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Ultrasonic Treatment of the 304 Stainless Steel Melt

Jinwu KANG, Xiaopeng ZHANG,* Yisen HU, Jiyu MA, Yongyi HU and Tianyou HUANG

Key Laboratory for Advanced Materials Processing Technology, Ministry of Education, School of Materials Science and Engineering, Tsinghua University, Beijing, 100084 China.

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Ultrasonic treatment of the melt metals is a hot research topic; however, it is hard to introduce ultrasound into liquid steel because of the requirement of resistance to high temperature and erosion in the melt. In this paper, the author proposed a new method to introduce ultrasound into liquid steel. The metal to be treated is cast together to the tip of the booster. During the test, the booster is placed upward with the metal to be treated connected on the top as a free end, which is melt by induction coils. The booster which generates ultrasound, by this way, acts on the melt pool. The determination of the length of the booster under this situation is given. The effect of ultrasonic treatment on the melt of 304 stainless steel is studied. The microstructure of the treated area is significantly refined and equi-axed grains are obtained; the fragmentation of inclusions occurs due to the ultrasonic treatment; the mechanism of the fragmentation of the dendrite and inclusions is discussed.

KEY WORDS: ultrasound; booster; stainless steel; refinement; microstructure.

Ultrasonic treatment has come into wide use in many fields since the application in medicine at the beginning of last century. In the field of foundry, ultrasonic treatment can refine the microstructure and reduce segregation of the metals and alloys.[1–11] The microstructure formed during the solidification has a direct impact on the properties of metal materials, refining the metal microstructure during solidification can evidently improve the mechanical properties of metal materials, which will satisfy the performance requirement that conventional casting cannot meet. Nowadays, the application of ultrasonic treatment on metal solidification is becoming more and more common, and substantive research results have been achieved about refining the solidification microstructure. Osawa *et al.*[12] applied ultrasonic treatment in the solidification of AZ91 alloy, as a result, fine and uniform microstructure was obtained, meanwhile, the yield strength and tensile strength were both improved significantly as well; Atamanenko *et al.*[13] carried out experiments to investigate the process of aluminum melt treatment using ultrasound, and determined the effect of cooling temperature on the refinement of microstructure during solidification. However, the melt with high temperature would corrode the booster which plays a very important role in introducing ultrasound into molten metal, for this sake, the research of refining microstructure with ultrasound mainly focused on low-melting-point metal,[14–20] there is relatively less research about the effect of ultrasonic treatment on the high-melting-point metals, such as steels. Liu *et al.*[21] treated T10 steel by using ultrasound introduced from the lateral, and studied the solidification microstructure, segregation, mechanical properties, and anticorrosion property under different powers. Although this method avoided corrosion of the booster, the loss of energy was still considerable, leading to low efficiency of ultrasonic application. Li *et al.*[22] have done research on the material properties of the booster, and concluded that Mo–Al_2O_3–ZrO_2 ceramic metal tube which could endure corrosion at high temperature and high- frequency vibration was fitful for introducing ultrasound into steels. But, that method faces the difficulty of processing metal ceramic tube.

An innovative method for introducing ultrasound into the melt is proposed in this paper, the metal to be treated and the booster which is placed upward in the experiment are processed as an unit piece, and the metal is melt by induction coils while ultrasonic vibration is introduced from the bottom of molten metal directly, avoiding erosion and power attenuation to the maximum extent. The refinement of solidification microstructure, the fragmentation of inclusions and the improvement of the mechanical property of 304 stainless steel are researched based on this newly proposed method.

1. Experimental Setup and Method

In the method applied in this experiment, the metal to be treated is processed as an unit piece with the booster, which is placed upward vertically, and the metal is melt by induction coils, after that, ultrasonic vibration is introduced into the melt from its bottom. By this method, the corrosion of the booster by the molten metal is avoided. Its height is adjustable, so, the energy loss is greatly reduced due to direct ultrasonic introduction. The transformer is cooled by recalculating cold water. A quartz tube is used to cover the metal to be treated and the adjacent part of the transformer, which will prevent the dripping of the melt. The schematic

* Corresponding author: E-mail: saviorggg@163.com
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of the experimental setup is shown in **Fig. 1**. Based on this method, the metallographic observation of solidification microstructure along with the inclusions in the melt before and after ultrasonic treatment is carried out, also the mechanism of the refinement of microstructure and fragmentation of inclusions is analyzed.

