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Ternary eutectic and peritectic solidification of undercooled liquid Fe–Mo–Si alloys



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ABSTRACT

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Keywords: Microstructure Phase transformation Rapid solidification Undercooling Liquid ternary Fe–6.5%Mo–18.5%Si eutectic and Fe–4%Mo–19%Si peritectic alloys were highly undercooled and rapidly solidified under containerless and microgravity condition inside the drop tube. In this situation, the cooling rate and undercooling of the alloy droplets increased obviously when their diameters decreased, and the alloy droplets exhibited maximum undercoolings up to 184 K and 468 K respectively. For the Fe–6.5%Mo–18.5%Si eutectic alloy, the ternary eutectic is composed of Fe₃Si, Fe₂Si and Fe₅MoSi₄ intermetallic phases. With the decrease of the droplet diameter, the volume fraction of the ternary (Fe₃Si + Fe₂Si + Fe₅MoSi₄) eutectic increases and the morphology of the ternary eutectic vary from lamellar structure to granular structure. In the Fe–4%Mo–19%Si peritectic alloy, the primary phase is Fe₂Si intermetallic phase, and the peritectic structure consists of Fe₃Si and FeSi intermetallic phases. With the increase of the undercooling, the primary phase vanishes, the peritectic transformation nearly accomplishes, and the growth of (Fe₃Si + FeSi) peritectic phases transform from faceted blocks into non-faceted granules.

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1. Introduction

The microstructure evolution of rapidly solidified alloys is a key research aspect of materials science and condensed matter physics [1]. The physical properties of materials are depending on the solidification process and the final microstructure [2]. Most actual applied materials are multicomponent alloys whose solidification processes are very complex and concern the competitive growth of different phases. Thus it is of great importance to research the rapid solidification of multicomponent alloys. In the past few years, more and more research activities were dedicated to multiphase solidification of ternary and multicomponent alloys [3–7].

Ternary eutectic transition $(L \rightarrow \alpha + \beta + \gamma)$ always occurs in the solidification of ternary and multicomponent alloys, but its mechanism is not fully understood [8–12]. One kind of peritectic transformation $(L + \alpha \rightarrow \beta + \gamma)$ is often found in the solidification of multicomponent alloys, as viewed from the reactant, it is similar to a binary peritectic transformation, but from the resultant it is similar to a binary eutectic transition. The mechanism of this transformation is not very clear and under active research [13–16]. Drop tube can simulate the space environment of "containerless, microgravity, high vacuum", thus the heterogeneous nucleation due to the container wall can be avoided, and rapid solidification can be realized [8,17].

The Fe–Mo–Si system has a series of complicated phase transformations including ternary eutectic and peritectic, and the reactants and the products concern many intermetallic phases [18]. The alloy systems of Fe–Mo, Fe–Si and Mo–Si have excellent properties such as high permeability and high yield strength, thus they are widely used in industry, and have been intensively studied by many scientists [19–21]. However, the research on rapid solidification of Fe–Mo–Si ternary system is rarely found [22], and the mechanisms of ternary eutectic transition and peritectic transformation are not well understood. In this work, the solidification features of ternary Fe–6.5%Mo–18.5%Si eutectic and Fe–4%Mo–19%Si peritectic alloys have been investigated by a 3 m drop tube. The solidification and the microstructure evolution are discussed, aiming at revealing the solidification mechanisms of ternary eutectic and peritectic transformations.

2. Experimental procedure

Two Fe–Mo–Si ternary alloys were prepared from pure Fe (99.999%), pure Mo (99.99%) and pure Si (99.999%) by arc melting method under argon atmosphere. Each sample had a mass of about 1 g. The rapid solidification of the alloys was accomplished in the 3 m drop tube. In an experiment, the sample was placed in a quartz tube with diameter of 16 mm, length of 150 mm and a ϕ 0.3 mm nozzle at its bottom. The quartz tube was then installed into the induction coil on the top of the drop tube. The drop tube was evacuated to 2 × 10⁻⁴ Pa and then backfilled with the mixture gas of highly purified Ar and He gases with pressure ratio of 2:1 to about 1 × 10⁵ Pa. After that the sample was melted by induction heating and superheated by about 200 K above its liquidus temperature for several seconds. Finally, the alloy melt was ejected through the nozzle and dispersed into fine liquid

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droplets by exerting a high pressure gas flow of Ar into the quartz tube. Then these liquid droplets solidified rapidly during free fall.

