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Publisher's version / Version de l'éditeur:
https://doi.org/10.1007/s11837-004-0148-9
JOM Journal of the Minerals, Metals and Materials Society, 56, 2, pp. 59-64, 2004-02-01

An On-Line Ultrasonic Cleanliness Analyzer for Molten Light Metals

Yuu Ono, Jean-François Moisan, Yuanbei Zhang, Cheng-Kuei Jen, and Chun-Yi Su

Ultrasonic techniques to evaluate the cleanliness of molten aluminum and magnesium using clad steel buffer rods are presented in this article. Backscattered ultrasonic signals from silicon carbide particles, added to molten aluminum, were detected with ultrasonic pulse-echo and pitch-catch modes at an ultrasonic frequency of 10 MHz. The scattered signals from inclusions, which might be oxide films and/or particles, in molten magnesium, were also detected. To establish the procedure and to investigate the optimum system configurations for evaluating melt cleanliness, a particle flow simulator was constructed that uses water and polyvinyl chloride particles. Also developed was a prototype of an on-line ultrasonic cleanliness analyzer for molten magnesium.

INTRODUCTION

In the processing of lightweight metals such as aluminum and magnesium, metal cleanliness is crucial for refining, part manufacturing, and recycling. The quality of products is frequently and critically associated with the presence of inclusions of intermetallic and/or non-metallic materials within the molten metals during manufacturing steps. The presence of these inclusions affects the mechanical properties of metals, such as strength and corrosion resistance, and results in deterioration of product quality. The inclusions can be oxide films and particles, other hard particles derived from the original smelting process, or other reaction products such as flux particles. The size of these inclusions can vary from less than 1 μm to greater than 100 m.

Several techniques are available for evaluating metal quality based on the extraction of a metal sample followed by analysis in a laboratory. Ideally, the cleanliness of molten metals would be evaluated for large quantities that could be rapidly analyzed with little or no sample preparation. This can only be achieved by analyzing the metal while it is still in the molten state. For aluminum processing, a commercial device, the Liquid Metal Cleanliness Analyzer (LiMCA, ABB Bomen, Quebec, Canada) is available.[1] The LiMCA method is based on the electric sensing zone principle for counting and sizing non-conductive particles passing through the orifice used in the system. However, the LiMCA is not yet applicable to molten magnesium due to the reactive nature of molten magnesium to the orifice materials. In addition, the LiMCA is not capable of effectively detecting conductive inclusions such as intermetallic particles, which are often present in molten aluminum and magnesium.

Ultrasonic techniques have been reported as on-line methods to monitor molten metal properties.[2–4] Ultrasound can propagate in molten metals without much attenuation and, when inclusions are present in the molten metals, the ultrasonic signatures such as velocity and attenuation of the liquid and the ultrasonic energy scattered by the inclusions will change. Since the variation of the velocity and attenuation is not sensitive to small amounts of inclusions (e.g., less than 30 ppm, which is of interest for this study), the detection of the scattered energy from each individual particle is required.[5] Ultrasonic probes used in the earlier studies were composed of an ultrasonic transducer and buffer rod having no cladding.[2–4] Due to the lack of cladding, signal-to-noise ratio (SNR) of the ultrasonic signal in such non-clad buffer rods was poor. The SNR is defined as the strength of the desired signal from the inclusions divided by the strength of the spurious signals (noises) produced in the rod. The inclusion detection sensitivity is directly related to the SNR. The higher the SNR, the better the detection sensitivity. The authors' approach is to install ultrasonic probes using high-SNR clad buffer rods[6,7] at the opening of a tube through

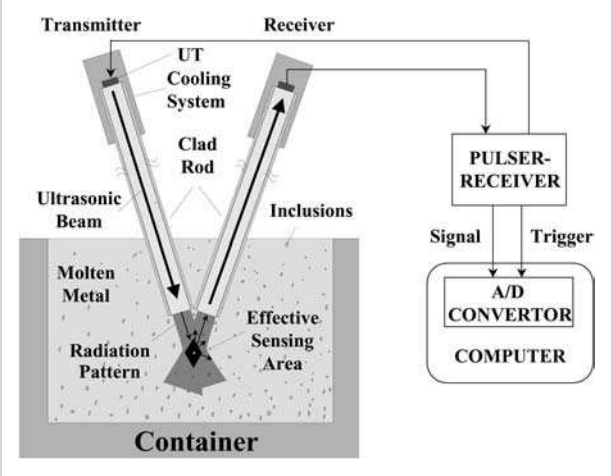

Figure 1. A schematic view of the configuration of inclusion detection in molten metals with the pitch-catch mode.