1.1. Experimental Procedure

The 304 stainless steel, a type of austenitic stainless steel with single-phase as-cast microstructure, is selected for this study. Its chemical composition is listed in **Table 1**. In the experiment, the booster is also made of the same steel; the part to be treated is cast together with the transformer. The length of the metal to be disposed is 36 mm, and that of the booster is 90 mm long. The metal to be treated is 26 mm, the same diameter as the top diameter of the booster. The parameters of the induction coils are frequency 15 kHz, voltage 190 V and current 50 A. The ultrasound power controller is shown in **Fig. 2**(a). The melting of the tip of the booster is shown in Fig. 2(b) and the booster after melting and solidification of the tip is shown in Fig. 2(c).

During the experiment, the top of the booster is heated by the induction coils to make sure the height of molten metal is 36 mm. As the steel is melt, hold it for 5 minutes, and then introduce ultrasound into the molten metal until all the metal solidifies. For all the cases in the experiment, the ultrasonic processing time of each sample is 5 minutes, the temperature of the treated metal varies from 1 600°C to 100°C, so the cooling rate in the experiment is 300 K/min, and the temperature gradient in the mushy zone is 236 K/cm. In this experiment the effect of the ultrasound power on the microstructure of the melt is studied, at levels of 0 W, 150 W, 200 W and 300 W. Then samples were taken from the middle section of the treated part, and the samples were ground, polished and etched for the observation of the metallographic structure using optical microscope and SEM.

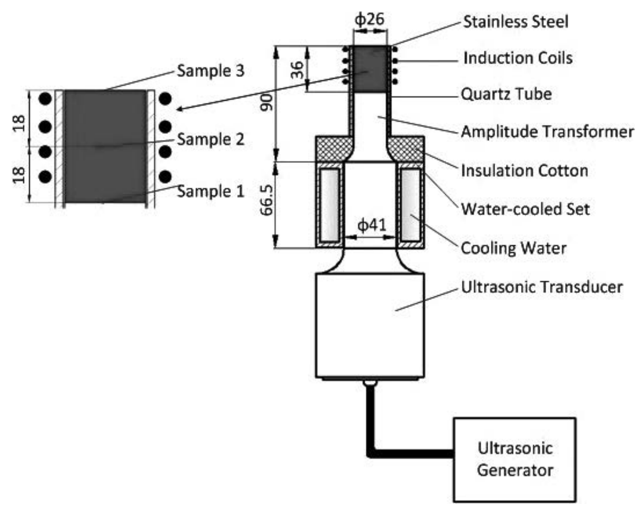


Fig. 1. Schematic of experiment device and positions observed.

1.2. Determination of the Length of Booster

The booster, whose main function is to amplify the amplitude of the vibration part and concentrate the ultrasonic energy on a small area, plays an important role in the ultrasound generating system. The performance and working conditions of the booster have an important effect on the efficiency of the ultrasonic device. Only when the length of booster meets the resonance requirement, the ultrasonic efficiency reaches the maximum value.

In traditional experiment setup, the length of booster is a fixed value suitable for resonance vibration at room temperature. However, when the top of booster is emerged in the molten metal, where high temperature and corrosion change the resonance condition, the resonance vibration cannot happen normally, as a result, the ultrasonic efficiency is reduced obviously.

In general, the resonance requirement is that the length of booster must be an integral multiple of the half wave length of ultrasound. Some researchers[15] show that the acoustic velocity decreases as the temperature rises, The relationship between temperature and the acoustic velocity for the 304 stainless steel can be expressed by:

$$c(T)=\begin{cases} 5816-0.8T, & T<600°\text{C} \\ 6416-1.8T, & 600°C<T<800°\text{C} \\ 5516-0.6T, & 800°C<T<1400°\text{C} \\ 34915.6-21.6T, & 1400°C<T<1466°\text{C} \end{cases} \quad \text{... (1)}$$

Table 1. Composition of 304 stainless steel (mass fraction, %).

