

Sensors for Monitoring the Quality of Molten Aluminum During Casting

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The quality of a cast product depends directly on the quality of the molten metal from which the product is cast. Techniques for determining melt quality often require the laboratory analysis of samples taken from the molten metal. In addition to reducing processing efficiency, delays associated with this laboratory analysis can lead to uncertainties because the melt conditions can change during holding at high temperatures. On-line monitoring of the melt quality provides more rapid feedback for improved process control. In this article, techniques for monitoring the important aspects of melt quality, specifically chemical composition, gas content, and inclusion content, are reviewed.

Keywords chemical sensors, gas content, hydrogen, inclusions, molten aluminum

1. Introduction

Increasing demands for improved quality control and process efficiency of industrial processes require the development of on-line sensors to provide feedback for process optimization. In the case of cast products, the cleanliness of the molten metal must be maintained to control the properties of, and to prevent defect formation in, the resulting castings. The three primary aspects of melt quality are chemical composition, gas content, and inclusion content (Ref 1-3).

Control of alloy properties requires the control of the alloy composition. For example, Na or Sr is used to control the eutectic microstructure and, thus, the properties of castings (Ref 4-7). These and other alloying additions can affect the castability (Ref 8) and mechanical properties (Ref 9) of cast products. Thus, loss of alloying additions due to preferential vaporization, or oxidation, while holding the melt at high temperatures can lead to degradation of the mechanical properties. In addition, Na and other alkali elements can lead to embrittlement or hot-cracking (Ref 10), and unwanted levels of these elements can be introduced during the Hall-Heroult process (Ref 11). However, like Na, Li is sometimes deliberately added as an alloying element, particularly for producing low-density alloys (Ref 12). Thus, controlling the amount of these elements in the desired range is critical to controlling the properties of aluminum castings. In addition to controlling the alloy composition prior to casting, monitoring the chemical composition is important in other processes, such as the removal of Mg during the reclamation of aluminum scrap (Ref 13).

The most important gas in processing aluminum is hydrogen, which is introduced when molten aluminum reacts with moisture in the surrounding atmosphere to form aluminum oxide and hydrogen. The solubility of hydrogen in solid alumi-

num is much lower than that in liquid aluminum, so dissolved hydrogen is evolved during solidification and can lead to porosity in the resulting casting (Ref 14, 15). Thus, the production of quality castings requires the removal of hydrogen from the molten aluminum by degassing processes (Ref 15-22). Efficient and effective control of these processes requires on-line measurement of the hydrogen content in the melt.

Inclusions can be incorporated in molten aluminum due to oxidation, erosion of refractories, or fluxing, and can lead to a variety of detrimental effects, such as degradation of the mechanical properties, appearance, and corrosion resistance (Ref 2, 22-24). Thus, the production of quality castings requires the development of processes for the removal of inclusions (Ref 2), and the control of these removal processes requires on-line monitoring of the sizes and amounts of inclusions in the molten metal.

There is clearly a need for monitoring the quality of molten aluminum, and the objective of this article is to review techniques for on-line monitoring of the chemical composition, gas content, and inclusion levels in molten aluminum.

2. Alloy Composition

The Office of Industrial Technologies of the U.S. Department of Energy has worked with the aluminum industry to identify key research and development needs for the industry (Ref 25, 26). The most recent Aluminum Industry Technology Roadmap, published in February 2003 (Ref 25), includes real-time chemical analysis among the seven issues identified as top priorities in the areas of melting, solidification, and recycling.

2.1 Laser-Induced Breakdown Spectroscopy

One promising technology for monitoring the chemical composition of alloys is laser-induced breakdown spectroscopy (LIBS) (Ref 26, 27). Laser-induced breakdown spectroscopy has been used for determining the composition of metal (Ref 27-32) and ceramic materials (Ref 33-35), including applications such as separating metal scrap (Ref 32) and analyzing historical objects (e.g., archeological artifacts [Ref 33] and daggerreotypes [Ref 36]). Laser-induced breakdown spectros-