After the experiment, the solidified droplets were collected, crosssectioned, polished and etched by a solution of 2.5 ml $HNO_3 + 5$ ml HF + 5 ml HCl + 90 ml H₂O for 15 s. The thermodynamic properties, phase constitution and microstructures were analyzed by WCR-2D differential thermal analyzer (DTA), Rigaku D/max 2500 V X-ray diffractometer (XRD), FEI Sirion 200 scanning electron microscope (SEM), and Oxford INCA 300 energy dispersive spectrometer fitted in SEM, respectively.

3. Results and discussion

3.1. Equilibrium solidification of eutectic and peritectic Fe-Mo-Si alloys

The studied ternary alloys are marked as E_1 and U_7 in Fig. 1(a) [18], in which E denotes the eutectic point, and U represents the peritectic alloy. It can be deduced from Fig. 1(a) that the equilibrium solidification processes of these alloys are as follows:

E1: Fe-6.5%Mo-18.5%Si: L $\rightarrow \alpha\delta$ Fe + FeSi + τ_1 ,

 U_7 : Fe−4%Mo−19%Si: L + Fe₂Si → αδFe + FeSi.

DTA experiments were conducted to get the information of near equilibrium solidification processes, and the thermograms are shown in Fig. 1.

The equilibrium solidification process of the Fe–6.5%Mo–18.5%Si (E₁) eutectic alloy is presented in Fig. 1(b). It can be found that during cooling there only occurs the eutectic transition $L \rightarrow \alpha \delta Fe + FeSi + \tau_1$

Table 1

Phase constitutions of two alloys determined by XRD and SEM analyses.

Alloy composition	Phase constitution
E ₁ : Fe-6.5%Mo-18.5%Si	Fe ₃ Si, Fe ₅ MoSi ₄ , Fe ₂ Si
U ₇ : Fe-4%Mo-19%Si	Fe ₃ Si, Fe ₂ Si, FeSi

at 1438 K, and the liquidus temperature of the alloy is 1460 K. While Fe–4%Mo–19%Si (U₇) peritectic alloy experiences two liquid–solid phase transformations during cooling, as illustrated in Fig. 1(c). The first is the solidification of primary Fe₂Si phase at 1434 K, the second is the peritectic transformation L + Fe₂Si $\rightarrow \alpha\delta$ Fe + FeSi at 1420 K, and the liquidus temperature of the alloy is 1469 K.

3.2. Microstructure evolution of rapidly solidified alloy droplets

From the results of XRD and SEM analyses, the phase constitutions of the solidified alloy droplets are summarized in Table 1, and the final $\alpha\delta$ Fe phase is determined as Fe₃Si phase. For the Fe–6.5%Mo–18.5%Si eutectic alloy, it can be deduced from Fig. 1(a) that the resultants of the equilibrium solidification process should be Fe₃Si, FeSi and τ_1 phase, but the actual products of the rapid solidification are Fe₃Si, Fe₅MoSi₄ and Fe₂Si intermetallic phases as shown in the XRD pattern of the alloy in Fig. 2. This means that the rapid solidification changes the solidification route. For the Fe–4%Mo–19%Si peritectic alloy, the products of the rapid solidification are Fe₃Si, Fe₂Si and FeSi intermetallic phases, which is shown in Fig. 3. Fig. 3 is the results of EDS analysis of the alloy, (a) shows the concentration patterns of Fe, Mo and Si in the



Fig. 1. Fe-Mo-Si ternary phase diagram and DTA thermograms of the ternary alloys. (a) Phase diagram, [18], (b) Fe-6.5% Mo-18.5% i eutectic alloy, and (c) Fe-4% Mo-19% i peritectic alloy.

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