C	Si	Mn	P	S	Cr	Ni	Fe
0.08	1.00	2.00	0.05	0.03	18.00	9.00	balance

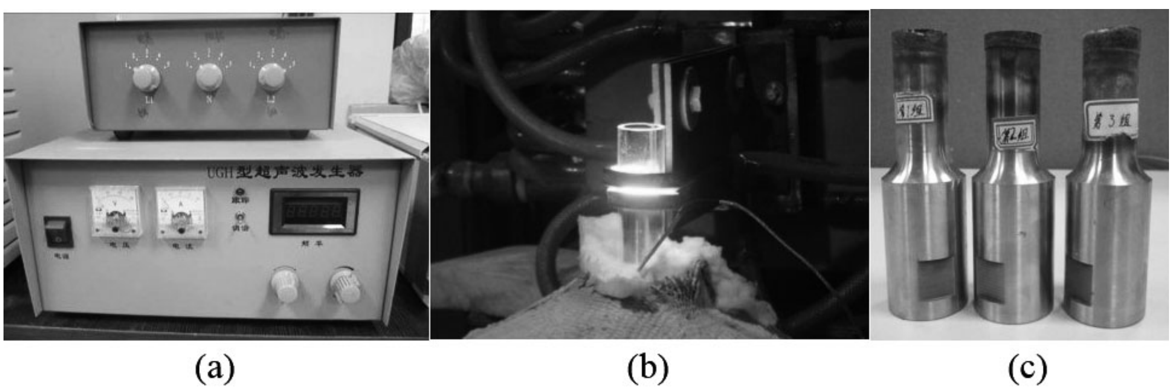


Fig. 2. Experiment of UPM for ultrasound treatment.

where, c stands for the acoustic velocity, a function of temperature T.

Establish coordinate system in the booster as shown in **Fig. 3**, and build the relationship between T and x. According to the above, the wave length can be calculated by:

$$\lambda(x)=\frac{c(x)}{f_P} \quad \text{(2)}$$

where, λ is the wave-length of ultrasound, f_P is the frequency of ultrasound.

Combining Eqs. (1) and (2), the resonance requirement should be:

$$\begin{cases} \dfrac{l}{\lambda}=\dfrac{1}{2} \\ \dfrac{l}{\lambda}=\displaystyle\int_0^l \frac{f_p}{c(x)}dx \\ \displaystyle\int_0^l \frac{f_p}{c(x)}dx=\int_0^{L_1}\frac{f_p}{5\,816-0.8T(x)}dx+\int_{L_1}^{L_1+L_2}\frac{f_p}{6\,416-1.8T(x)}dx \\ \qquad +\displaystyle\int_{L_1+L_2}^{L_1+L_2+L_3}\frac{f_p}{5\,516-0.6T(x)}dx \\ \qquad +\displaystyle\int_{L_1+L_2+L_3}^{L_1+L_2+L_3+L_4}\frac{f_p}{34\,915.6-21.6T(x)}dx \end{cases} \quad \text{(3)}$$

l stands for the length of booster. So, in order to calculate the length of the booster, the temperature at different positions of the booster is needed.

2. Experiment Results

2.1. Macrostructure

The test sample near the solid-liquid interface is obtained to observe the macrostructure after being polished and etched. It can be seen from **Fig. 4** that above the solid-liquid interface, the solidification macrostructure is mainly columnar crystals, whose growing direction is the same as the direction of heat transfer. With the introduction of ultrasound of 200 W, the solidification macrostructure is refined, and the equi-axed structure is obtained. As illustrated in Fig. 4(b), the area where equi-axed structure appears in a limited zone.

