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Optimizing the microstructure and mechanical behaviors of in-situ TiC-γ′/Ni composites by subsequent thermal treatment

Wenqiang Hu [a], Zhenying Huang [a, b, *], Guangming Zheng [a], Yuanbo Wang [a], Cong Lei [a], Leping Cai [a], Hongxiang Zhai [a], Yang Zhou [a]

[a] *Institute of Materials Science and Engineering, School of Mechanical and Electronic Control Engineering, Beijing Jiaotong University, Beijing, 100044, China*
[b] *Key Laboratory of Vehicle Advanced Manufacturing, Measuring and Control Technology (Beijing Jiaotong University), Ministry of Education, Beijing, 100044, China*

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ABSTRACT

Previous studies revealed that Ni-based composites with *in-situ* formed TiC and γ′-Ni_3(Al,Ti) reinforcements possess unusual microstructural and thermal features, which could greatly pin the grain growth as well as affect dislocation motion and contribute the mechanical performances. However, the as-prepared TiC-γ′/Ni composite reported previously is insufficiently strong for engineering applications, and the size and morphology of heterogeneous TiC-γ′ which have critically influenced mechanical properties are urgently needed to be optimized. In the present work, the tailored microstructural features were obtained under different fabrication procedures combined with subsequent heat treatments, which provides the insights about the factors influencing the critical strength in TiC-γ′/Ni composites. It was found that the synthesis temperature significantly affect the size and morphology of TiC particles, while they always remain thermodynamically stable under long-term annealing. However, annealing treatments contribute to the more amount of γ′ precipitation out of γ-Ni matrix. The TiC-γ′/Ni composites fabricated under 1200 °C with subsequent solution and aging treatments exhibit tensile strength of ~1300 MPa with ~6.2% ductility. The superior combination of strength and ductility can be attributed to the more homogenous distribution and location of ultrafine TiC (100–700 nm) and γ′ (50 nm) nanoparticles in the Ni matrix, which played a synergistic effect on determining the enhanced mechanical behaviors.

1. Introduction

Nickel matrix composites (NMCS) are attractive for various applications, including aerospace, automotive, chemical and nuclear industries, due to their remarkable mechanical behaviors at room and elevated temperatures, excellent fatigue and corrosion resistance [1,2]. Like the other particles reinforced metal matrix composites, the design strategy is generally based on one principal constituent and adds other minor reinforcing particulates (et. TiC, WC and SiC) [3–14]. It was found that a single carbide particulates reinforced Ni matrix is insufficiently strong for practical applications due to the limited wettability and great discrepancy of thermal expansion coefficient between the reinforcement and Ni matrix [5–10]. Herein, in our previous investigations [15–18], a new Ni-based composite design concept, namely *in-situ* dual-phase TiC-γ′ reinforced Ni matrix composite, was proposed. The basic idea is to simultaneously generate two reinforcements by triggering decomposition of MAX phase material to promote the *in-situ* precipitation of hard TiC and coherent γ′ grains in the Ni matrix.

It is well established that the ceramic particles size has a significant influence on the mechanical properties of metal matrix composites. Finer reinforcements have better strengthening effect than the coarse counterpart. In-situ formed TiC particulates transformed from the decomposition of Ti_2AlC have effectively reinforced Ni matrix [15,16]. At the same time, Ti_2AlC degraded into TiC accompanied by releasing enough Al and remnant Ti atoms into Ni matrix, $L1_2$ ordered nano γ′ particles subsequently precipitated out of Ni matrix [17,18]. In Ni-based superalloy, superior mechanical properties of the alloy are generally obtained by the precipitation

* Corresponding author. Institute of Materials Science and Engineering, School of Mechanical and Electronic Control Engineering, Beijing Jiaotong University, Beijing, 100044, China.
E-mail address: zhyhuang@bjtu.edu.cn (Z. Huang).

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strengthening of γ′ phase [19]. Extensive researches have also suggested that the size and volume fraction of γ′ precipitates have substantial influences on mechanical properties and deformation modes of the Ni-based alloys [20,21]. The contribution of γ′ precipitation strengthening is complicated, because it depends on the complex microstructure which is generally modulated by the heat treatment conditions. Volume fractions, size and particle spacing of γ′ precipitates all impact on the deformation and hardening mechanisms. Humphreys strengthened effects of particle stimulated nucleation (PSN) and the Zener pinning effect of second phases to the deformation behaviors [22,23].

Based on the above discussion, it is possible to optimize the mechanical properties of synthesized composites by controlling the size and fractions of both reinforcements including TiC and γ′ precipitates. To accomplish this objective, a virgin TiC-γ′/Ni composites were firstly fabricated by hot-press sintering Ti_2AlC and Ni powders at lower temperature to impede the growth of TiC particles. Subsequently, different heat treatment procedures were performed on the as-resulted samples to reveal the nanoscale γ′ precipitates evolution in the fcc matrix. To provide insight into the underlying mechanisms, both microstructural characterization and mechanical behavior were investigated. The results are expected to provide valuable information for the future design of *in-situ* TiC and γ′ synergistically reinforced Ni matrix composites in service.