Figure 2. The double-taper-shaped clad steel buffer rod used for ultrasonic probes.

which the molten metals are guided. The inclusions that exit from the tube will scatter the ultrasonic energy and can be detected by the clad buffer rod probes. The amplitude of this scattered ultrasonic energy may be used to measure the inclusion size. Each detection may render a count for the inclusion.

INCLUSION DETECTION IN MOLTEN ALUMINUM AND MAGNESIUM

Figure 1 shows a schematic view of a configuration for inclusion detection in molten metals. In pitch-catch mode, one probe (the transmitter) transmits the pulsed ultrasound into the molten metals and another one (the receiver) receives the ultrasonic signals scattered by the inclusions with a pulser-receiver. On the other hand, in pulse-echo mode, only one probe is used to serve as both the transmitter and the receiver of ultrasound. Figure 2 shows a 280-mm-long, double-taper-shaped clad steel buffer rod used in the experiments. Rods with different lengths are available, up to 1 m. The selection of rod length depends on the depth of the container and the amount of the molten metals examined. The rod consists of a mild steel core and a stainless-steel (SS) cladding. An ultrasonic transducer (UT) is attached on one end of the rod; the other is the probing end immersed in the molten metals. For focused ultrasonic waves, ultrasonic-focused probes are available with a spherical concave lens at the probing end.[5] The focused probes can detect smaller inclusions than the flat-end probes for plane waves. Aluminum or magnesium contained in the SS container was heated and melted by an electric resistance furnace. To prevent oxidation, argon gas for molten aluminum or 1% sulphur hexafluoride (SF_6) gas for molten magnesium[8] was supplied above the surface of the melts. The oxides are considered major inclusions, especially in magnesium.[9,10] The dimensions of the container were 90 mm in diameter and 100 mm in depth, and the depth of melts was about 80 mm. The sensing area was at the center of the container, which was about 30 mm below the surface of the melts.

The inclusion-detection experiment with molten aluminum was conducted first. The silicon carbide (SiC) particles, with an average size of 50 m and a size range of 30 m to 60 m, were suspended in the molten aluminum (aluminum alloy A356) as inclusions after the aluminum completely melted. The molten aluminum was well stirred manually to distribute the inclusions uniformly before acquiring the data. The results obtained using the buffer rods having flat probing ends with the pitch-catch mode are shown in Figure 3a. The temperature of the molten aluminum was 780°C. The signals were recorded at 2 ms per frame with a time window of 20 s covering the entire detection area where the desired signals were reflected from the inclusions. The figure shows only the frames from every 40 ms with the time window of 5 s. The backscattered ultrasonic signals from the inclusions were observed when the inclusions passed through the sensing area, as shown in Figure 1. Movements of inclusions were visible. The scattered signals were also observed with the pulse-echo mode using a single probe with and without an ultrasonic lens at the probing end (the data are not shown here). However, the pitch-catch mode had a higher SNR than the pulse-echo mode. It should be noted that molten aluminum corrodes steel and stainless steel, which are the materials of the buffer rods used here. Although the steel rod can sustain corrosion in the molten aluminum for a short period of time, the authors are developing probe materials and/or coating materials for the probe that have good ultrasonic wetting and sufficient corrosion resistance to transmit and receive the stable ultrasonic signals. Ultrasound cannot be transmitted from the probe into materials without the ultrasonic wetting between the probe and liquid.