2.2. Microstructure

The solidification microstructure in three typical sampling positions under different treating power of 0, 150 W, 200 W, 300 W is illustrated in **Fig. 5**. It can be observed from samples without ultrasonic treatment, that the solidifi-

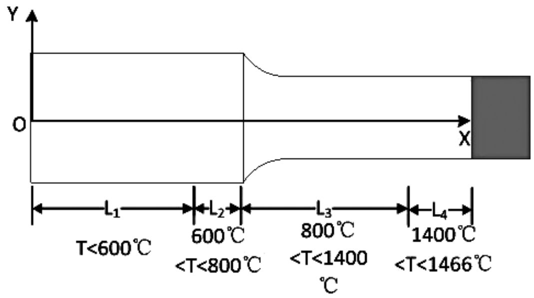
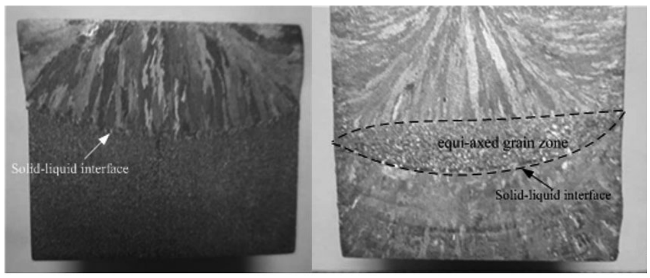
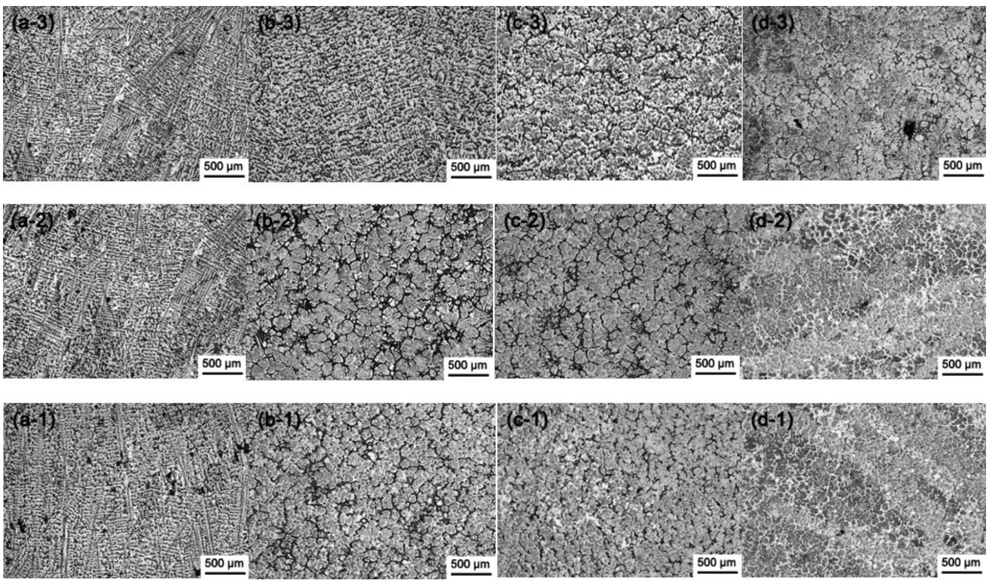


Fig. 3. Coordinate diagram of the booster.



(a) 0W (b) 200W

Fig. 4. The effect of ultrasonic treatment on the macrostructure of 304 stainless steel.



(a)0W (b)150W (c)200W (d)300W

Fig. 5. Solidification microstructure of stainless steel treated with different ultrasound power.

cation microstructure of untreated stainless steel is coarse dendrites. Because there is obvious temperature gradient which is helpful for the growth of dendrites due to the temperature decrease from inside to outside of the molten metal, large quantity of dendrites form under no ultrasonic treatment. In position 1, which is near the bottom of the molten metal, due to the repulsive water-cooling method, the fast solidification causes the occurring of retardation in the growth of dendrites, short staggered dendrites appear as a result.

Ultrasonic treatment can obviously change the solidification microstructure of stainless steel, after the introduction of ultrasound into the molten steel, the solidification microstructure changes from dendrites to equi-axed grains, as illustrated in the microstructures under 150 W, 200 W and 300 W.

