Coupling effect of adding Al-Ti-C grain refiner and introducing ultrasound to molten melt on the microstructure of 7050 aluminum alloy

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Ultrasound was introduced into 7050 aluminum alloy added Al-Ti-C grain refiners with different mass fractions. Influences of three methods of treating molten melt: applying single ultrasound, single Al-Ti-C grain refiners and the two together, on nucleation in the solidification process were studied through the analysis of the microstructures. The results show the cavitation effect caused by ultrasonic vibration changes the microstructure of Al-Ti-C grain refiners, increases the diffusion and wettability of the TiC clusters, restrains and smashes the growth of $TiAl_3$ clusters, and then enhances the refinement effect of Al-Ti-C grain refiner. Therefore, when the melt was treated synchronously by ultrasound and grain refiner, the finer grains can be obtained than that by a single way.

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1. Introduction

As a traditional method for microstructure refining, adding grain refiners has its irreplaceable place in industry. Also, As a novel way, the injection of ultrasonic energy into molten alloys gains ever-growing attention for its advantages of convenient operation and the property of non-contamination to alloys. In recent years, many scholars have embarked on studying on the influencing factors which are applied to grain refiners with ultrasonic treatments, but most studies are aiming at Al-Ti-B refiners and little progress has been made about the effect of the ultrasonic field on Al-Ti-C refiners. In this experiment, 7050 aluminum alloy is investigated. It is widely used in the spaceflight and aviation for its properties of high strength, high toughness and good resistance to corrosion. As the grain refiner of 7050 aluminum alloy, Al-Ti-B refiner will easily generate alloy pollution of Zr, Cr and Mn [1-4]. Al-Ti-C refiner gains attention initially as the substitute of Al-Ti-B refiner. With the development of the technology of grain refinement, researchers found Al-Ti-C grain refiner would not generate metal atom poisonous, and have more resistance to attenuation [1-3]. Adding Al-Ti-C refiner into the molten alloy has become an important method in industry casting, but there are rarely researches on applying ultrasound and grain refiners to molten alloy simultaneously. So this paper mainly investigate laws of grain refinement when added Al-Ti-C refiners of different contents together with ultrasonic vibration.

2. Experiment

2.1 Experimental material

The material used in this experiment was 7050 aluminum alloy. Its chemical compositions are shown in Table 1.

Table 1. The components of 7050 aluminum alloy.

Main components	Zn	Mg	Cu	Zr	Fe	Si	Ti	Al
Mass percentage（wt. %）	6.56	2.37	2.60	0.10	0.09	0.06	0.018	Bal

2.2 Experimental setup

The experimental schematic diagram was shown in Fig. 1. The main devices used in this experiment include a set of ultrasonic vibration system which consisted of a piezoelectric ceramic transducer, a steel ultrasonic amplitude transformer and a Φ50 mm × 110 mm titanium alloy radiator, a set of homemade digital ultrasonic generator which can be adjusted with a output power of 0 to 2000w continuously and track frequency between 14 kHz and 22 kHz automatically and some other auxiliary equipments: electric resistance heating furnace, temperature control and detection system, clay-graphite crucible with a inner diameter 180 mm, 18 mm thick and 200mm high, thermal couple, counter balance, Leica metallographic microscope and so on.

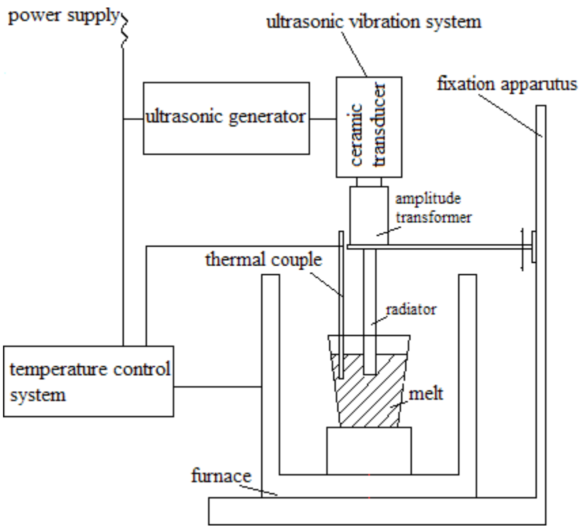

Fig. 1. Schematic diagram of ultrasonic casting experimental apparatus.