The same experiment was conducted with molten magnesium (purity: 99.8%). The temperature of the magnesium was 680°C. Figure 3b shows typical signals obtained using the buffer rods having flat probing ends with the pitch-catch mode. The backscattered ultrasonic signals from inclusions were observed as they were in the molten aluminum experiment. However, it should be noted that the scattered signals were observed

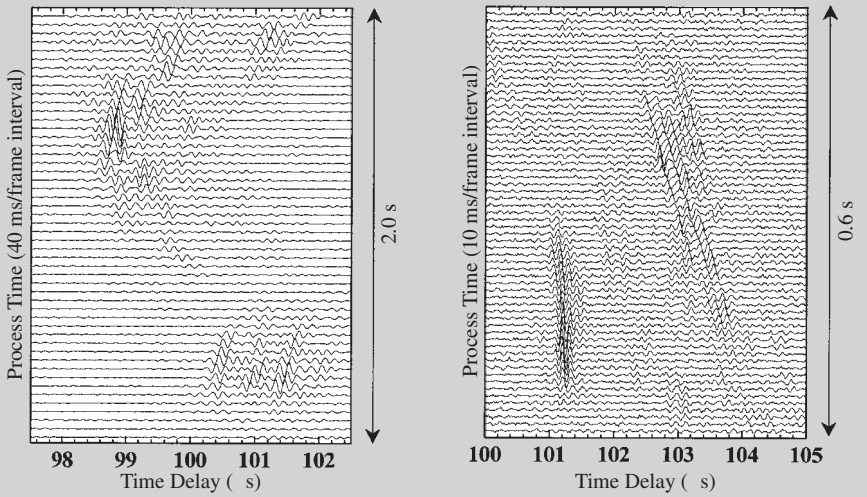

Figure 3. The typical detected backscattered signals from inclusions (a) in molten aluminum at 780°C and (b) in molten magnesium at 680°C with the pitch-catch mode.

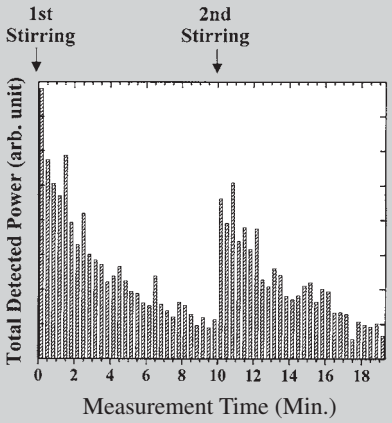

Figure 4. The variation of total power of the detected signals in molten magnesium with respect to the measurement time after stirring the molten magnesium.

without adding any inclusions to the molten magnesium. The inclusions detected might be oxides, intermetallic particles, and/or protective films in the molten magnesium.[8–10] Experimental and signal-acquiring conditions were the same as those for the molten aluminum except that about 20 dB more electric amplification of the receiver were required for the detected signals to have the same signal amplitude as the molten aluminum. Furthermore, the scattered signals could not be observed with the pulse-echo mode. These differences are mainly due to the fact that wettability and ultrasonic coupling at the interface between the probing end and molten metal were better for the molten aluminum than for the molten magnesium. Better wettability can transmit more ultrasonic energy into the liquid and result in the detection of stronger ultrasonic signals scattered from the inclusions. After a few hours immersion of the probing end of the rod in the molten magnesium at around 700°C, no significant corrosion was observed on the probing ends. Therefore, the clad steel buffer rod may be a promising candidate for the molten magnesium process monitoring. It is known that no intermetallic phase can form between solid iron and pure molten magnesium and that the mutual solubility between them is low.[11] In addition, it should be noted that, in previous work,[7] the authors did not notice any physical degradation of the clad steel rod during the 330 h operation in the molten magnesium in an industrial environment.

RELATIVE CLEANLINESS MEASUREMENT

Since the movements of the inclusions were random in the melts under the experimental condition shown in Figure 1, it is difficult to determine the size and number of the inclusions in the melts using the signals shown in Figure 3a and b. Here, the relative cleanliness evaluation of molten aluminum and magnesium is investigated with the measurement configuration shown in Figure 1. For the molten magnesium, after stirring, signals were acquired for a period of 5 s with an interval of 20 s. The temperature range of the melt was between 690°C and 700°C throughout the experiments. A bar graph shown in Figure 4 presents the variation of total power of the detected signals with respect to the measurement time. The total power is the summation of the power of detected signals. Just after the first stirring, the detected power was the greatest at 0 min., gradually decreasing with respect to measurement time. This is because the inclusions that were heavier or lighter than the molten magnesium settled to the bottom or top of the melt, respectively, and less and smaller inclusions passed through the sensing area at the center of the melt. After 10 min., a second stirring was performed and the detected power increased again. This result indicates that the inclusion distribution (i.e., cleanliness) in the melt pool could be evaluated by moving the probes in the melt; dirty melts scatter back more ultrasound than clean melts. A similar result was obtained when the same experiment was performed with molten aluminum (the data are not shown).