It can be observed that there is severe attenuation of ultrasound in the molten metal, for example, when the power is 150 W, the microstructure of the samples from position 1, 2 and 3 is fine-grained equi-axed structure, coarse-grained equi-axed structure and short dendrites respectively. As position 1 is the nearest to the booster, the effect of ultrasonic treatment is the strongest, the sample at position 1 has the smallest average grain size; when the ultrasound arrives at position 2, the power is not so strong as that in position 1 due to the attenuation, and the average size of the equi-axed structure becomes larger; at position 3, where the effect of ultrasound is greatly reduced, the microstructure of sample is mainly short dendrite.

The effect of ultrasonic treatment increases with the rise of power, for example, at position 2, some equi-axed structures are choosed randomly in different samples and the average size is calculated, it is revealed that the average size of equi-axed structure is about 160 μm, 120 μm, 100 μm under the ultrasonic power of 150 W, 200 W, 300 W respectively.

2.3. The Fragmentation of Inclusions

The scanning electron microscope (SEM) was used to observe the appearance of inclusions on the cross section of the metal after being cleaned and polished. **Figure 6** shows the appearance and distribution of inclusions. And the chemical compositions of the matrix and inclusions are listed in **Table 2**. From their compositions it can be deduced that Points 1 and 2 refer to the matrix of stainless steel, points 4 and 7 stand for silicate and manganese oxide respectively, points 3 and 8 the compound inclusions of silicate and manganese oxide, points 5 and 6 the compound inclusion of manganese oxide and manganese sulfide.

Twenty viewing fields of the test sample are selected randomly for observation through SEM, the software "Image-Pro" is applied to obtain the pixel diameter of the inclusions, whose real diameter is determined then. The average diameter of inclusions in the twenty viewing fields is considered to be the mean diameter of inclusions in the test sample.

The size of inclusions treated under different power with the treating time of 5 minutes is illustrated in **Table 3**. In **Table 4**, the size of inclusions treated for different time under the power of 200 W is listed.

It can be concluded from the two charts above that the

Table 2. The contents of the matrix and inclusions.

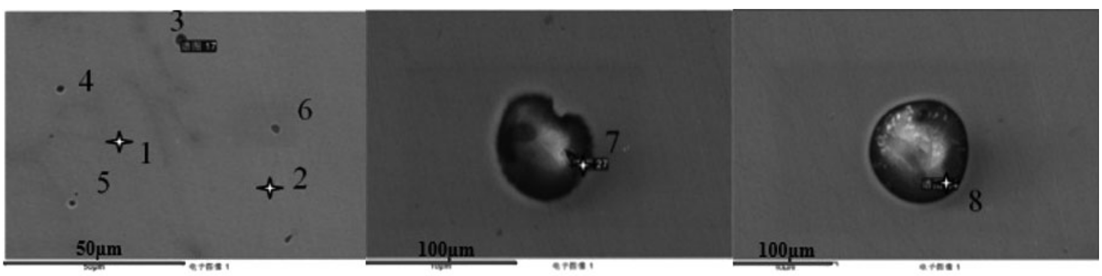
		C	O	Si	S	Cr	Mn	Fe	Ni
matrix	1	6.60	–	0.47	–	17.78	0.63	67.74	6.78
	2	6.66	–	0.39	–	17.10	0.76	66.55	8.55
Inclusions	3	4.72	30.10	13.42	1.16	13.88	13.25	21.49	1.99
	4	4.50	13.43	6.31	1.57	15.51	2.63	51.95	4.09
	5	5.96	9.52	2.67	8.11	19.77	12.65	37.54	3.78
	6	7.88	5.19	1.35	19.95	26.00	19.58	18.31	1.73
	7	3.17	9.64	11.73	2.22	9.93	60.62	2.69	–
	8	11.06	42.05	20.76	1.06	5.30	18.78	0.99	–

Table 3. The size of inclusions treated for 5 minutes.

Power/W	Size of inclusions/μm
0	5.36
150	4.67
200	4.28
300	3.54

Table 4. The size of inclusions treated under power of 300 W.

