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A NiAl- and TiC-reinforced Fe-based nanocomposite prepared by the rapid-solidification thermite process

Wenjun Xi*, Haijing Wang, Jing Li, Chaoliang Shi

School of Materials Science and Engineering, Beihang University, Beijing 100191, PR China

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ABSTRACT

A NiAl- and TiC-reinforced iron-based nanocomposite FeNiCrAl/NiAl–TiC was prepared using the rapid-solidification thermite process. The composite consisted of ferrite, nano-scale intermetallic NiAl and micro-scale TiC. After an aging treatment at 600 °C, the nano-scale NiAl precipitated in the matrix ranged from 20 nm to 50 nm in diameter, which led to an increase in the tensile strength of the material. This composite exhibited excellent oxidation resistance, and the mass gain after 100 h of isothermal exposure at 1000 °C in air was less than 1 mg/cm^2, which is approximately 1/70 of the mass gain of 304 stainless steel under equivalent conditions.

1. Introduction

Modern nickel-based superalloys possess excellent mechanical properties at elevated temperatures because of the presence of ordered γ′ precipitates (Ni_3Al-type, $L1_2$ structure) with volume fractions ranging up to 60%. These precipitates are coherent with the matrix and thermodynamically stable. If a coherent and thermodynamically stable strengthening phase could be obtained in the ferritic matrices of Fe–Cr–Al alloys, analogous to the γ′ precipitates in the superalloys, the high-temperature strength of the ferritic alloys could be increased dramatically. The intermetallic compound NiAl (β phase) with a B2 ordered structure (CsCl prototype) is a derivative of the bcc structure, and its lattice parameter (0.2880 nm) is close to that of α ferrite (0.2876 nm) [1] such that β phase satisfies the lattice parameter requirement in bcc ferritic alloys. In the early 1970s, the strengthening of steels with β precipitates was investigated by Pickering [2]. This work led to the development of alloys, such as precipitation-hardened stainless steel (17–7 PH or PH 13-8 Mo) [3,4]. In the last three decades, the mechanical properties at room and elevated temperatures and the creep behaviour of ferritic alloys strengthened by β precipitates have been investigated in detail [5–11]. However, the β phase should not be compared directly with the γ′ phase for its strengthening effect because of its low volume fraction (usually less than 10%) in the ferritic alloys. In addition, the coarsening rates of the β precipitates are large. Precipitates lose their coherency with the ferritic matrix when they exceed a certain size [5]. Furthermore, β-strengthened alloys generally require long aging times (sometimes longer than 300 h) to obtain a suitable volume fraction of the β precipitates, thereby increasing the fabrication cost of the alloys. In the present work, a thermite process was used as an alternative to the conventional metallurgical method for producing a melt containing Fe, Ni, Cr, Al, Ti and C. The melt was rapidly solidified in a copper mould, using a method known as the rapid-solidification thermite process (RST process). Through these methods, an iron-based composite reinforced by a coherent β phase with a high volume fraction was successfully synthesised. Moreover, TiC particles with a grain size of 0.1–3 μm were synthesised in situ in the composite. These carbide particles can restrict the coarsening of the β phase and maintain their coherent strengthening efficiency at higher temperatures. The TiC particles can also reinforce the ferritic matrix and further increase the strength of the composite at high temperatures. The RST process is inexpensive and fast, and it thus provides a novel alternative approach to developing high performance iron-based high-temperature structural materials.

2. Experimental procedures

Fig. 1 schematically shows the RST process used in this study. The Fe_2O_3, CrO_3, Cr_2O_3, NiO, Al, C and Ti powders were blended, and the mixed powders were placed in a graphite crucible that was enveloped in refractory bricks. A hole made in the crucible bottom was covered with an aluminium foil. The composition of the thermite mixture is listed in Table 1. The graphite crucible with the

* Corresponding author. Tel.: +86 1082338190; fax: +86 1082339772.
E-mail address: xiwj@buaa.edu.cn (W. Xi).

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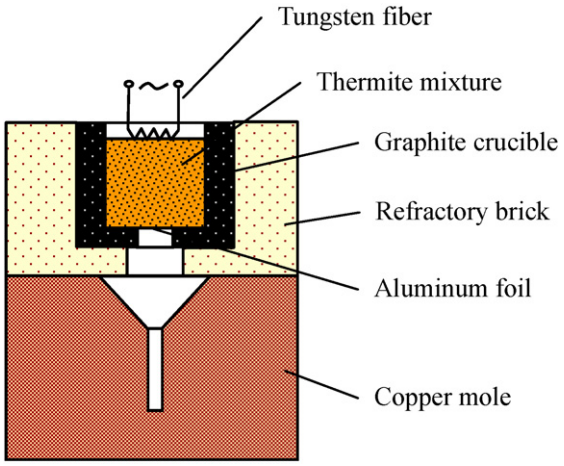

Fig. 1. Schematic representation of the RST process.