PARTICLE FLOW SIMULATOR FOR COUNTING AND SIZING INCLUSIONS

A particle flow simulator using water and polyvinyl chloride (PVC) particles has been developed to establish the procedure and to investigate the optimum system configurations for counting and sizing inclusions in liquid. This water simulator plays an important role in the development of an ultrasonic method and system for evaluating molten metal cleanliness. The water simulator has several advantages. First, the experimental situations are observed visually, hence, one knows the interface conditions between the probing end and the water. Such a visual observation can provide reliability, reproducibility, and confidence in the experiments. In addition, PVC particles can be well dispersed in water, meaning they are not easily clustered, and any scattered ultrasonic signals may very well correspond to the single PVC particle.

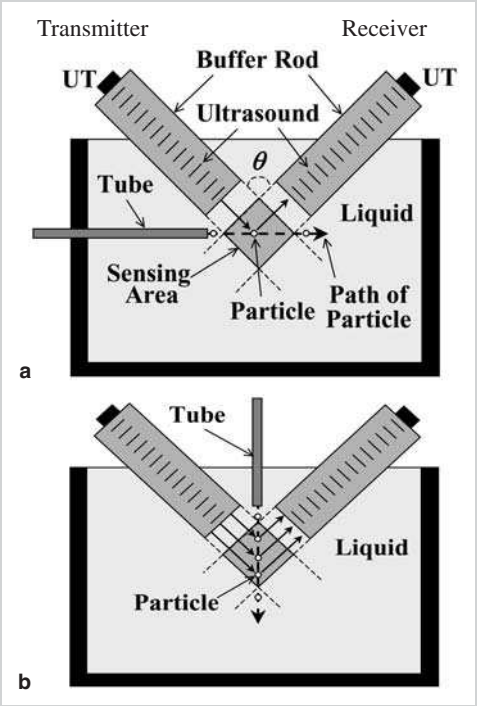

Figure 5. The method of particle detection in liquids using the pitch-catch mode. The particles are injected (a) horizontally or (b) vertically through the tube.

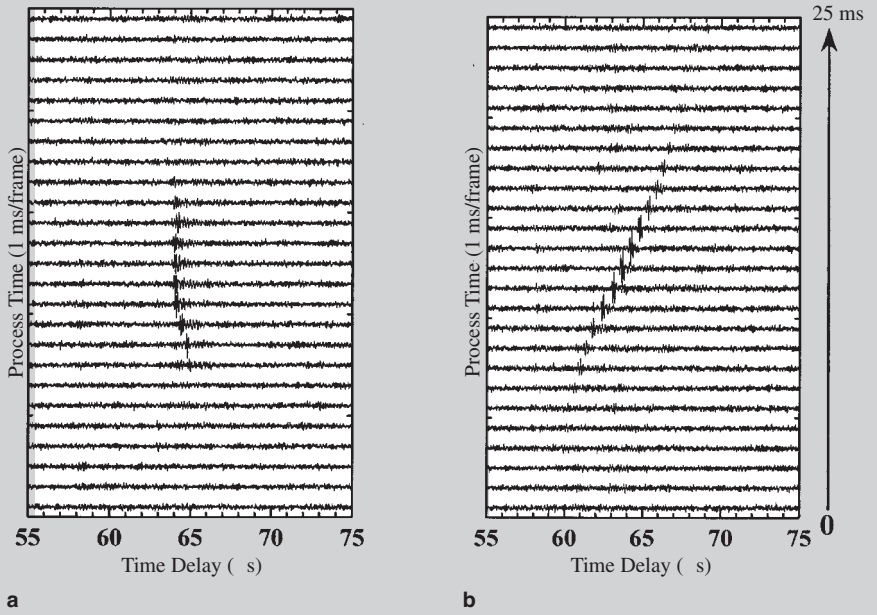

Figure 6. Typical signals scattered from a PVC particle obtained with (a) horizontal and (b) vertical injection configurations shown in Figures 5a and b, respectively.