Treating time/min	Size of inclusions/μm
0	5.36
1	4.68
3	4.11
5	3.54



(a) (b) (c)

Fig. 6. The inclusions in stainless steel.

mean size of inclusions becomes smaller as the ultrasound power rises, also, if the treating time is prolonged, the mean size of the inclusions decreases. In order to break the inclusions into smaller size of pieces, refine the microstructure, and consequently improve the mechanical properties of stainless steel materials, it is necessary to increase the power and prolong the treating time.

2.4. Effect on Mechanical Property

The test samples obtained from positions 1 and 2 were machined to the size of 3 mm×3 mm×6 mm to analyze the effect of ultrasonic treatment on the mechanical property of stainless steel. The microcomputer control electron universal testing machine WDW3050 is applied for compressive test. For the test samples from positions 1 and 2 under different treating power of 150 W, 200 W and 300 W, the stress-strain curves are plotted in **Figs. 7** and **8**, respectively. It can be seen from Figs. 7 and 8 that the stress is significantly increased with ultrasonic treatment. For the position 1, the increase of strength is significant as the ultrasound power increases from 150 W to 300 W. However, at position 2, the intensity of ultrasound is not so high as that at position 1 due to the attenuation of ultrasound, as a result, the effect of ultrasonic treatment becomes weak.

It can be seen from the figures above that there is no yield platform for 304 stainless steel, so the strain corresponding to 0.2% residual plastic deformation is taken as yield strength. The yield strength is determined and listed in **Table 5**.

The yield strength of stainless steel without ultrasonic treatment is only 224 MPa, however, the yield strength of the sample from position 1 under the power of 300 W is greatly improved to 345 MPa. Meanwhile, the slope of elastic deformation line increases evidently with ultrasonic treatment, which means the elastic modulus is enhanced, indicating that ultrasonic treatment with high power improves the mechanical property of stainless steel apparently. The yield strength increases with the rise of power, and the effect of treatment at position 2 is weaker than that at position 1 as the attenuation of ultrasound occurs.

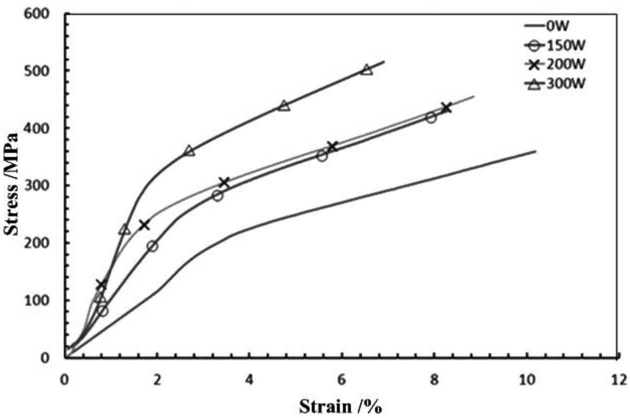


Fig. 7. Curves of stress-strain of the samples from position 1.

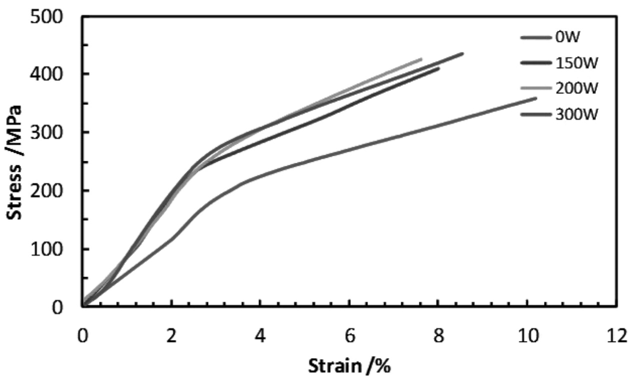


Fig. 8. Curves of stress-strain of the samples from position 2.

