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Original Research

Dendrite growth and micromechanical properties of rapidly solidified ternary Ni-Fe-Ti alloy

Ying Ruan, Haizhe Zhu, Qingqing Wang, Fuping Dai, Delu Geng, Bingbo Wei*

Department of Applied Physics, Northwestern Polytechnical University, Xi'an 710072, China

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ABSTRACT

The rapid solidification of undercooled liquid $Ni_{45}Fe_{40}Ti_{15}$ alloy was realized by glass fluxing technique. The microstructure of this alloy consists of primary γ-(Fe, Ni) phase and a small amount of interdendritic pseudo-binary eutectic. The primary γ-(Fe, Ni) phase transferred from coarse dendrite to fragmented dendrite and the lamellar eutectic became fractured with the increase of undercooling. The growth velocity of γ-(Fe, Ni) dendrite increased following a power relation with the rise of undercooling. The addition of solute Ti suppressed the rapid growth of γ-(Fe, Ni) dendrite, as compared with the calculation results of Fe-Ni alloy based on LKT model. The microhardness values of the alloy and the primary γ-(Fe, Ni) phase increased by 1.5 times owing to the microstructural refinement caused by the rapid dendrite growth. The difference was enlarged as undercooling increases, resulting from the enhanced hardening effects on the alloy from the increased grain boundaries and the second phase.

1. Introduction

The undercooled melt is far from the thermodynamic equilibrium state, accordingly the dendritic morphology of an undercooled alloy varies, which will influence the final performances of the alloy. LKT/BCT model has been successfully applied to describe the dendrite growth mechanism of pure metals and binary alloys [1–3]. In recent decades, the researchers are attempting to investigate the dendrite growth mechanism of ternary or multicomponent alloys by means of theoretical simulation methods, i.e. phase field, cellular automaton, etc. [4–6], however there still requires an amount of necessary elaborative experimental results. The relationship between dendrite growth velocity and undercooling is crucial to understanding the rapid dendrite growth mechanism. Moreover, the experimental investigation on dendrite kinetics is helpful for the microstructure controlling and performance optimizing [7–11].

Ni–Fe based alloys with the outstanding high-temperature creep strength and satisfactory corrosion resistance, have obtained wide attention. The addition of a small amount of Ti improves the applied performances of Ni–Fe based alloys due to its excellent corrosion resistance and higher strength [12–14]. In the case of Ni–Fe–Ti ternary alloy system, Gupta et al. plotted a partial liquidus projection for the Ti-lean region [15,16]. Cacciamani et al. carried out a critical evaluation of the Ni – Fe – Ti alloy system and presented a liquidus projection for the whole composition range [17,18]. Keyzer et al. launched a thermodynamic assessment of the Ni – Fe – Ti alloy system and optimized the thermodynamic parameters according to available experimental data [19]. The phase relations in the Fe – Ni – Ti system were studied and two isothermal sections as well as a revised liquidus projection were established by Duarte et al. [12]. So far, most research on Ni – - Fe – Ti ternary alloy system focused on phase constitution, phase equilibrium and thermodynamic characteristics [15–21]. The existing literatures on Ni – Fe – Ti alloy system provide the valuable information for further investigating the dendrite growth in the alloy system in our work. Moreover, the solute element content and type are crucial to the grain size especially equiaxed grain size variation subject to undercooling in Ni-Fe-based alloys [22]. The influence of Ti solute on dynamical crystal growth and in turn that of rapid dendrite growth on mechanical properties are worthy to be clarified.

Rapid dendrite growth under non-equilibrium conditions can be realized by means of various undercooling techniques. The glass fluxing technique, in which the contamination from crucible walls is avoided, achieves a large undercooling for a metal sample. The objective of this work is to realize the undercooling and rapid solidification of $Ni_{45}Fe_{40}Ti_{15}$ ternary alloy using the glass fluxing technique. The rapid dendrite growth, microstructure and micromechanical properties are studied in this work.

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* Corresponding author.