2.3 Experimental procedure

10Kg of Aluminum blocks were put into the crucible, heated to melt by resistance furnace. When the temperature of metal liquid reached 750 ^{o}C and held for a while. After removing the oxidation layer, Al-Ti-C grain refiner with 0.1wt.% was introduced and stirred for 90 seconds so that the grain refiner could be fully melted and mixed uniformly with the molten alloy. Then ultrasonic vibration was introduced. Ultrasound was introduced through the acoustic radiator being inserted 25mm deep into the centre of the aluminum melt. The ultrasonic power was 1000W and ultrasonic processing time was 10 minutes as well as the frequency was 20kHz. After the ultrasonic treatment, the crucible was taken out of the furnace and cooled in the air until the melt solidifies completely. Change the quantity of the grain refiner and take the same technological parameters of ultrasonic treatments. The mass fraction of the grain refiners adding into the melt were 0.2wt.%, 0.5wt.%, 1wt.%, and 1.5wt.% respectively. The experiment was executed according to the above steps in order. And then, only change the quantity of grain refiners, and repeat the experiments without ultrasonic treatment. All the ingots obtained from the experiments were classified and labeled according to table 2. Analytical samples were obtained from the middle of the ingots. All the samples were treated through coarse grinding, fine grinding, polishing and corrosion, and then were observed by metallographic microscope to investigate the microstructure. The grain size of each position was measured through using a 400 mm mesh to get the grain numbers in six visual fields and calculate the average diameter of the grain.

Table 2. Classification of the sample.

The sample label	0	1	2	3	4	5	6	7	8	9	10	11
Al-Ti-C mass fraction (wt. %)	0	0.1	0.2	0.5	1.0	1.5	0	0.1	0.2	0.5	1.0	1.5
Ultrasound was introduced or not	N	N	N	N	N	N	Y	Y	Y	Y	Y	Y

3. Results and analysis

3.1 The effect of adding single grain refiner on the microstructure

Fig. 2 showed the microstructure and grain size of casting ingots obtained by adding different mass fractions of Al-Ti-C grain refiner. It can be seen from Fig. 2 that the grain size of the aluminum ingots decreased with the increase of the amount of grain refiners until the mass fraction reached 0.5wt.%. At this time, the grain size was minimized. That was the best refining effect. While the amount of grain refiner increased continually the refining effect was weaken and maintained a stable state at last.

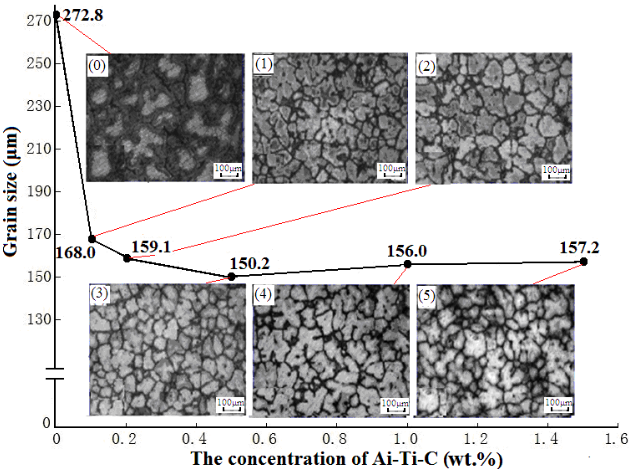

Fig. 2. Grain size and Microstructure of casting ingots obtained by adding single grain refiners of (0) 0wt.%, (1) 0.1wt.%; (2) 0.2wt.%; (3) 0.5wt.%; (4) 1wt.% (5) 1.5wt.%.

