evolution of the γ' phase in K403 alloy [1], in particular to the solute segregation behaviors during thermal exposure of the K403 alloy. A better understanding of the microstructure evolution mechanisms of the γ' phase in K403 alloy is, therefore, still of great necessity to predict the service life and reliability of K403 alloy.

In terms of the carbides and TCP phases, various carbides and TCP phases have been reported [2,15,16]. It is generally accepted that carbides and TCP phases are composed of Ni, Cr, Mo, Co, W and Re. However, the primary function of these elements in the K403 alloy is improving the resistance to creep. The precipitation of carbides and TCP phases will definitely deplete these elements within the matrix and thereby reduce their solid strengthening effect. Moreover, the precipitation of carbides and TCP phases is very often related to the formation of voids, which may potentially act as initiation sites for fracture. Therefore, the precipitation of carbides and TCP phases causes the deterioration of mechanical properties and thereby reduces their service life and reliability. However, the investigation on the microstructure evolution of the carbides and TCP phases in K403 alloy is also still very limited [1]. A better understanding of the precise mechanisms of the precipitation of carbides and TCP phases is also of great necessity to predict the service life and reliability of K403 alloy.

The present investigation is aimed to elucidate these two issues. Firstly, the precipitation and transformation of carbides and TCP phases is investigated, with a special focus on the nucleation of the σ phase and the in-situ transformation from the σ phase to the P phase. Secondly, the solute segregation behavior during thermal exposure and its effect on the morphology of γ' precipitates is investigated and discussed in terms of the stain energy and the interfacial energy. The present investigation is aimed to provide a better understanding on the high temperature performance, which might lead to a more accurate prediction of failure and thereby enhancing the reliability and service life of the K403 Ni-based superalloy.

2. Experimental methods

Ni-based superalloy K403 was melted in a vacuum induction furnace. The alloy melting was poured into test bars in vacuum. The bars were placed in a SX2-10-13 chamber electric furnace for thermal exposure in air at 800 °C for 50, 100, 150, 200 h, and 950 °C for 5, 25, 50, 100, 150, 200 h, respectively.

The samples for scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) investigation were mechanically ground and electro-polished in a solution consisting of 8% perchloric acid and alcohol. SEM investigation was performed using a Zeiss 1525 SEM equipped with an Energy-dispersive X-ray spectroscopy (EDX) system. The EBSD investigation was performed using a Zeiss 1525 SEM equipped with an EDAX EBSD system. The data evaluation was undertaken with orientation imaging microscopy (OIM) software.

The samples for transmission electron microscope (TEM) investigation were mechanically ground, polished and dimpled to about 30 μm, and then ion-beam milled using a Gatan Precision Ion Polishing System (PIPS, Gatan model 691). The preparation temperature (about - 10 °C) was kept constant by using a cold stage during ion beam polishing to prevent any modification of the sample structure during preparation. TEM investigation was performed using a Philips CM12 microscope operated at 120 kV equipped with a CCD-camera (GATAN Model 794 MSC BioScan). HAADF STEM imaging and EELS were performed using a Nion UltraSTEM100 aberration corrected dedicated STEM. The microscope was operated at an acceleration voltage of 100 kV and an electron probe convergence semi-angle of 31 mrad, which resulted in an estimated minimum electron probe size of 0.8 Å. The cold field emission gun of the microscope has a native energy spread of 0.35 eV. The HAADF detector semi-angles were 83–185 mrad and the spectrometer collection semi-angle was 36 mrad. EELS maps where then created by integrating the EELS signal of each edge: Ni $L_{2,3}$ (855 eV), Cr $L_{2,3}$ (575 eV), Co $L_{2,3}$ (779 eV), Ti $L_{2,3}$ (456 eV) and C *K* (284 eV), over a suitable energy window after subtracting the preceding exponential background fitted with a power law. All EELS edges were identified following reference [17]. EELS spectra were de-noised using Principle Component Analysis (PCA) as implemented in the MSA plugin for [18] for Gatan's Digital Micrograph software. The intensities of the EELS maps were displayed on a false color scale, so that within each map, a low intensity (black) corresponds to a lower relative concentration, while increased contrast (color) corresponds to an increase in elemental concentration.

In situ synchrotron diffraction experiment was performed on the 4B9A beamline of BSRF (Beijing Synchrotron Radiation Foundation). The samples were illuminated with a monochromatic X-ray beam at an energy of 7708 eV ($\lambda = 0.161261$ nm). The samples were placed in a furnace evacuated down to 10^{-2} mbar. The samples were ramped from 80 °C to 480 °C at a rate of 10 K/min, and then kept constant for subsequent scanning. A Mythen detector, which allows simultaneous acquisition of full X-ray diffraction profiles without mechanical rotation, was used. The acquisition time was set for 60 s per frame to acquire higher resolution. The data was calibrated at the beamline by a Matlab program [19].

3. Results

3.1. As-cast microstructure

Fig. 1a shows SEM image of the as-cast microstructure in K403 alloy. The as-cast microstructure consists of a γ phase, γ' phase, ($\gamma + \gamma'$) eutectic phase and MC phase, where “M” designates one or more types of metal atoms (i.e. Ti, Mo, W). The γ phase is the Ni-based austenite phase with multiple solution elements (i.e., Co, Cr, Mo, and W) [20]. The γ' phase is precipitated coherently in the solid state from the γ matrix [20]. The γ phase and γ' phase can be more clearly observed at high magnification, as shown in Fig. 1b. The morphology of the γ' phase appears to be cubic with a size of