δ/γ transformation in non-equilibrium solidified peritectic Fe-Ni alloy

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Through phase transformation kinetic analysis and experimental observation, the δ/γ transformation occurring in the non-equilibrium peritectic Fe-4.33at.%Ni alloys was systematically investigated. According to JMA solid-state transformation kinetic theory, the Time-Temperature-Transformation (TTT) curves of the δ/γ transformation in peritectic Fe-Ni alloy were calculated. On this basis, the physical correlation between the δ/γ transformation and the initial undercooling of melt (ΔT) was elucidated. The results indicate that the change of ΔT can alter not only the overall δ/γ transformation pathways but also the transformation fraction with respect to each transformation mechanism.

δ/γ transformation, undercooling, non-equilibrium solidification, peritectic reaction, Fe-Ni alloy

1 Introduction

Solidification and solid-state transformation are two basic and correlated physical processes in the course of materials preparation. Recently, with the development of non-equilibrium techniques, the non-equilibrium characteristics involved in materials preparation become more and more remarkable. High undercooling rapid solidification can make the alloy melt achieve a large ΔT prior to nucleation, driven by which, the crystal growth incorporated in solidification can be very rapid, even can reach to the order of 10 m/s, and then results in the non-equilibrium solidification. Many researches have demonstrated that the solidification course of an undercooled melt is dominated by ΔT . If ΔT exceeds a critical value, the solidification behavior and the as-solidified microstructures will exhibit totally different characteristics in comparison with the case of near-equilibrium solidification, e.g. the change of solidification behavior, the alternation of primary phase and grain

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refinement, etc.^[1]. Those features are bound to predominantly influence the subsequent solid-state transformation, and in turn the final as-solidified microstructures. Hence, the research on the effect of non-equilibrium solidification on subsequent solid-state transformation is very necessary for understanding and controlling the materials microstructural formation process. Unfortunately, due to the limited development of theory and technique, the study on such fields is still lacking.

Fe-based alloy is a kind of widely used materials and the δ/γ transformation is a most common and important solid-state transformation process in Fe-based alloy. Many researches^[1–8] have indicated that the change of ΔT can predominately alter the solidification behavior of undercooled Fe-based alloy, which can also unavoidably influence the following δ/γ transformation. Thus, to study the influence of ΔT on the δ/γ transformation in undercooled Fe-based alloy is not only valuable for theoretical development but also of importance in the engineering field. Considering that many Fe-based alloys have similar solidification and phase transformation behavior with Fe-Ni system, in present work, the peritectic Fe-4.33at.%Ni alloy was selected as the research object. This paper aims to investigate the physical correlations between ΔT and the δ/γ transformation in the non-equilibrium Fe-4.33at.%Ni alloy.

2 Experimental procedures

Figure 1 shows the Fe-Ni phase diagram, and the selected composition lies in the peritectic point.

Figure 1 Fe-Ni phase diagram^[5,8].

The master alloy ingots were prepared by *in situ* alloying high purity Fe (99.9%) and Ni (99.99%) elements in an ultrahigh vacuum chamber by high frequency induction melting under high purity Ar atmosphere. After melting practices, the as-prepared master alloys were cut into small blocks. Then, the blocks (weighing 5 g) together with some dehydrated B₂O₃-glass (weighing about 15%–20% of the sample's mass) to avoid the oxidation were placed into a silica crucible and remelted by a high frequency induction coil. The sample was superheated 100 K above the melting point for 5–10 min, then the power was shut down and the sample began to solidify spontaneously. After solidification, as the system temperature decreased to about 1473 K, the sample was rapidly quenched into Ga-In-Sn bath (with a composition of Ga 62at.%, In 25at.%, Sn 13at.% and a temperature of 20°C) to suppress the solute redistribution after δ/γ transformation and the eutectoid reaction occurring at low temperature. The thermal behavior of the sample was monitored by a two color infrared pyrometer with a response time of 5 ms and a relative ac-

curacy of ± 1 K, which was calibrated by a PtRh30-PtRh6 thermal couple.

The as-solidified morphologies were observed by optical microscopy and transmission electron microscopy (TEM). Phase constitutions of the room temperature (RT) microstructures were analyzed by X-ray diffraction (XRD) method. The Ni element distribution in the RT microstructure was measured by electrical microprobe (EPMA).