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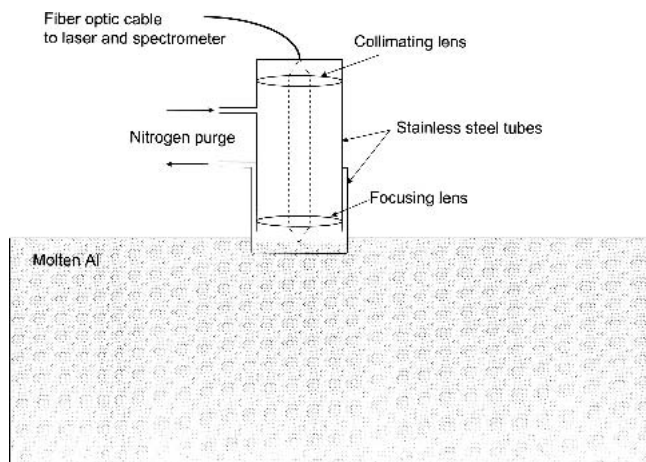


Fig. 1 Schematic of LIBS for the chemical analysis of molten metals (Ref 50)

copy has been used to determine the amounts of common alloying elements, such as Mg, Cu, and Si, in aluminum (Ref 37-41). Although most commonly used at low temperature, LIBS has been adapted for high-temperature applications, such as monitoring radioactive glass melts (Ref 35) and welding processes (Ref 42-45). These developments have led to the use of LIBS for measuring the composition of molten metals (Ref 46-52), including molten aluminum (Ref 26, 27, 49-52).

A schematic of a LIBS sensor for measuring the composition of a molten metal is shown in Fig. 1. The probe is connected to the analytical system with a fiberoptic cable that is used to transmit a pulsed laser, which is focused on the metal surface to create a plasma for chemical analysis. The surface of the molten metal must be situated in the focal plane of the laser, so the position of the molten metal surface is adjusted with the internal gas pressure by controlling the flow rate of the nitrogen purge gas. The spectral distribution of the radiation in the plasma generated by the laser is analyzed through the fiberoptic cable using a beam splitter.

Some of the issues in the use of LIBS include ensuring that the probe is sampling metal below the slag layer and that emission from the slag or slag particles is not affecting the measurement. In addition, the signal for a particular element is affected by the volatility of that element in response to excitation by the laser pulse, so these differences must be accounted for in the analysis.

2.2 Galvanic Cell-Based Chemical Sensors

Another approach to monitoring the chemical composition of molten aluminum is through the use of chemical sensors based on galvanic cells (Ref 53-58). These sensors are much simpler than a LIBS system, but each sensor is designed to measure a particular element, so multiple sensors are required for the simultaneous detection of multiple elements.

In galvanic cell-based chemical sensors, two electrodes (reference and sensing) are separated by an electrolyte, such that a voltage is generated across the electrolyte. The magnitude of the voltage is proportional to the ratio of the logarithms of the concentrations of the element to be detected at the two electrodes. Thus, the measurement of this voltage (typically hundreds of millivolts) for a known reference potential can be used to determine the concentration at the sensing electrode.

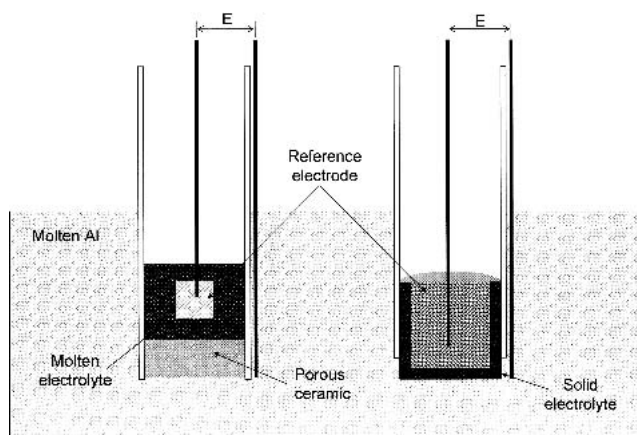


Fig. 2 Schematics of liquid and solid electrolyte-based chemical sensors (Ref 70). E is the cell voltage.

Solid and liquid electrolytes have been used in chemical sensors for use in molten aluminum. Molten electrolytes typically have faster response times compared with solid electrolytes. However, as shown in Fig. 2, the sensor design is slightly more complicated, because a porous ceramic is required to contain the molten electrolyte. In addition, during long operating times, some of the electrolyte can leach into the alloy, which can lead to erroneous readings. Sensors based on solid electrolytes are typically simpler and more durable compared with those based on molten electrolytes. However, the conductivities of solid electrolytes are typically lower than those of liquid electrolytes, so improved durability is sometimes achieved at the expense of a slower response time.