3. Analysis and Discussion

After the treatment of molten metal using ultrasound, the solidification microstructure changes from dendrite to equi-axed structure, whose grain is refined as the ultrasonic power rises. It is generally believed that, after the introduction of ultrasound into molten metal, the pressure inside the melt changes periodically between high and low pressures due to the ultrasonic vibration, which is one reason for the refinement of crystal grain. Negative pressure appears due to the local tensile stress, and when the pressure reaches the saturation point of gas dissolved in the liquid melt, bubbles will form, move, grow and escape from the molten metal. As the pressure changes into compressive and reaches a peak, these bubbles will quickly contract or collapse in a sudden. When the collapse occurs, high temperature, high pressure and shock wave appear consequently in the liquid metal, leading to ultrasonic cavitation, which can break up the dendrites and causes the formation of equi-axed grains, too. In order to illuminate the effect of ultrasonic pressure and cavitation pressure on the solidification microstructure, it is necessary to discuss the magnitude of ultrasonic pressure and the requirement of cavitation.

3.1. Ultrasonic Pressure

When the ultrasound is introduced into the molten metal, the sonic pressure P_k[23] is expressed by the following form:

$$P_k = \sqrt{\frac{2P\rho_s C_s}{s}} \quad \text{.............................} (4)$$

where, P is the power of ultrasonic vibration, ρ_s is the density of liquid metal with a value of 6 900 kg/m^3, C_s is the travelling velocity of ultrasound in molten metal, set as 3 250 m/s, S stands for the cross section area of the end of the booster.

It is figured out through calculation that when the ultra-

Table 5. The yield strength of 304 stainless steel under different treating conditions.

Treating condition	Yield strength/MPa
0 W	224
150 W position 1	260
150 W position 2	251
200 W position 1	268
200 W position 2	260
300 W position 1	345
300 W position 2	289

sonic power is 150 W, 200 W and 300 W, the sonic pressure P_k is 3.1 MPa, 3.6 MPa and 4.4 MPa, respectively.

3.2. Ultrasonic Cavitation

The cavitation threshold, the lowest sonic pressure required for cavitation, has the expression as below:[24)]

$$P_B = P_0 - P_v + \frac{2}{3\sqrt{3}}\left[\frac{\left(\frac{2\sigma}{R_0}\right)^3}{P_0 - P_v + \frac{2\sigma}{R_0}}\right]^{\frac{1}{2}} \quad \text{............ (5)}$$

where, P_B is the threshold pressure of cavitation, P_0 is the static pressure in liquid metal, in this article, P_0 = 101 325 Pa, P_v is the vapor pressure of the bubble, σ is the surface tension of the molten steel, set as 1.665 N/m, R_0 stands for the initial radius of cavitation core, the value is given as R_0 = 1 – 10 μm,[25)] the assumption is that the gas in the bubble is considered as ideal gas, the bubble is spherical and is moving in radial direction.

Calculation reveals that the threshold sonic pressure of cavitation ranges from 0.44 MPa to 3.89 MPa. It is apparent that the sonic pressure is higher than cavitation threshold, implying that cavitation does occur in the liquid metal with the introducing of ultrasound.

Noltingk–Neppiras model[26)] describes very well the pulsation of cavities in an acoustic field:

$$R\frac{d^2R}{dt^2} + \frac{3}{2}\left(\frac{dR}{dt}\right)^2 + \frac{1}{\rho_L}\left[P_0 - P_v - P_{UT} + \frac{2\sigma_L}{R} - \left(P_0 + \frac{2\sigma_L}{R_0}\right)\left(\frac{R_0}{R}\right)^{3\gamma}\right] = 0 \quad \text{.... (6)}$$

however, it does not determine accurately the collapse velocity and related peak pressure in the cavities, as it assumes an incompressible liquid. The use of Herring–Flinn model and Kirkwood–Bethe–Gilmore model[26)] can improve the Noltingk–Neppiras model, as it allows for liquid compressibility, viscosity effects, cavity motion, and free enthalpy change at the surface of a spherical cavity.

The maximum instantaneous pressure in the molten melt, P_{max}, is expressed as follows:[24)]

$$P_{max} \approx 4^{-\frac{4}{3}}\left(\frac{R_m}{R}\right)^3 P_0 \quad \text{......................... (7)}$$

where, R_m is the maximum radius of cavitation bubbles, R is the minimum radius of the shutting bubbles, Eq. (7) is suitable for calculating the maximum cavitation pressure in liquid phase including both low melting alloys and high melting alloys. It is figured out from calculation that P_{max} ranges from 3 924 MPa to 3.9 MPa as R varies in a range from 1 μm to 10 μm.