Another advantage is that the scattered signal strength from the PVC particle in water is close to that from aluminum oxide in molten aluminum and to that from magnesium oxide in molten magnesium. Finally, it can reduce the cost for design and optimization of the system for molten metals, and it is safe. The experiments can be carried out at room temperature.

Figure 5 shows a method for counting and sizing the particles in liquids with the pitch-catch mode. The detail of the system was presented elsewhere.[12] An ultrasonic focused probe, having a spherical concave lens at the probing end, also can be used as transmitter and/or receiver, which is not shown in the figure. Furthermore, the configuration of the pulse-echo mode is also available. The strength of ultrasound has spatial distribution and depends on the position in the sensing area. Hence, the particle movement needs to be controlled using a tube and a pump to pass in the middle of the sensing area where the largest scattering signals are obtained. The amplitude of the scattered ultrasonic signal can be used to measure the particle size; each detection provides a particle count.

Two configurations are shown in Figures 5a and b, in which the particles are injected into the liquid horizontally and vertically, respectively. It is assumed that the path of the particle movements is straight and passes in the middle of the sensing area, as shown in Figure 5. In the case of (a), since the time delays of the signals reflected from the particles anywhere on the path are nearly the same, only one particle must be present in the sensing area in order to obtain the signals reflected from this single particle. On the other hand, in the case of (b), the time delays of the signals reflected from the particles at different positions on the path are not the same, hence, the signals could be obtained separately. Therefore, multiple particles could be present and detected in the sensing area at the same time. This means that the configuration of (b) is preferable to that of (a). The particle detection experiments were conducted using the water simulator. The PVC particles, with a density (1.4g/cm^3) approximating that of water, were added as inclusions and well dispersed without being clustered in water. An average diameter, D, of the PVC particles was 128 m. This size was arbitrarily chosen. Water with the PVC volume concentrations, C, of 5 ppm or 10 ppm, was prepared. A flow rate, Q, of the water, which flowed to the sensing area through the tube by a water pump, was 2.16 mL/s. The flow rate that is related to the inspection volume could be increased further. An SS tube with an inner diameter of 1.6 mm was used since the sizes of the inclusions in molten aluminum or magnesium are, in general, less than 1 mm.

Figures 6a and b show the typical signals obtained with the horizontal and vertical injection configurations, respectively. In the case of horizontal injection, shown in Figure 6a, a series of signals scattered from the PVC particle appeared nearly at a constant time delay

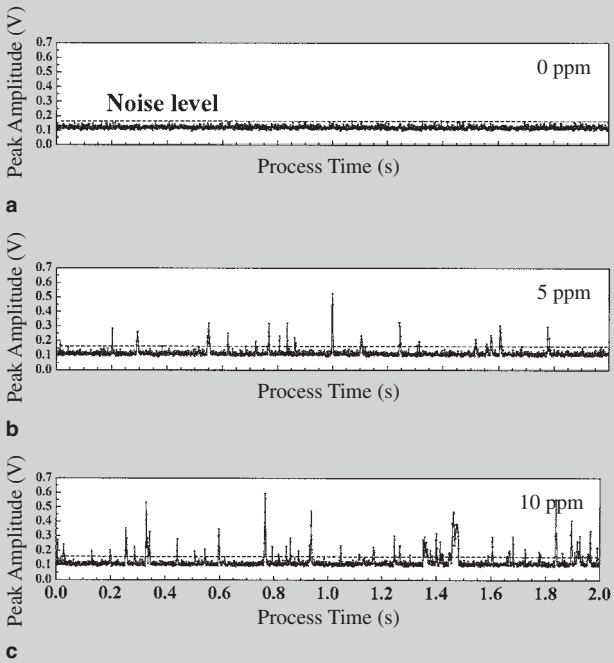

Figure 7. The peak amplitude obtained from the backscattered signals from inclusions with the horizontal injection configuration shown in Figure 5a. The volume concentrations of the PVC particles in water were (a) 0 ppm, (b) 5 ppm, and (c) 10 ppm. Dashed lines indicate the noise level.