3.2 The effect of applying ultrasound and grain refiner together on the microstructure

Fig 3 showed the microstructures and grain size of casting ingots obtained by applying grain refiners and ultrasonic vibration together. It was seen that the grain refining effect was reinforced with the increasing of concentration of grain refiners. The best refining effect was achieved when the concentration of Al-Ti-C grain refiners was 1wt.%. However, the grain size would increase when the concentration of refiner was over 1wt.%. Through the contrast (1) to (4) in Fig. 2 and (7) to (10) in Fig. 3, It was seen that the refining effect obtained with ultrasonic treatment was better than that without when the additive amount of the grain refiners was the same.

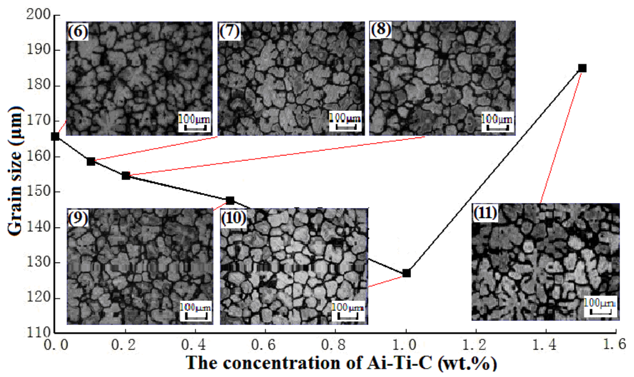

Fig. 3. Grain size and Microstructure of casting ingots obtained by applying ultrasound together with grain refiners of (6) 0wt.%, (7) 0.1wt.%; (8) 0.2wt.%; (9) 0.5wt.%; (10) 1wt.% (11) 1.5wt.%.

3.3 Results analysis

Fig. 4 showed the relationship between grain size and the concentration of grain refiner under two kinds of casting technology. It can be seen from the curves: (1) when ultrasonic treatments and grain refiner were applied to the melt together, the refining effect on the microstructure of casing ingots was better than that only with single ultrasonic treatments or grain refiner; (2) when equally added grain refiner, the refining effect of the microstructure with ultrasonic treatments is much better than the one without; (3) without ultrasonic treatments, and when the amount of the grain refiner was 0wt.% to 0.5wt.%, the ingots were refined effectively. When the amount of the grain refiner was more than 0.5wt.%, refining effect of the refiner was weakened. It was to say the limit value of the amount of the refiner was 0.5wt.% in our test. With ultrasonic treatments, this value increased to 1wt.%. It indicated that the introduction of ultrasonic vibration may improve the nucleating ability of the refiners and enhance the grain refining effect of the casting ingots.

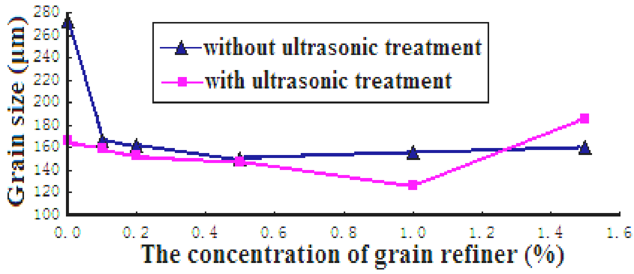

Fig. 4. The curves of grain size and concentration of refiner in different casting technology.

Al-Ti-C master alloys include aluminum matrix, $TiAl_3$ and TiC. In the process of the grain refinement of aluminum alloys, $TiAl_3$ is not a stationary phase from the view of thermodynamics, and it can be dissolved in aluminum melt, so TiC becomes the only sites of heterogeneous nucleation. It shows in the post researches [5, 6] that TiC particle is the cubic structure, the lattice constants rate to Al is 6.69% which means the structure of TiC is similar to that of Al, and gives Al atoms the possibility of heaping on the surface of TiC.

When Al-Ti-C grain refiners were added into the aluminum melt, $TiAl_3$ phase dissolves quickly, but as the wettability of TiC was weak, it was repelled by the aluminum melt and segregated into TiC grain cluster at the grain boundary. $TiAl_3$ which dissolved in the melt accumulates would surround TiC particle group and form the segregation region of Ti atoms. When the concentration of Ti atoms reach 0.15%, peritectic reaction $L_{Al}+TiAl_3 \rightarrow \alpha\text{-Al}$ took place between $TiAl_3$ wrapping around TiC and aluminum matrix so that α-Al nucleation was started [4]. TiC became the nucleating site of α-Al and was engulfed, and then grains of the melt were refined.