E-mail address: bbwei@nwpu.edu.cn (B. Wei).

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2. Experimental

The $Ni_{45}Fe_{40}Ti_{15}$ alloy samples were prepared using pure elements of Fe (99.99%), Ni (99.99%) and Ti (99.99%) in an arc melting furnace and each sample has a mass of about 2 g. The sample covered with a designed denucleating agent was placed in an alumina crucible with the size of 12 mm (OD) × 10 mm (ID) × 16 mm. The vacuum chamber was evacuated to 2×10^{-4} Pa and backfilled with argon gas. The sample was melted and superheated to 200–300 K above its liquidus temperature by high frequency induction heating and then cooled through adjusting the heating power. The process was repeated several times in order to obtain different undercoolings. The temperature was monitored by a Yunnan-Land NQO8/15 C infrared pyrometer calibrated by a thermocouple of NiCr-NiSi.

After experiments, the solidified samples were cross-sectioned, polished and etched. The solidified microstructures were analyzed by a Zeiss Axiovert 200 MAT optical microscope and an FEI Sirion 200 scanning electron microscope. The thermodynamic properties were determined with a differential scanning calorimeter (DSC, Netzsch DSC 404 C) under the heating-cooling rate of 10 $Kmin^{-1}$. The phase constitution was analyzed by an X-ray diffractometer (XRD, Rigaku D/max 2500 V). The Vickers hardness of the undercooled alloys was studied by a microhardness tester (Tai Ming HXD-2000TMC/LCD) equipped with a diamond pyramid indenter and a microscope. Loads of 2000 gf and 25 gf were chosen for the microhardness measurements of the alloy and γ-(Fe,Ni) phase respectively and a dwell time of 10 s was applied.

3. Results and discussion

3.1. Microstructural characteristics of rapidly solidified $Ni_{45}Fe_{40}Ti_{15}$ alloys

The composition point of $Ni_{45}Fe_{40}Ti_{15}$ is located in the γ-(Fe, Ni) phase region in the equilibrium phase diagram, as marked by point A in Fig. 1 [17]. Fig. 2a presents the DSC curve of heating-cooling process with the sample mass of 50.74 mg. The liquidus temperature is 1544 K. During heating three endothermic peaks occur. The lowest peak at 1384 K matches the ternary eutectic transition, i.e. γ-(Fe, Ni) + Fe_2Ti + Ni_3Ti → L, marked as E_1 in Fig. 1 with the reported transition temperature of 1381 K [12]. On cooling this final solidification step is obviously suppressed in the DSC sample. The undercooling range from 28 to 118 K was achieved in the experiments. The phase constitution of the alloy at different undercoolings was determined by XRD analysis, as shown in Fig. 2b. The undercooled alloy consists of γ-(Fe, Ni) and Fe_2Ti phases. The (111) peak of γ-(Fe, Ni) phase at 43.6° is the strongest diffraction peak for both master alloy and undercooled alloys.

The microstructural morphologies of the solidified alloy at different undercoolings are illustrated in Fig. 3a and b. EDS analysis indicates that the white phase is γ-(Fe, Ni) and the black phase is Fe_2Ti [23]. At a low undercooling of 34 K, the primary γ-(Fe, Ni) phase formed as a coarse dendrite with the evident preferred growth orientation. The dendrites grew well and the maximum dendrite trunk length is up to 1.093 mm. In the interdendrite region, γ-(Fe, Ni) and Fe_2Ti phases grew cooperatively to form lamellar eutectic, as shown in Fig. 3c. As undercooling increased, the large amount of the latent heat released during solidification and the copious nucleation at the solidification front induced by a pressure pulse due to the collapse of shrinkage cavities [24] fracture the dendrites and the interdendritic lamellar eutectic, as well shown in Fig. 3d.