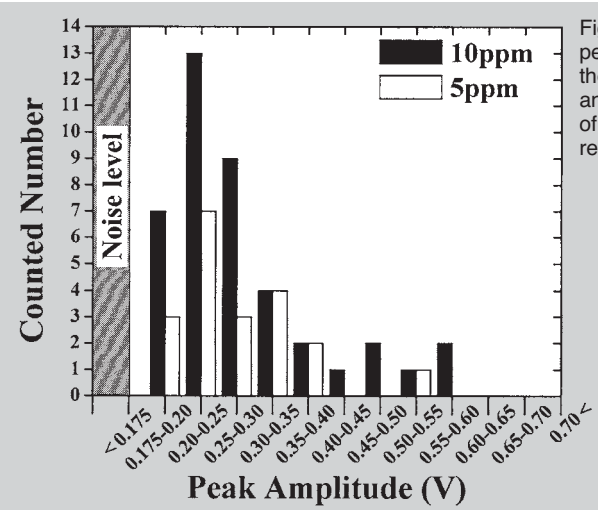

Figure 8. The distributions of peak amplitude obtained from the data shown in Figures 7b and c for the PVC concentrations of 5 ppm and 10 ppm in water, respectively.

of 64 s. This suggests that the particle moved straightly, as shown in Figure 5a. The signals from the particle were observed for a process time of about 10 ms, which is associated with the moving speed of particles and the effective sensing area. In the case of vertical injection, shown in Figure 6b, the time delay of a series of signals scattered from the PVC particle increased linearly as process time. The largest signal was obtained at a time delay of 64 s. This also means that the particle moved straightly, as shown in Figure 5b, with a constant speed. Therefore, it was verified that the particle movement in the sensing area was controlled by the water pump and tube used in the system.

Using the results obtained by the water simulator, the PVC particles in water were counted. The maximum value of amplitude of the signal for each frame was plotted with respect to the process time. Figures 7a, b, and c show the results for the PVC concentrations of 0 ppm, 5 ppm, and 10 ppm with the horizontal injection configuration for the process time, T, of 2 s. At 0 ppm, water without PVC particles flowed and signals were recorded. This result provides the noise level of 0.175 V, indicated by the dashed lines in Figure 7, under the presented measurement conditions. One can see pulselike shapes in the curves shown in Figures 7b and c. Each pulse is composed of the signals from the single PVC particle. The number of the pulses having the peak value more than the noise level (0.175 V) was counted. The counted numbers were 20 for 5 ppm and 41 for 10 ppm, while the calculated numbers were 20 for 5 ppm and 39 for 10 ppm using the values of the parameters, C, Q, T, and D, in the experiments. Therefore, the experimental results had good agreement with the calculated results. After many measurements, the errors of the counts obtained by the presented method were always within 10% or two counts of the calculated results both in the horizontal and vertical injection configurations.

Next, the size distribution of the PVC particles in water was investigated. It is reasonable to consider that the stronger ultrasonic signal is scattered from the larger particle. Therefore, the peak amplitude values of the pulses shown in Figures 7b and c are associated with the size of particle detected. The numbers of peak values in each range of 0.05 V were counted using the data shown in Figures 7b and c. The results are presented in Figure 8. The peak amplitude distribution corresponds to the size distribution of the particles. It should be noted that the smallest amplitude region of 0.175–0.2 V in Figure 8 is the half range of 0.05 V due to the noise level of 0.175 V. Therefore, the small particles, which gave the peak amplitude values of less than 0.175 V, might be missed in the counting. Detection sensitivity needs to be improved further to detect such small particles by increasing the SNR of the system. For example, PVC particles of 30 m have been successfully detected using a short and/or focused clad buffer rod probe (the data are not shown). It is concluded that the particle size and its distribution could be determined when the relationship between the amplitude and the particle size is obtained using the PVC particles having different diameters with known sizes, which are commercially available. Further study will be conducted to determine the sizes of particles using the water simulator in the future.

ULTRASONIC CLEANLINESS ANALYZER FOR MOLTEN MAGNESIUM

Based on the water simulator study, a prototype of an on-line ultrasonic cleanliness analyzer for molten magnesium has been constructed. Figure 9 shows a schematic view of the system and experimental setup for inclusion detection in the molten magnesium with the pitch-catch mode. Two clad steel buffer rods (shown in Figure 2) were used

