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# A triple comparative study of primary dendrite growth and peritectic solidification mechanism for undercooled liquid $Fe_{59}Ti_{41}$ alloy

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The rapid solidification kinetics of undercooled hypoperitectic $Fe_{59}Ti_{41}$ alloy was quantitatively investigated by electrostatic levitation (ESL) and electromagnetic levitation (EML) methods combined with a high-speed photography technique. The maximum undercoolings $\Delta T$ obtained by ESL and EML methods were 200 K (0.12 $T_L$) and 315 K (0.19 $T_L$), respectively. Double recalescence processes corresponding to the primary dendrite growth and subsequent peritectic reaction were recorded at various undercoolings. The dependence of primary dendrite growth velocity $V$ on the undercooling $\Delta T$ satisfied a double-exponential relation. A longest incubation time and a highest undercooling of peritectic reaction were experimentally determined at the same critical undercooling of about 86 K. In contrast, the peritectic reaction time decreased linearly with enhanced $\Delta T$. As $\Delta T$ rised in ESL and EML experiments, primary $Fe_2Ti$ phase successively appeared as well-defined coarse dendrites, greatly refined dendrites and finally evolved into a maze-like morphology composed of vermicular dendrites. Meanwhile, the layer thickness and volume fraction of peritectic FeTi phase remarkably reduced, which suggested that peritectic reaction was suppressed to some extent. The solid solubilities of $Fe_2Ti$ and FeTi phases were significantly extended during rapid solidification. As another comparison, drop tube experiment was also conducted to explore the solidification behaviors under larger undercoolings and cooling rates. A mechanism transition of 'peritectic solidification → metastable coupled-growth between primary and peritectic phases' was observed with decreasing alloy droplet size.



## 1. Introduction

In recent years, the peritectic alloys [1–3] have attracted much attention because of their potential applications in manufacturing industries. For typical peritectic alloys, they usually undergo a peritectic reaction during cooling. The peritectic alloy solidifies with the preferential nucleation of primary phase that reacts with the remaining liquid to form the peritectic phase. Simultaneously, the primary phase is consumed [4,5]. As is well known, the peritectic reaction is controlled by atom interdiffusion across the product phase and cannot completely proceed. Up to now, the attention to the solidification kinetics of peritectic-type alloys is still limited. Therefore, it is highly desirable to investigate the solidification behaviors and microstructure characteristics of undercooled liquid peritectic alloys especially under the containerless processing conditions.

Extensive investigations about the rapid solidification of undercooled liquid peritectic alloys mainly focused on two aspects [6–9]. Firstly, it is of great importance to determine the relationship between the dendritic growth velocity and undercooling, which contributes to revealing the dendritic growth kinetics of primary phase before the peritectic reaction [10]. By using a photodiode technique [11,12] or a high-speed camera system [13,14], the dendritic growth velocity could be experimentally measured under the glass fluxing [14], electro-magnetic [10] and electrostatic levitation [15] conditions. In most cases, the reported growth velocity exponentially rises with undercooling without limit [10,12]. Nevertheless, this should not be the case due to the thermally activated process of melt crystallization in substantially undercooled state. As a consequence, there should be a maximum growth velocity at a certain undercooling, beyond which the growth velocity slows down. Because of the difficulties for obtaining substantial undercoolings, a highest growth velocity is rarely observed at a critical

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undercooling. In addition, the transition mechanisms of dendritic growth kinetics can be theoretically analyzed by the current dendritic growth models, such as the LKT/BCT and phase-field models [16–19]. The second aspect is to understand the peritectic reaction characteristics after the first recalescence for the formation of primary phase. So far, it is practically impossible to provide a quantitative determination for the peritectic incubation time and reaction time. Taking into account that the peritectic reaction is a relatively sluggish process, the forced convection and cooling rate play an important role in determining the peritectic reaction characteristics [20–22], which have rarely been involved up to now. Though individual work has been performed on the solidification behavior of undercooled peritectic alloys, much work still needs to be done to clarify the issues mentioned above.

The microstructure of peritectic solidification is characterized by the peritectic phase growing around the primarily precipitated phase. Recent investigations showed that the solidification mode of highly undercooled peritectic alloys can be drastically altered in contrast with that in equilibrium cases [23,24]. Once the undercooling exceeds a critical value, the peritectic phase can form directly from the undercooled melt [6] by suppressing the nucleation of primary phase and subsequent peritectic transformation. In another case, if the incubation time for the occurrence of peritectic reaction is longer than the time required for the complete solidification of primary phase, the peritectic reaction can also be thoroughly restrained and the solidification microstructure is only composed of primary phase [23]. The possibilities of coupled growth mode for peritectic-type alloys have been discussed over the years [3,25–28]. So far, the coupled growth phenomenon of primary and peritectic phases was merely observed within directionally solidified peritectic alloys. There are few experimental evidences for metastable coupled-growth in substantially undercooled peritectic alloys by now. Accordingly, it is of great significance to explore the possibility of metastable coupled-growth phenomena in highly undercooled peritectic alloys.