Molten electrolytes, specifically magnesium chloride-based electrolytes, have been used in the development of Mg sensors for molten aluminum (Ref 59-65). Solid electrolytes, including β -alumina (Ref 66, 67) and magnesium fluoride (Ref 68-70), have also been used for Mg sensors, but additional work is needed to avoid Na interference with β -alumina and to develop cost-effective fabrication techniques for fluoride electrolytes. Molten chloride-based sensors are more fully developed than solid electrolyte-based sensors and are likely to be commercially available first.

One of the most widely studied electrolytes is β -alumina, which is a sodium-aluminum oxide compound with a planar structure allowing for high ionic mobility. Although β -alumina can be doped with other ions, the most common material is a Na-ion conductor, which is suitable for use in a Na sensor. In addition to being used to monitor Na content (Ref 71-80), β -alumina has also been used for the controlled addition of Na by applying a current across the electrolyte (Ref 75). A Na sensor based on a fluoride electrolyte has also been reported (Ref 81, 82).

Solid electrolyte-based sensors have been developed for detecting several other elements including Sr (Ref 66, 82-84), Li (Ref 67, 76, 78, 85-87), Ca (Ref 66), and P (Ref 88). Electrolyte-based sensors are well-suited for measuring alloying elements, which are more active than the base element, but the detection of less active elements, such as Cu or Si in aluminum, is more difficult.

3. Gas Content

The most commonly used techniques for measuring the hydrogen content in molten metal involves analyzing the hydro-

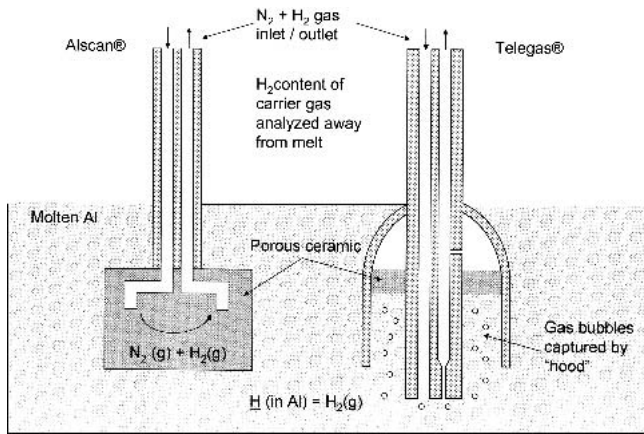


Fig. 3 Schematics of the AlScan and Telegas systems for the measurement of hydrogen content in molten aluminum (Ref 15)

gen content in a sample taken from the melt (Ref 1, 24, 89-91). The sample is heated in a vacuum, or nitrogen gas, and the amount of evolved hydrogen from either the solid or liquid sample is determined. To provide a faster response, techniques have been developed to determine the hydrogen content of the sample while it is solidifying. The most common of these techniques is the reduced pressure test (RPT) in which a sample is taken from the melt and placed directly in a vacuum chamber (Ref 15, 89, 92, 93). The change in pressure during solidification and the amount of porosity in the solidified sample are used to determine the hydrogen content in the melt. A variation on the RPT is the initial bubble test, in which observation of the initiation of bubbling during cooling of the molten sample is used to determine the hydrogen content (Ref 24). Although faster than laboratory tests, a more rapid response would improve process efficiency. More importantly, variable sampling procedures and inconsistent analysis can lead to erroneous results, so improved techniques for hydrogen measurement are needed.

3.1 On-Line Measurements Using a Carrier Gas

One approach to the on-line measurement of the hydrogen content in molten metals is to pass an inert gas (typically, nitrogen) through the melt and then analyze the hydrogen content in the gas (Ref 1, 15, 24, 89, 94-98). Schematics of commercial versions of this type of sensor (AlScan, Bomem, Quebec, QC, Canada, and Telegas, Alcoa, Pittsburgh, PA) are shown in Fig. 3. The nitrogen gas is either bubbled through the melt or passed through a porous ceramic that is in contact with the melt, so that the partial pressure of hydrogen in the gas can equilibrate with the hydrogen dissolved in the molten aluminum according to reaction 1:

$$H(\text{in Al}) = \frac{1}{2} H_2(\text{g}) \quad (\text{Eq 1})$$

such that the hydrogen partial pressure in the carrier gas is proportional the amount of dissolved hydrogen to the one-half-power according to Sievert's law.