3 Discussion

3.1 Undercooling dependent solidification behavior

The nucleation, growth, and phase selection behavior of undercooled peritectic Fe-4.33at.%Ni alloy have been reported detailedly in our previous work^[9]. The experimental and calculated results suggest that subject to the spontaneous nucleation, δ phase can always solidify as the primary phase from the undercooled melt. The preferential solidification of δ phase is due to its larger driving force for nucleation (i.e. Gibbs free energy between liquid phase and solid phase) and its smaller resisting force for nucleation (i.e. interfacial energy between liquid phase and solid phase) in comparison with γ phase.

Although δ phase can always act as the primary phase in the achieved undercooling range, with respect to a critical undercooling, $\Delta T^* \approx 130$ K, the solidification behavior is totally different. When $\Delta T < \Delta T^*$, δ phase solidifies as the primary phase, which results in the first recalescence event; while the temperature decreases below the peritectic plateau temperature, the peritectic reaction, $L + \delta \rightarrow \gamma$, takes place and forms a peritectic layer around the primary δ phase, following which, the remaining liquid grows epitaxially on the as-formed peritectic layer, leading to the second recalescence, see Figure 2. If $\Delta T > \Delta T^*$, because the incubation time for occurrence of the peritectic reaction is longer than the time essential for complete solidification of the δ phase dur-

Figure 2 Two typical schematic recalescence curves of undercooled Fe-4.33at.%Ni. The left corresponds to $\Delta T < \Delta T^*$, and the right to $\Delta T > \Delta T^*$, where T_l^δ , T_l^γ , T_p and T_s^δ is liquidus temperature of δ phase, liquidus temperature of γ phase, equilibrium peritectic reaction plateau temperature and solidus temperature of δ phase, respectively.

ing post-recalcsence, the peritectic reaction and in turn the solidification of γ phase are suppressed, and only δ phase can solidify from the melts. In this case, only one recalcsence event can be observed, see Figure 2.

3.2 Undercooling dependent δ/γ transformation kinetics

During continuous cooling, once the as-formed δ phase enters into the γ phase area in the phase diagram, the occurrence of δ/γ transformation is thermodynamically possible. For the non-equilibrium microstructure of the peritectic Fe-4.33at.%Ni alloy, the δ/γ transformation can proceed thru three modes: (1) peritectic reaction, i.e. $L+\delta\rightarrow\gamma$, which occurs at the triple point (L, δ, γ) and proceeds thru the short range diffusion of solute atoms in the liquid ahead of the primary phase and the peritectic phase; due to a high-speed short-range diffusion in the liquid, peritectic reaction can proceed very quickly^[10]; (2) volume diffusion controlled transformation (VDCT), where the solute redistribution at the interface and the long-range compositional change take place; generally such a phase transformation progresses very slowly^[11]; (3) interface controlled massive transformation (MT), which is determined by atomic processes in the immediate vicinity of the interface; unlike volume diffusion controlled transformation, massive transformation can proceed very rapidly, and the bulk chemical composition of product and parent remains invariable during the transformation^[11]. The necessary condition for the occurrence of case (1) is the appearance of undercooling and triple point. For VDCT and MT, the phase transformation kinetics should be considered. Based on JMA phase transformation kinetic theory, TTT curves of the δ/γ solid-state transformation of Fe-4.33at.%Ni alloy were calculated; detailed calculated procedures were available in Appendix. As shown in Figure 3, if the cooling rate of the system $\dot{T} < 5$ K/s, δ/γ solid-state transformation will occur through the mode of VDCT, and *vice versa*. Also, according to Figure 3, the cooling rate needed for suppressing the δ/γ transformation has the order of 10^4 K/s. It was said that in the present experiment, the δ/γ transformation cannot be suppressed.

Figure 3 TTT curves of δ/γ solid-state transformation of Fe-4.33at.%Ni alloy.

In terms of the XRD analyses, it was found that in the achieved range of ΔT , the as-solidified microstructures all belong to b.c.c lattice structure, see Figure 4. Because both martensite and α -ferrite possess b.c.c structure, to further clarify the detailed phase constitutions of the as-solidified microstructures, TEM observation was carried out. The results indicate that at low ΔT , the as-solidified microstructure exhibits the single martensite characteristic (see Figure 5);

whereas, at high ΔT , it consists of lathy martensite (Figure 6(a)) and a few α ferrite particles (Figure 6(b)). Obviously, martensite results from the transformation of $\gamma \rightarrow$ martensite, and those α ferrite particles are the RT products of the untransformed δ phase from δ/γ transformation. On this basis, it was concluded that in the achieved range of ΔT , δ/γ transformation always takes place.