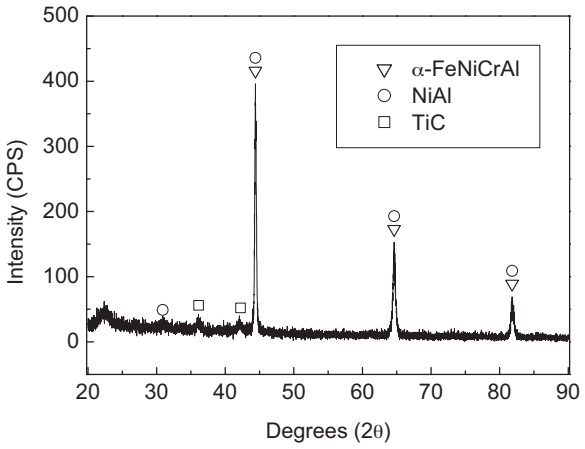

Fig. 3. XRD pattern of FeNiCrAl/NiAl–TiC.

Table 1
Chemical composition (wt%) of the starting materials.

Element	Fe_2O_3	CrO_3	Cr_2O_3	NiO	Al	Ti	C
Wt/%	34.9	14.1	9.3	12.3	24.8	0.9	3.7

thermite mixture was placed in a drying oven and preheated for 2 h at 200 °C. The crucible was placed on a copper mould, and the thermite mixture was immediately ignited by a tungsten filament. Over the course of the reaction, the oxides were reduced by aluminium, resulting in the formation of the molten product containing Fe, Ni, Cr, Al, Ti, C and alumina (Al_2O_3). The chemical reactions generated in the thermite process can be expressed as follows:

$$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3 \quad (1)$$

$$Cr_2O_3 + 2Al \rightarrow 2Cr + Al_2O_3 \quad (2)$$

$$CrO_3 + 2Al \rightarrow Cr + Al_2O_3 \quad (3)$$

$$3NiO + 2Al \rightarrow 3Ni + Al_2O_3 \quad (4)$$

$$3NiO + 5Al \rightarrow 3NiAl + Al_2O_3 \quad (5)$$

$$Ti + C \rightarrow TiC \quad (6)$$

The combustion temperature was sufficiently high that all reacted products became liquid. Due to differences in density, the alumina was on top of the reaction products and separated naturally from the desired metal melt. The molten products melted the aluminium foil at the bottom of graphite crucible and subsequently flowed down into the copper mould. After cooling to room temperature, the alumina (Al_2O_3) on top of the casting was removed to yield an iron-based composite (FeNiCrAl/NiAl–TiC) reinforced by β (NiAl) and TiC.

A Leo-1530 Field Emission scanning electron microscope (SEM, LEO Co, Germany) operating at 10.00 kV and a JEOL-2100F Transmission electron microscope (TEM, JEOL, Tokyo, Japan) operating at 200 kV accelerating voltage were used for the analysis of the composite morphologies and microstructures. The structure was analysed using a D/max-2500 X-ray diffractometer with CuKα radiation.

Compression samples with a diameter of 5 mm and a height of 15 mm were prepared according to ASTM (American Society for Testing and Materials) standards. The compression properties at room temperature were tested using an MTS 880 (MTS System Corporation) material testing machine at a strain rate of $5 \times 10^{-4}\ s^{-1}$. High-temperature compression tests were conducted with Gleeble 2000 servo-hydraulic test machine at a strain rate of $1 \times 10^{-4}\ s^{-1}$ to ~40% strain.

The tensile mechanical properties were tested using samples with a size of 2 mm × 5 mm × 12 mm (and a total length of 42 mm) under a strain rate of $1 \times 10^{-4}\ s^{-1}$ using an MTS 880 between room temperature and 900 °C.

The oxidation behaviour was studied in air. The oxidation test coupons were 10 mm × 10 mm × 1 mm, and the surfaces were polished with 800-grit emery paper. The mass change data after 100 h of isothermal exposure at 1000 °C in air for the investigated composite and 304 stainless steel were measured using a Sartorius CP225D electronic balance with a sensitivity of 0.01 mg.

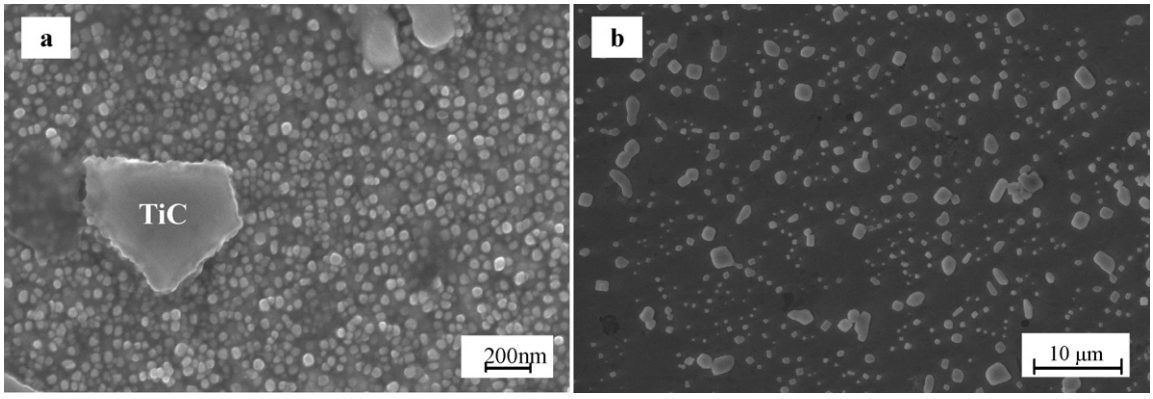

Fig. 2. SEM micrograph of FeNiCrAl/NiAl–TiC (4% TiC). (a) The matrix of the composite, composed of ultrafine grains of approximately 50 nm. (b) TiC particles in the matrix.