The outputs of the AlScan and Telegas systems are more precise and less sensitive to operating procedure compared with the RPT test, but the results are generally less reliable compared with those from laboratory analytical techniques.

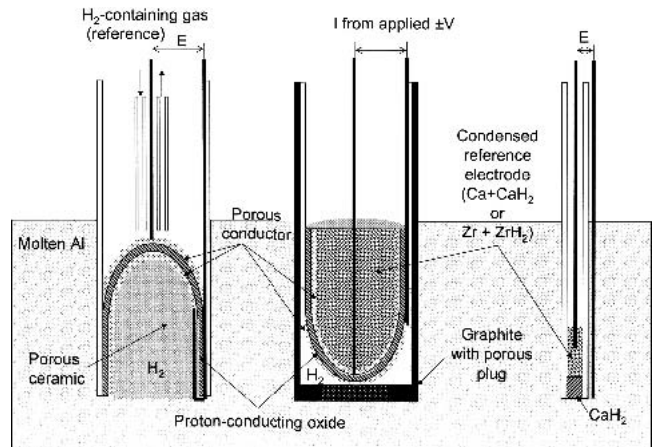


Fig. 4 Schematics of solid electrolyte-based hydrogen sensors (Ref 100, 105-107, 118, 119). E, I, and V are the cell voltage, current, and applied voltage, respectively.

However, the measurements take 10 to 20 min, and thus can be used for on-line process control.

3.2 Solid Electrolyte-Based Hydrogen Sensors

Another approach to measuring hydrogen content in molten aluminum is to use a galvanic cell similar to those described above for determining chemical composition, in which the cell voltage is proportional to the logarithm of the hydrogen content (Ref 99). Schematics of some solid electrolyte-based sensors are shown in Fig. 4. Gee and Fray (Ref 100) showed that a calcium hydride electrolyte with a Ca reference electrode could be used to measure hydrogen content (right side of Fig. 4). However, this system was not developed due to stability problems. The discovery of proton-conducting oxides provided a source of potential electrolytes with improved stability. Hydrogen sensors using the most studied of these proton-conducting oxides, strontium cerate (Ref 101-103), were found to have poor stability in molten aluminum. The proton-conducting oxide that has shown the best performance in molten aluminum is calcium zirconate (Ref 104-116), which is typically doped with indium oxide to increase the ionic defect concentration. Figure 4 (left side) shows a schematic of the calcium zirconate-based hydrogen sensor. There are two differences between this hydrogen sensor and the chemical sensors shown above in Fig. 2. One is that the reference electrode is a gas, which requires tubes for the transport of gas to and from the probe. The other is that the sensing electrode is not in direct contact with the melt. The sensor measures the partial pressure of hydrogen gas in the porous ceramic in contact with the melt.

A proton-conducting electrolyte can also be used to pump hydrogen into or out of the melt by passing a current through the electrolyte (Ref 117). Although this is not an effective method for removing large amounts of hydrogen, it can be used in a hydrogen sensor (the middle of Fig. 3) (Ref 118, 119). In this sensor, a voltage is applied across the electrolyte to pump hydrogen from one electrode to the other. The ratio of the currents in the forward and reverse directions is related to the hydrogen content in the melt. Another important feature of this sensor is that a solid reference electrode is used, which eliminates the need for the transport of a reference gas to and from the probe.

One of the challenges in the electrochemical measurement

of hydrogen is possible interference from oxygen and water. Conductivity measurements (Ref 104) indicate that calcium zirconate may be an oxygen-ion conductor in the very low oxygen partial pressures that are present in molten aluminum. However, oxygen-ion conductors have been shown to respond to hydrogen partial pressure (Ref 120-123), due to the establishment of a mixed potential, so this is not necessarily an insurmountable problem.

4. Inclusions

Two techniques for characterizing inclusions in molten aluminum are the porous disk filtration apparatus and the liquid aluminum inclusion sampler (LAIS), both of which involve passing the molten metal through a filter that collects the inclusions (Ref 1, 2, 23, 89, 124). The weight of the residue is used to determine the number of inclusions. Metallographic analysis of the filled filter can provide information on the sizes and types of inclusions. The prefil footprinter is a derivative of the LAIS technique in which, in addition to the collection of the filtrate, the filtration rate is recorded. The slope and curvature of a plot of the amount of filtrate as a function of time is used to evaluate the number of inclusions. Questions have been raised regarding the reliability of using results from the prefil filtration rate curves (Ref 3, 24), although a subsequent report has suggested that the reliability has been improved with a new crucible (Ref 125).