Figure 4 XRD pattern of the as-solidified microstructure.

As mentioned above, it has been clearly demonstrated that the change of ΔT can drastically alter the solidification behavior of undercooled Fe-4.33at.%Ni alloy. It was believed that such a change is bound to predominately influence the subsequent δ/γ transformation. In the following section, taking ΔT^* as a critical point, the δ/γ transformation behavior of undercooled peritectic Fe-4.33at.%Ni alloy will be separately illuminated.

Figure 5 TEM bright field image of the microstructure at $\Delta T = 47$ K, showing the typical martensite characteristic.

(i) Undercooling dependent δ/γ transformation pathways. For undercooled Fe-4.33at.%Ni alloy, when $\Delta T < \Delta T^*$, after primary solidification of δ phase, the peritectic reaction will take place and form a peritectic layer on the as-solidified δ phase. Following the peritectic reaction, the remaining liquid will be subject to an epitaxial growth on the as-formed layer and result in the second recalescence (see Figure 2)^[9,12]. Once the peritectic layer forms, the liquid and primary phase will be separated. In the following, on the one hand, the peritectic layer will solidify towards the liquid; on the other hand, it will grow into the inner region of the grain thru δ/γ solid-state transformation. During this course, because of a high system temperature and a small rate of tempera-

ture change, the phase transformation kinetics tends to favor the VDCT controlled δ/γ solid-state transformation. Such a conclusion can be confirmed by the Ni element distribution in the as-solidified microstructures. Through the EPMA analysis, it was found that for the microstructures of undercooled Fe-4.33at.%Ni alloy with $\Delta T < \Delta T^*$, between the inner and outer region of a grain, a clear solute transition layer (STL) was observed and the Ni element distribution in STL exhibits a clear VDCT controlled segregation pattern, see Figure 7(a). Obviously, the STL should be the product of the initial stage of δ/γ solid-state transformation. Controlled by VDCT, the Ni concentration in STL exhibits a gradual transition characteristic. Once the solidification finishes, $\dot{T}$ is totally controlled by the experimental heat extraction condition. In the present experiment, after solidification, $\dot{T} \approx 20$ K/s. The calculated TTT curves (Figure 3) imply that the remaining δ phase should transform into γ phase thru MT. Also, the Ni element distribution (see Figure 7) strongly suggests that the δ/γ transformation of the inner part of a grain should be dominated by the MT mechanism. It should be pointed out that with ΔT increasing, the volume fraction of primary δ phase will increase continuously. Therefore, at a relatively large ΔT , the δ/γ transformation cannot be completed, which leads to the existence of a few untransformed α ferrite particles in the as-solidified microstructures. From the aforementioned analysis, in the case of $\Delta T < \Delta T^*$, in the initial stage, the δ/γ transformation is deemed to be controlled by the peritectic reaction and VDCT mechanisms; whereas, as the solidification is completed, such a transformation will be dominated by the MT mechanism.

Figure 6 TEM bright field images of the microstructure at $\Delta T = 207$ K, (a) showing the morphology of α ferrite particle, and (b) showing the morphology of martensite matrix.

If ΔT exceeds ΔT^* , the peritectic reaction will be suppressed and the whole liquid will solidify as δ phase. So, during solidification, δ/γ transformation will not take place. After solidification, $\dot{T} \approx 20$ K/s, δ/γ transformation thus will be totally controlled by MT mechanism. Furthermore, with ΔT increasing, the solute trapping effect incorporated in the rapid solidification will increase abruptly, which can make the level of solute segregation decrease predominantly. As pointed out by Massalski et al.^[13], a high degree of solute uniformity in the parent phases will favor MT. Therefore, a low level of solute segregation in the undercooled Fe-4.33at.%Ni alloy in the range of $\Delta T > \Delta T^*$ will also promote the occurrence of δ/γ massive transformation. Certainly, in such a range of ΔT , the δ/γ transformation can proceed thru MT, which is mainly determined by its phase transformation kinetics. On the other hand, because all of the solidification products are δ

phase and δ/γ transformation totally proceeds in the solid state, under the present experimental conditions, the δ/γ transformation cannot be completed, which causes some untransformed α ferrite particles to be retained in the RT microstructures. It was interesting to find that the composition of these particles is nearly the same with the parent matrix phase (martensite). It was indicated that during the transformation the compositional change does not occur, which further confirms that in the case of $\Delta T > \Delta T^*$, the δ/γ transformation is controlled by the MT mechanism.