The introduction of ultrasonic vibration was regarded as applying an extrinsic energy field in the solidification process of aluminum melt. The cavitation and acoustic streaming effect brought about by ultrasound would influence the solidification and phase changing of aluminum melt. According to the literature [7], the equation of motion of cavitation bubble is

$$R\frac{d^2R}{dt^2}+\frac{3}{2}\left(\frac{dR}{dt}\right)^2=\frac{1}{\rho}\left[P_0\left(\frac{R_0}{R}\right)^{3k}-P_m\right] \qquad (1)$$

Ignoring the surface tension of the cavitation bubbles and assuming the collapsing of the cavitation bubbles is an adiabatic process, we get the equations of the highest pressure and temperature of the bubbles when they collapse [7]:

$$T_{\max}=T_{\min}\left[\frac{P_m(k-1)}{P_v}\right] \qquad (2)$$

$$P_{\max}=P_v\left[\frac{P_m(k-1)}{P_v}\right]^{\frac{k}{k-1}} \qquad (3)$$

Where R is the radius of the cavitation bubbles; R_0 is the initial radius of the cavitation bubbles; T_{min} is the temperature of liquid; P_m is the external pressure when bubbles are closing; P_v is the vapour pressure of the cavitation bubbles, there is mainly hydrogen in aluminum melt, the pressure is assuming 2.33×10^3Pa; k is absolute constant.

When aluminum alloy is in liquid state, its cavitation threshold is about 1MPa. Calculated by Eq.2 and Eq.3, the highest temperature generated by the collapsing of the cavitation bubble is 8.9×10^4K, and the highest pressure is 1.64GPa [8]. These local prodigious temperature and pressure will generate strong shock waves. When the shock waves are shot into the melt, $TiAl_3$ phase is broken up, and the collapsing of the cavitation bubble and the high pressure strengthen this process. At the same time, the cavitation with a large amount of bubbles finally collapsing can cause acoustic energy to be absorbed and dissipated as heat. The heat flow will move from the side of which $TiAl_3$ is stressed to that $TiAl_3$ is not stressed. This deformation generated by the heat flow lags behind the pressure changing and causes thermal loss. When free grains flow in the metal melt, thermal fluctuation and interference effect of the heat flow will cause $TiAl_3$ phase dissolve partially [9-11]. The microstructure of TiC in the conventional casting is cluster structure due to its small size and high interface energy. When ultrasound is introduced into the melt, the shock waves and acoustic stream act on TiC cluster. This external energy make TiC diffuse into the melt. Besides, ultrasound enhances the wettability of TiC clusters [11], provides good conditions for the diffusion of TiC cluster, and makes the distribution of the TiC cluster more uniformly.

The fragmentation and dissolution of $TiAl_3$ phase and the diffusion of TiC cluster decrease the concentration of the segregation of Ti atoms, restrain the growth of the grains which take TiC as the nucleating site, refine the structure of the aluminum alloys, and make it more uniformly. The mechanism which ultrasound make the $TiAl_3$ phase more uniformly lies in that the prodigious local pressure of cavitation generates undercooling of the melt instantaneously decreases the critical nucleation radius, increase the nucleation rate, and then refine the grains.

4. Conclusion

By adding single grain refiner, the best grain refinement is obtained when the additive amount of Al-Ti-C was 0.5wt%. However, the best refining effect is achieved with 1.0wt% Al-Ti-C by applying ultrasound synchronously. It is worth noting that grain refining effect gain in this technology is better than in other technology of applying single ultrasound or grain refiner. In the casting of 7050 aluminum alloy, the introduction of ultrasound improves the microstructure of Al-Ti-C grain refiners, reduces the concentration of segregation of Ti atoms, makes the distribution of the nucleation sites of TiC more uniformly, observably increases heterogeneous nucleation rate, and then enhances the effect of grain refinement.

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