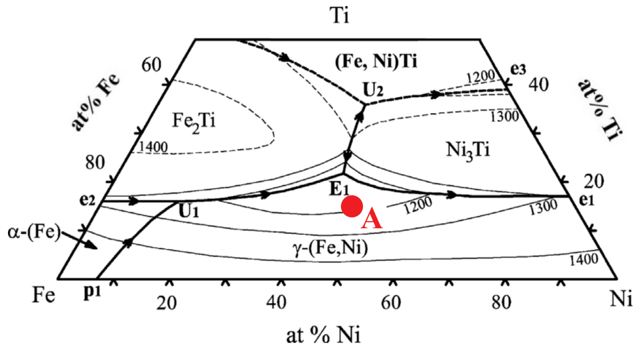

Fig. 1. The composition location of $Ni_{45}Fe_{40}Ti_{15}$ alloy in the phase diagram [17].

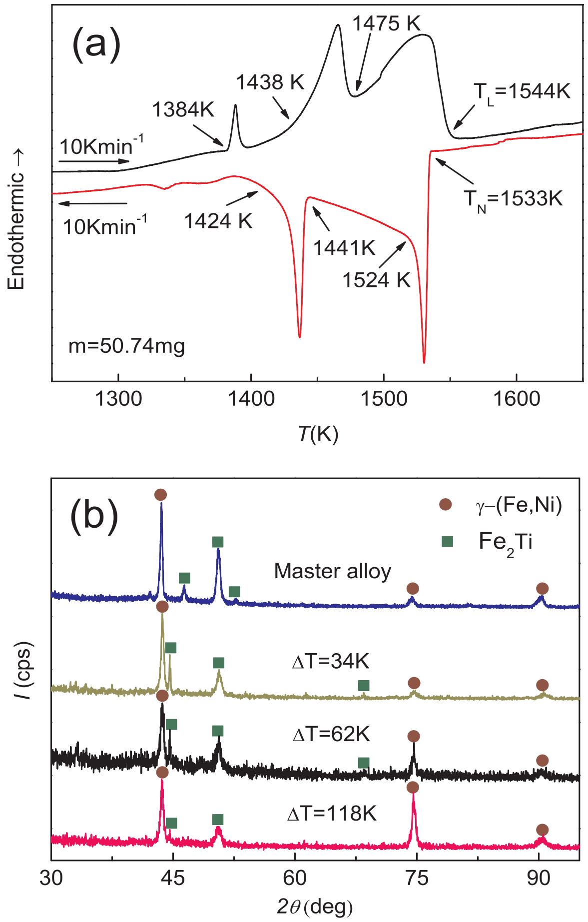

Fig. 2. Analytical characteristics of $Ni_{45}Fe_{40}Ti_{15}$ alloy: (a) DSC curve and (b) XRD patterns.

3.2. Dendrite growth at large undercoolings

The measured growth velocities of γ-(Fe,Ni) dendrite from the undercooled alloy are presented in Fig. 4a. γ-(Fe,Ni) dendrite grew sluggishly, and its growth velocity value is on the order of mms^{-1}, which is far less than that of Fe or Ni dendrite in binary alloys [25,26]. The dendrite growth velocity V increased with the rise of undercooling ΔT. The relationship between V and ΔT can be fitted by the following power equation

$$V = 6.28 \times 10^{-2} \Delta T^{1.03}. \qquad (1)$$

The dendrite growth velocity increased gradually from 2.6 to 8.9 mms^{-1} as the undercooling was up to 118 K. The addition of 15 at%Ti caused γ-(Fe,Ni) dendrite growth to become sluggish with the velocity of mms^{-1} order, as compared with its growth velocity of ms^{-1} in Fe−Ni or Fe−Ni−Cr alloys [27,28]. It is well known that Fe and Ni elements display complete solid solubility to form isomorphous alloy. In the case of Ni−Fe−Ti alloy, the diffusion velocity of Ti in the melt was low and the partition coefficient of Ti in the γ-(Fe,Ni) phase was relative small, contributing to the sluggish dendrite growth.

The Ti content in the γ-(Fe,Ni) phase was measured to characterize