Figure 7 The Ni concentration profiles measured with EPMA (line in the insert micrograph) in the microstructures of undercooled Fe-4.33at.%Ni alloys. (a) $\Delta T = 30$ K; (b) $\Delta T = 207$ K.

In summary, the δ/γ transformation pathways in the undercooled Fe-4.33at.%Ni alloys are determined by ΔT . With respect to ΔT^* , δ/γ transformation will proceed thru two different pathways (see Figure 8). For $\Delta T < \Delta T^*$, in the initial stage of transformation, it is controlled by the peritectic reaction and VDCT mechanisms and completed by MT after solidification; while in the case of $\Delta T > \Delta T^*$, such a transformation totally proceeds in the mode of MT.

(ii) Influence of undercooling on δ/γ transformation course. The above kinetics analysis implies that the δ/γ transformation occurring in non-equilibrium microstructures is remarkably different from the common solid-state transformation. The real phase transformation course depends not only on the cooling rate but also on the solidification behavior and parameters, where the most dominative factor is ΔT .

For Fe-4.33at.%Ni alloy, subject to the near-equilibrium solidification, the solidification process will comply with the lever rule of the equilibrium phase diagram, i.e. δ phase solidifies as the primary phase, and when the system temperature decreases to the peritectic plateau temperature, all of the as-formed primary δ phase will transform into the peritectic γ phase thru the peritectic reaction. It means that under the near-equilibrium condition, the δ/γ transformation should be totally controlled by the peritectic reaction mechanism. Certainly, such a case cannot occur in the undercooled melts. For the undercooled Fe-4.33at.%Ni alloy with $\Delta T < \Delta T^*$, the volume fraction of the δ phase follows a positive linear law with ΔT increasing. Hence, as the peritectic reaction takes place, the volume fraction of the primary δ phase and liquid phase will not satisfy the lever rule of the equilibrium phase diagram. This means that the δ/γ transformation cannot be completed through the peritectic reaction mechanism, and part of the δ phase must change into the γ phase through solid-state transformation. As illuminated above, during the second recalescence, after complete coating of the primary δ phase by the peritectic γ phase during the peritectic reaction, the δ/γ transformation will proceed in the mode of VDCT. Also, such a transformation is known to

be driven by the compositional difference between the outside (in contact with the liquid phase) and the inside (in contact with the δ phase) of the layer of peritectic γ phase. In the undercooled melt, influenced by the solute trapping effect incorporated in the rapid solidification, the solidification segregation will decrease with ΔT increasing, which will lead to the decrease of the driving force for VDCT, and in turn the decrease of the transformation fraction with respect to VDCT. As a consequence, the thickness of STL was found to be decreased with ΔT increasing (see Figure 9).

Figure 8 δ/γ transformation pathways of the undercooled Fe-4.33at.%Ni alloy.

Figure 9 The thickness of STL in the microstructures of undercooled Fe-4.33at.%Ni versus ΔT .

Obviously, the increase of primary δ phase and the decrease of the driving force for VDCT with ΔT increasing will raise the transformation fraction corresponding to MT. When $\Delta T > \Delta T^*$, the solidification of undercooled Fe-4.33at.%Ni alloy completely deviates from the equilibrium, and the liquid phase totally transforms to the δ phase. In this case, the peritectic reaction is suppressed, and the δ/γ transformation proceeds thru a manner, i.e. MT, which cannot absolutely occur under the equilibrium condition. Certainly, the occurrence of massive δ/γ transformation is mainly determined by the phase transformation kinetics, namely the cooling rate after the solidification. The above analyses imply that the change of ΔT can not only result in the shift of the mode of δ/γ transformation, but also change the volume fraction controlled by each transformation mechanism.