The electrostatic levitation (ESL) and electromagnetic levitation (EML) methods have been frequently applied to quantitatively measure the growth velocity of undercooled liquid alloys. Owing to the forced cooling of flowing helium gas, EML can give a chance to study the dendritic growth kinetics of primary phase under substantially high undercooling condition. However, the poor stability of electromagnetically levitated bulk samples after the first recalescence for the formation of primary phase brings considerable difficulties in determining the peritectic reaction kinetics. Thus, the ESL method could be used to not only demonstrate the measurement reliability of growth velocity in the EML experiments, but also determine the peritectic reaction time due to its excellent stability [15]. It contributes to the quantitative studies of the peritectic reaction characteristics. The maximum undercooling of tiny droplets during drop tube processing is larger than that of bulk samples processed by the levitated methods [6]. To make clear whether the direct nucleation of peritectic phase or metastable coupled-growth occurs at substantially large undercoolings, the drop tube (DT) experiments should be also carried out and compared with that under the levitation conditions. During peritectic reaction, the forced convection inside the electromagnetically levitated alloys and the high cooling rate of freely falling alloy droplets are crucial to the microstructures of peritectic solidification. Hence, the primary dendrite growth and peritectic solidification mechanism of undercooled liquid peritectic alloys can be further understood by designing the comparative experiments under various containerless processing conditions.

Most of the previous studies were mainly concentrated on the peritectic systems with a narrow temperature interval between liquidus temperature and peritectic temperature, while there is little related work on the peritectic alloy system with a broad temperature interval, such as Fe-Ti alloy system [29]. Taking $Fe_{59}Ti_{41}$ hypoperitectic alloy as an experimental example, the purpose of this work is to explore the dendritic growth kinetics of primary $Fe_2Ti$ phase and the succedent peritectic reaction characteristics of $L + Fe_2Ti \rightarrow FeTi$ by using the ESL, EML and drop tube (DT) methods. Special attention is paid to the growth-mechanism transition from peritectic transformation to metastable coupled-growth under substantial undercoolings.

## 2. Experimental procedures

### 2.1. Electrostatic levitation experiment

The solidification experiments were firstly performed by the electrostatic levitation (ESL) method. The master alloys of $Fe_{59}Ti_{41}$ were made from high purity elements of 99.999% titanium and 99.999% iron in an ultrahigh vacuum arc-melting furnace. An alloy sample of about 3 mm in diameter was levitated in an ultrahigh vacuum environment, which reaches up to $10^{-5}$ Pa. Afterwards, the electrostatically levitated alloy was heated by a continuous wave SPI SP300 fiber laser, and then cooled by turning off the laser system. The alloy temperature was simultaneously monitored by a CellaTemp PA 40 single-color pyrometer with an absolutely accuracy of $\pm 5$ K, which was calibrated using the liquidus temperature of $Fe_{59}Ti_{41}$ alloy. A series of melting and cooling cycles were repeated until the confirmation of consistent heating and cooling curves. The dendritic growth velocity of primary $Fe_2Ti$ phase and the subsequent peritectic reaction time were obtained by a photoelectric detector system and a high-speed camera system. These recalescence images in $256 \times 192$ pixels were recorded at a frame rate of 2000 frames per second (fps), which allows a quantitative determination of the dendritic growth velocity and peritectic reaction time mentioned below.

### 2.2. Electromagnetic levitation experiment

Considering that the obtained maximum undercooling of liquid $Fe_{59}Ti_{41}$ alloy under the electromagnetic levitation (EML) condition was higher than that of electrostatically levitated $Fe_{59}Ti_{41}$ alloy, the solidification experiments were also conducted by EML method as a comparison. Before the experiments, the ultrahigh vacuum chamber was evacuated to $10^{-5}$ Pa at first and then backfilled with argon gas to $10^5$ Pa. Afterwards, a sample of about 6 mm in diameter was levitated and meanwhile melted in a containerless state. The levitated sample was cyclically melted and overheated by approximately 200 K for about 5–10 s to evaporate oxygen from the sample. Subsequently, it was cooled with flowing helium gas to achieve a desirable undercooling. The sample temperature was monitored by an infrared pyrometer, and the recalescence time was measured by a photoelectric detector and a high-speed camera.

### 2.3. Drop tube experiment

The maximum undercooling of small droplets processed by drop tube (DT) method is much larger than that of bulk samples under both EML and ESL conditions. The solidification experiments of $Fe_{59}Ti_{41}$ alloy were also carried out inside a 3 m drop tube, expecting to investigate the solidification microstructures at the condition of ultrahigh undercooling as an extension. Each alloy had a mass of 2 g and was placed inside a $\Phi 16$ mm $\times$ 150 mm quartz tube, which had a small orifice about 0.3 mm in diameter at the bottom. The quartz tube was then installed on the top of drop tube. Before the experiment, the drop tube was evacuated to $2 \times 10^{-5}$ Pa at first, and then backfilled with a mixture of helium gas