Chemical analysis has also been used to evaluate inclusion contents, but chemical composition is not always directly related to inclusion content (Ref 2, 23). Another technique for evaluating inclusion content is the K-mold tests (Ref 24, 89). In the K-mold test, a sample with a specific geometry is cast and fractured. The fracture surfaces are examined, and the number of inclusions is recorded.

As with chemical composition and gas content, a technique for on-line determination of the inclusion content would provide information to allow for better control of the processes for removal of inclusions.

4.1 Electromagnetic-Based Inclusion Detection

The electrical properties of inclusions are typically different from those of the molten metal, so electromagnetic probes can provide a means for measuring inclusion content. One such sensor is the liquid metal cleanliness analyzer (LiMCA). The system is derived from the Coulter counter (Ref 126), which is based on a change in the effective resistivity of a fluid upon the incorporation of particles. Specifically, if an electrical current (I) is passed between two electrodes, which are separated by a conducting fluid passing through an orifice with diameter D , the ohmic voltage drop (ΔV) resulting from the change in resistance (ΔR) between the electrodes is related to the size of the particle (d) according to:

$$\Delta V = I \cdot \Delta R = I \cdot \frac{4 \cdot \rho \cdot d^3}{\pi \cdot D^4} \quad (\text{Eq 2})$$

where ρ is the resistivity (Ref 127). This principle has been used in the development of sensors for measuring the amount and size of inclusion in molten aluminum (Ref 2, 23, 128-142).

As shown in the schematic in Fig. 5, the LiMCA system uses a tube with a small orifice. Metal is forced in and out of

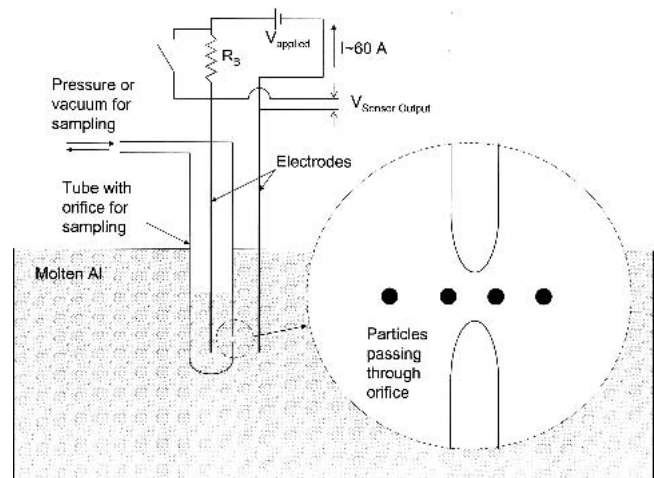


Fig. 5 Schematics of the LiMCA system for the detection of inclusion in molten aluminum (Ref 128, 129, 132-134). R_B , V_{applied} , $V_{\text{sensor output}}$ and I are the resistance, applied voltage, output voltage, and current, respectively.

this tube (through the orifice) by alternatively evacuating and pressurizing the inside of the tube. As a particle passes through the orifice, which is referred to as the electric sensing zone, a voltage change is measured according to Eq 2. The magnitude of the voltage change can be used to estimate the particle size, and the number of voltage changes can be used to count the number of particles. The duration of the voltage change is related to the transient time, which is related to the size of the particle. Thus, the relationship between the transient time and the magnitude can, in some cases, be used to determine the density of the particle (Ref 132). This analysis can be complicated if the particles are not spherical in shape.

The detectable particle size depends on the size of the orifice, but particles of 15 to 20 μm can be detected (Ref 133). In addition to strongly affecting the magnitude of the voltage (Eq 2), the size of the orifice must be properly selected to obtain reliable results. Too large an orifice may allow multiple particles to pass through the orifice simultaneously or in close succession, which may be interpreted as a single particle. On the other hand, too small an orifice may restrict particle movement, such that particles may become stuck or block the orifice, and the inclusion level may be underestimated.