4 Conclusions

In the present work, the δ/γ transformation in the non-equilibrium microstructures of undercooled Fe-4.33at.%Ni alloy was systematically investigated. The main conclusions are as follows:

(1) Subject to $\Delta T < \Delta T^*$, in the initial stage of phase transformation (i.e. during the second recalcrescence), the δ/γ transformation is controlled by the mechanisms of the peritectic reaction and VDCT. Once the solidification finishes, such a transformation will proceed in a mode of MT; while for $\Delta T > \Delta T^*$, the δ/γ transformation is totally dominated by MT mechanism.

(2) The increase of ΔT can change not only the δ/γ transformation pathways but also the transformation fraction corresponding to each mechanism.

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Appendix: Calculation procedure of TTT curves for δ/γ solid-state transformation of undercooled Fe-4.33at.%Ni alloy

The kinetics of δ/γ solid-state transformation of undercooled Fe-4.33at.%Ni alloy can be described by TTT (Temperature-Time-Transformation) curve. The time-dependent volume fraction during phase transformation, $x(t)$, can be expressed by the well-known Johnson-Mehl-Avrami-Kolmogorov equation^[11]:

$$x(t) = 1 - \exp\left[-\frac{\pi}{3}u^3I_{ss}t^4\right], \quad (1)$$

where I_{ss} , u , and t are steady state nucleation rate, growth rate, and time, respectively.

According to the classical nucleation theory, I_{ss} can be expressed as^[11]

$$I_{ss} = N_0 \exp\left(-\frac{\Delta G^*}{k_B T}\right), \quad (2)$$

where N_0 is a constant ($\approx 10^{30}$)^[11], ΔG^* is the critical nucleation work, k_B and T is Boltzmann constant and temperature, respectively. ΔG^* can be given as

$$\Delta G^* = \frac{16\pi\sigma^3}{3(\Delta G_v)} f(\theta), \quad (3)$$

where σ is interfacial energy, $\Delta G_v (= \Delta S_{\delta \rightarrow \gamma} \Delta T_{tr})$ is the Gibbs free energy difference between δ phase and γ phase with $\Delta S_{\delta \rightarrow \gamma}$ as the entropy change during phase transformation, ΔT_{tr} is the undercooling prior to phase transformation, and $f(\theta)$ is the catalytic factor which can be calculated using the following equation:

$$f(\theta) = \frac{2 - 3\cos\theta + \cos^3\theta}{4}, \quad (4)$$

where θ is the contact angle.

Previous research indicates that for δ/γ solid-state transformation, the nucleation of γ phase generally takes place at the boundary between two parent grains. Then, $\cos\theta = \sigma_{\delta/\delta}/2\sigma_{\delta/\gamma}$, with $\sigma_{\delta/\delta}$ being the interfacial energy between two parent δ grains and $\sigma_{\delta/\gamma}$ the interfacial energy between δ phase and γ phase. Using the physical parameters listed in Table 1, $\cos\theta$ can be calculated as 0.585 and in turn $f(\theta)$ as 0.1113.

For VDCT, u can be expressed as^[11]

$$u = D/(2(Dt)^{1/2}), \quad (5)$$

$$D = D_0 \exp\left(-\frac{Q_D}{kT}\right), \quad (6)$$

where D is the diffusion coefficient, D_0 the pre-exponential factor for diffusion, and Q_D the activated energy for diffusion. For MT, u can be given as

$$u = u_0 \exp\left(-\frac{Q_N}{kT}\right) \left(1 - \exp\left(-\frac{\Delta G_v}{kT}\right)\right), \quad (7)$$

where u_0 is the pre-exponential factor for growth, Q_N the activation energy for the transfer of atoms through the parent phase/new phase interface. u_0 for Fe-Ni alloy can be estimated as about 10^6 m/s from the Wilson's work^[14].

Table 1 Physical parameters essential for the calculation^[15–20]

Parameters	Values
$\sigma_{\delta/\delta}$ (J·m ⁻²)	0.468
$\sigma_{\delta/\gamma}$ (J·m ⁻²)	0.4
Q_D (kJ·mol ⁻¹)	251.9
Q_N (kJ·mol ⁻¹)	126
$\Delta S_{\delta \rightarrow \gamma}$ (J·mol ⁻¹ ·K ⁻¹)	0.64
D_0 (m ² ·s ⁻¹)	8.2×10^{-6}
T_{tr} (K)	1780

Assuming $x(t) = 0.001$, the TTT curves with respect to VDCT and MT can be calculated using eqs. (1)–(7), as shown in Figure 3.

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