2. Experimental process

TiC-γ′/Ni composites were fabricated by hot-press sintering the mixture of 30 vol% Ti_2AlC precursor and 70 vol% Ni powders. Two different sintering procedures were applied in the present work. One is a two-step sintering method. In detail, the as-resulted blending powders together with the mold were placed into vacuum hot-pressing furnace (ZT-30-22Y, Chenhua, China) and heated to 1400 °C holding for 30 min with a heating rate of 10 °C/min in Ar atmosphere to make a uniform dispersion of transformed TiC particles in the molten Ni matrix. For densification, a uniaxial pressure of 30 MPa was then applied for 30 min at 1230 °C for Ti_2AlC/Ni samples. The pressure of 30 MPa was maintained until furnace temperature cooled down to 500 °C. The second one is a one-step sintering method. The Ti_2AlC/Ni powders fixed in the mold were placed into vacuum hot-pressing furnace and directly heated to 1200 °C with a heating rate of 10 °C/min in Ar atmosphere, and holding for 1 h with the pressure of 30 MPa until furnace temperature cooled down to 500 °C. Besides, details about the method of synthesis of Ti_2AlC and mixing process of Ti_2AlC and Ni powders can be found elsewhere [18,24]. The resulted samples fabricated under 1400 °C (labeled as A0) were subsequently exposed to aging treatments at 750 °C for 20, 50 and 100 h in an electrically heated tube furnace (TSK-5-14, FNS, Beijing) followed by air cooling to produce microstructures with variations in their precipitate size while maintain the respective chemical compositions (from here on referred to as samples A1, A2, A3, respectively), as reported in Refs. [25–27]. By contrast, the resulted samples fabricated under 1200 °C (labeled as S0) were firstly subjected to a heat treatment procedure consisting of a solution heat treatment at 1200 °C for 1 h followed by quenching in water. Then, the TiC-γ′/Ni specimens were aged at 750 °C for 20, 50 and 100 h (from here on referred to as samples S1, S2, S3, respectively) followed by air cooling. The different processing conditions of Ti_2AlC/Ni samples in the present study are also summarized in the Table 1.

Phase evolutions of the sintered samples were identified by X-ray diffractometer (XRD) using Cu Ka radiation (X0 PERT-PRO MPDT, Netherlands). The microstructural characterizations were performed using a scanning electron microscope (SEM, ZEISS EVO 18, Germany) and a transmission electron microscope (TEM, FEI Tecnai G2 F20). XRD and SEM specimens were initially polished to 2000-grit SiC paper and, subsequently, polished to 0.5 μm by diamond paste and finally cleaned using ultrasonic bath and ethanol. The specimens corresponding to any given aging treatment were polished and then etched in a solution of 5 ml HNO_3 + 95 ml C_2H_5OH to reveal γ′ precipitates. The size distributions and volume fractions of γ′ precipitates were statistically counted following the procedures described in Ref. [28].

Rectangular cuboidal tensile samples with a gauge length of 8 mm and a cross-section of 2 mm × 1 mm were machined from as-sintered discs. Tensile test was performed on a universal electron testing machine (WDW-100E) with a nominal strain rate of 1×10^{-3} s^{-1}. The edge notched bending test specimens with dimension of 2.5 mm × 5 mm × 26 mm with a notch of 0.2 mm in width and 2.5 mm in depth were used for the evaluation of fracture toughness (K_{IC}) by universal electron material testing machine under a loading speed of 0.05 mm/min. The span size is 20 mm. The surface of all tensile and K_{IC} specimens were polished down to a 2000-grit SiC paper to eliminate scratches. 3–4 measurements were conducted for the tensile and fracture toughness tests, respectively. Vickers hardness (HV) test was performed on a TH-700 hardness tester (Shidai, China) with a load of 50 N and a diamond prism cone indenter. Seven measurements in different areas were taken for each sample. Each reported hardness is an average of seven measurements.

3. Results and discussion

3.1. Initial microstructure observation

SEM micrographs obtained from the surface of TiC-γ′/Ni composite fabricated under 1400 °C (sample A0) are compiled in Fig. 1a–c. From Fig. 1a, the good dispersion of TiC grains in the composite can be noted. No obvious porosity is observed on the surface, which indicates that the composite tend to be fully consolidated by HP process in light of perfect penetration of Ni matrix to *in-situ* TiC particulates. Fig. 1b is an etched image taken from Fig. 1a, it presents that *in-situ* formed TiC particles are mainly dispersed on the boundaries of the Ni alloy grains. Moreover, nanoscale γ′ precipitates in the isometric morphology with the grain size of 100 nm are homogenously distributed inside the γ-Ni matrix, as found in Fig. 1c. Fig. 1d illustrates the microstructure consisting of heterogeneous TiC and γ′ particulates observed from TEM. Meanwhile, HRTEM indicates an excellent interfacial bonding formed without any impurity between TiC and γ-Ni matrix (Fig. 1e). Besides, γ′/γ interface remained completely coherent as indicated from the inset corresponding to the electron diffraction pattern (Fig. 1f), which coherent relationship, according to previous report [27,28], will maintain in spite of exposing to long-term duration of annealing. The fine TiC and γ′ precipitates can effectively inhibit the growth of Ni grain due to the grain growth stagnation related to Zener pinning model resulting in a fine microstructure [29], which has also been detailed in our previous work [11]. It is well known that the mechanical properties of a Ni-based superalloy are dependent upon structure parameters such as volume fraction and size of γ′ precipitate [30]. Whether the properties of this novel Ni matrix composites reinforced with *in-situ* dual-phase TiC-γ′ can be optimized by tailoring the microstructure? The following content about the effect of heat treatment on the microstructure and properties of TiC-γ′/Ni composite will unveil the answer.

3.2. The influence of different experimental procedures on sample morphologies

The effect of annealing on the phase structure of TiC-γ′/Ni