3.3. Mechanism of Refinement of Solidification Microstructure and Fragmentation of Inclusions

The fragmentation of the dendrites occurs due to two reasons, cavitation pressure and ultrasonic pressure, as illustrated in **Fig. 9**. The effect of explosion shock caused by instantaneous high pressure released by the collapsing bubbles leads to dendrite fragmentation, is show in Fig. 9(a); on the other hand, in Fig. 9(b), the uniform pressure imposing on the dendrite is the ultrasonic pressure, during the solidification, the torque at the root of the dendrites increases as the dendrites grow, leading to the fragmentation of dendrites. Under the effect of cavitation pressure, the dendrites break up completely and transform into fine equi-axed grains, as shown in Figs. 5(b-1), 5(b-2), 5(c-1), 5(c-2), 5(d-1), 5(d-2). But due to the attenuation of ultrasound, only some dendrites with low-strength which forms at the initial stage of solidification break up, resulting in the formation of short dendrites, as can be seen in Fig. 5(b-3), the fragmentation of the dendrites is mainly caused by the vibration of liquid metal due to ultrasonic pressure. In Figs. 5(c-3), 5(d-3), the refinement of microstructure is still obvious in spite of attenuation, but the effect of ultrasonic treatment at position 3 is not so strong as that at position 1 and 2, coarse equi-axed structure forms at position 3.

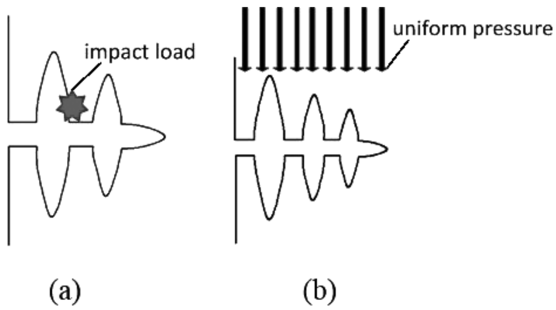


Fig. 9. Dendrite fragmentation mechanism.

As has been mentioned above, the ingredients of inclusions in the molten metal are mainly silicate and manganese oxide, whose yield strength is more than 30 MPa, apparently, only the strong pressure released by cavitation bubbles can break up the inclusions. And from the size of inclusions, it can be concluded that the inclusions are gradually desquamated into smaller sizes. Resulting from the refinement of solidification microstructure and the fragmentation of inclusions, the mechanism property of 304 stainless steel is greatly improved.

4. Conclusions

(1) A new method called unit piece method (UPM) to introduce ultrasound into high temperature molten metal is proposed. This method can avoid the erosion of booster in high temperature molten metal. Meanwhile, the determination of the length of the booster to meet the resonance condition has been presented.

(2) By the proposed method, the effect of ultrasound on the microstructure of 304 stainless steel is studied. Ultrasonic treatment transforms the microstructure from dendrites to equi-axed grains and refines the dendrites as well. The effect of refinement is more and more obvious with the power of ultrasound increasing from 150 W to 300 W. The effect of refining improves with the increase of power and treat time. And the size of inclusions are reduced with the ultrasonic treatment. The mechanical property of 304 stainless steel is greatly improved with the treatment of ultrasound, and the effect of the enhancement of mechanical property increases

with the rise of power.

(3) The fragmentation of the dendrites is caused by two reasons, cavitation pressure and ultrasonic pressure. Under the effect of cavitation pressure, the dendrites can break up completely and transform into equi-axed grain, but only some dendrites with low-strength which forms at the initial stage of solidification break up and transform into short dendrites. The inclusions are desquamated gradually into smaller size by the pressure released from the cavitation bubbles. Due to the refinement of solidification microstructure and fragmentation of inclusions, the mechanism property of 304 stainless steel is greatly improved.

Acknowledgement

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