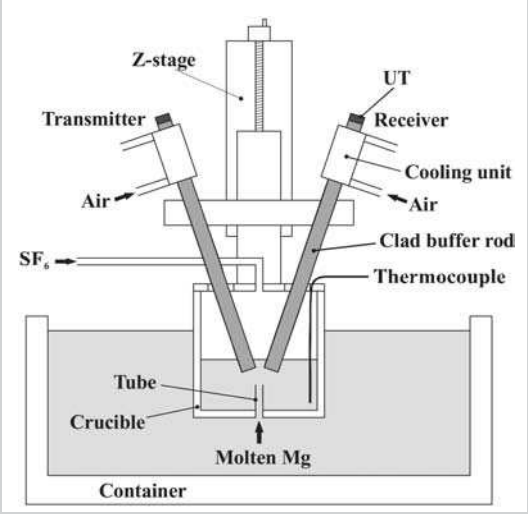

Figure 9. A schematic view of the experimental setup for counting and sizing inclusions in molten magnesium using the tube with the pitch-catch mode.

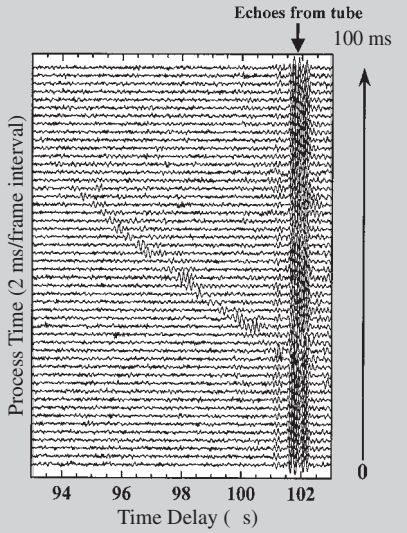

Figure 10. Measured ultrasonic signals scattered from inclusion injected through the tube into ultrasonic sensing area in molten magnesium at 778°C.

for ultrasonic probes as transmitter and receiver. These ultrasonic probes were attached on an SS crucible. An SS tube with an inner diameter of 4.8 mm was fixed at the bottom of the crucible to realize the vertical injection configuration shown in Figure 5b. It should be noted that melt flowed vertically upward in the sensing area in this system, which was opposite of the water simulation experiment shown in Figure 5b. The size of the ultrasonic sensing area formed by the two clad buffer rods was large enough to cover the area where the molten magnesium passed through the tube from the container. Due to the lack of a molten metal pump at present, the crucible with ultrasonic probes was sunk slowly into the molten magnesium in the container using a vertical-translation stage (Z-stage) on which the crucible was mounted, so that the molten magnesium in the container flowed into the crucible gradually through the tube. Once the crucible was filled completely with the molten magnesium, the crucible was lifted up so that the melt in the crucible flowed out to the container. Hence, the measurement was repeatable. The melt flow through the tube would be controlled more precisely and the measurements could be more efficient when the system is equipped with a molten metal pump in the future.

Magnesium (magnesium alloy: AZ91D) in the container, for which cleanliness would be evaluated, was heated and melted by an electric resistance furnace. Figure 10 presents typical signals obtained when the molten magnesium in the container flowed into the crucible through the tube. The melt temperature was 778°C. Echoes reflected from the top tip of the tube were always observed at a time delay of 102 s, since the tip was located inside the sensing area (but not at the center of the sensing area). The amplitude of the echoes from the tube could be used as a reference signal to calibrate the amplitude of the signals from inclusions, since the signal amplitude would change due to the experimental conditions such as melt temperature. A series of scattered signals from the inclusion were observed in a time delay range of 94.5 s to 100.5 s for a process time of 34 ms. One can see that the time delay of the scattered signal from the inclusion decreased linearly as process time and the largest signal was obtained at a time delay of 98 s. This means that the inclusion moved nearly in a straight line with a constant speed. Therefore, the movement of the inclusion in the ultrasonic sensing area was controlled by the tube used in the system. The size of the inclusions could be determined using the amplitude when the relationship between the amplitude and size of inclusion is obtained using several standard particles whose sizes are known. Magnesium oxide might be one of the candidates as a standard particle. Further study for evaluating the characteristics of the inclusion such as type, size, and shape will be conducted in the future.

ACKNOWLEDGEMENT

The authors are very grateful to H. Hébert for his assistance in data acquisition. This work is supported by the National Research Council-NSC (Taiwan) Research Funding, the Canadian Lightweight Materials Research Initiative, and the Natural Sciences and Engineering Research Council of Canada.

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