Other techniques based on changes in electrical properties have taken advantage of the possibility of measuring electromagnetic properties without direct contact with the molten metal. For example, the measurement of the electromagnetic field induced by flowing current through a coil wrapped around a tube with molten metal has been used to measure inclusions and bubbles in molten Na (Ref 143). The induced electromagnetic field can also generate a force on the particles, and this phenomenon has been used to detect inclusions in molten aluminum (Ref 2, 144). Specifically, an electromagnetic Archimedes force is generated to force the particle to the liquid metal surface, and the particles at the surface are then detected and characterized with an optical imaging system.

4.2 Acoustic-Based Inclusion Detections

Ultrasonic probes have been used to characterize the inclusion content of molten aluminum (Ref 2, 23, 27, 145-157). Two such ultrasonic detection systems are shown in Fig. 6. In one

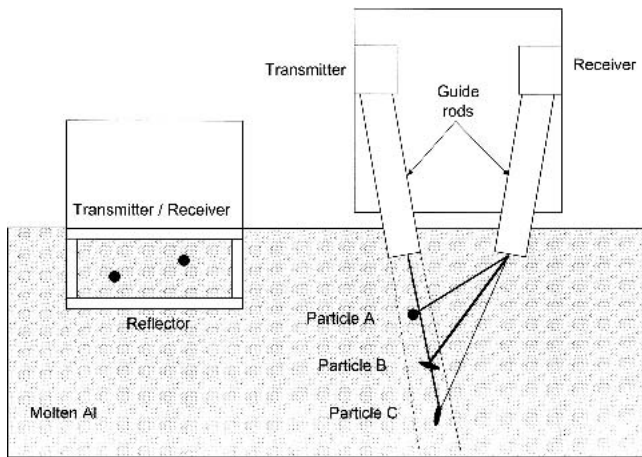


Fig. 6 Schematics of ultrasonic-based inclusion detection systems (Ref 147, 151, 152)

case (Ref 2, 23, 27, 145-148), the ultrasonic wave is transmitted down to a reflector, so that the particles measured are those that pass between the transmitter and the reflector. The other case (Ref 149-157) does not have a reflector, so that particles all the way to the bottom of the crucible can be detected. The former case, with the reflector, samples a smaller volume of metal but produces a simpler and, thus, more easily interpretable signal. The latter case, with more directed ultrasonic waves, monitors the melt to the bottom of the crucible but requires more analysis of the signal to extract the larger amount of useful information.

The particles are detected by increasing the amplification until a signal is measured, so that the smaller the particle, the larger the amplification required for detection. The signal generated can be affected by the shape and orientation of the particle. For example, in Fig. 6 the signal generated by particle B would be larger than that generated by particle C, due to the relative orientations to the ultrasonic excitation. There is a time lag between the transmitted and the received signals. This time lag increases with increasing distance from the melt surface, and thus, can be used to determine the vertical position of the particle. The horizontal position of the particles can be determined by focusing ultrasonic signal using waveguides. The development of sensors for use in molten zinc has shown that this spatial resolution, as well as the signal-to-noise ratio, can be improved through improvements in the waveguide materials (Ref 158-162). The number of reflected signals corresponds to the number of particles. Thus, the ultrasonic signal provides information on the size, position, and number of particles.

The minimum size of the particle detected depends on the frequency of the ultrasonic wave. Particles 10 to 15 μm in size have been detected using a 2.25 MHz signal (Ref 153). In addition, large numbers of small particles (i.e., those smaller than the detectable limit) result in an overall attenuation of the signal. Thus, in addition to the characterization of the sizes and distribution of large particles, clouds of small particles, which can also affect casting quality, can be detected (Ref 152).

In addition to particles, ultrasonic waves can be reflected from other interfaces, such as molten-metal/flux or solidified-metal/molten-metal interfaces, and can generate signals. For example, the signal from the crucible bottom, and a similar signal from the flux/molten-metal interface has been used to measure the level of molten aluminum below the cryolite dur-

ing the reduction of alumina by the Hall-Heroult process (Ref 163). In addition, ultrasonic measurements can be used to monitor metal levels, the position of molten metal fronts, and the properties of liquid-solid mixtures during the thixotropic processing of metals (Ref 164-166).

5. Conclusions

Although improvements in the reliability, sensitivity, and response times of techniques for monitoring the quality of molten metals have improved the quality of cast products, the additional refinement of existing techniques and the development of new techniques can provide further improvements in process control. The integration of monitoring techniques with degassing, filtration, and other processes will allow for the consistent production of high-quality castings.

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