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APPLICATION OF HEAT PIPE TECHNOLOGY IN THERMAL ANALYSIS OF METALS

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Thermal Analysis has been used in foundry applications to assess the quality of the melt before casting. The high-end thermal analysis techniques such as DSC or DTA are expensive and not suitable for foundry applications. The Computer-Aided Cooling Curve Analysis (CA-CCA) method based on one thermocouple has been widely used as a batch process with poor control over heat extraction and cooling rates during solidification.

A heat pipe apparatus has been developed as a thermal analysis tool. The apparatus can assess the melt quality more accurately, as well as, allow for better control of heating and cooling rates. Moreover, the solidification process can be modeled more accurately, and thus the casting parameters affecting the casting quality can be closely simulated and consequently controlled. In this paper the principles of a heat-pipe assisted thermal analysis system are highlighted. The advantages of the new system are described and the possibility of its adoption in melt assessments is discussed.

Keywords: *aluminum, control, cooling rate, heat pipe, solidification, thermal analysis*

Introduction

In the metal foundry, thermal analysis is often used to monitor the quality of the molten alloy prior to pouring. In the case of aluminium alloys, for example, thermal analysis has been used as a tool of detecting the extent of grain refining and the degree of eutectic modification. Although the beneficial role of thermal analysis to obtain on-line feedback on the above parameters is now well understood, the method of performing thermal analysis has hardly evolved over the past sixty years.

The typical conventional thermal analysis apparatus consists of a sampling cup, a microcomputer for data acquisition and analysis, and appropriate software. After pouring, the temperature of the solidifying sample is recorded as a function of time as shown in Fig. 1 alloys.

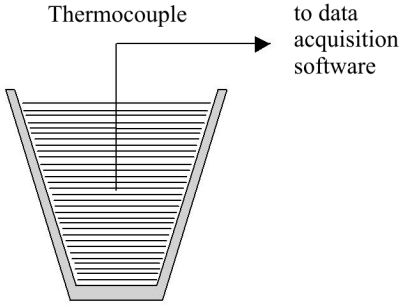

Fig. 1 A conventional thermal analysis apparatus

Although simple and inexpensive, classical thermal analysis methods have their own limitations. Besides being batch operations, these methods offer no precise control over the cooling rate of the solidified melt, and the cooling rate is controlled by the physical and thermal characteristics of the sampling cup. Therefore, to simulate a range of cooling rates, several, and sometimes different materials and/or shapes of cups have to be used. Control over the cooling rate becomes important when it is desired to use thermal analysis to predict the microstructure of a casting, which solidifies at a particular rate. To overcome limitations associated with the classical methods of thermal analysis, a novel lab-scale probe, based on heat pipe principles, has been built and tested for the thermal analysis of aluminum.

Heat pipes in metallurgy

Despite considerable development in the theory and technology of heat pipes and their industrial applications, there are fields, such as metallurgy, in which the heat pipe is slowly finding grounds for application. A number of papers concerned with specific applications of the heat pipe in metallurgy can be found in the open literature. However, those concerned with metal casting are few, particularly relative to the extensive attention, which has been devoted to the basic studies of the

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heat pipe. The possibility of exploitation of the heat pipe in solidification was first discussed by Bahadori [1] and subsequently by Steininger and Reed [2]. Bahadori studied analytically the prospect of controlling the solidification rate by the application of the heat pipe principal. However, his results were only illustrative and without any experimental verification. The present work illustrates a new application of heat pipes in the filed of metallurgy.

The use of heat pipes as a TA tool

A new way of intelligent evaluation of melt quality through thermal analysis has been devised. The operating principles of the new thermal analysis system are based on heat pipe technology. In order to better rationalize the use of heat pipes, a brief introduction highlighting the principles of heat pipe operations is given below.

In simple terms, a heat pipe is an innovative high-heat transfer device capable of transferring large amounts of heat from a source to a sink by taking advantage of the high heat transfer rates associated with the evaporation and condensation of a working fluid placed within the device. Heat is absorbed at the heat source (evaporator) and dissipated at the heat sink (condenser) at approximately isothermal conditions. Since heat is transferred by utilizing a phase change, the thermal resistance is small and thus a large amount of heat is transferred within small temperature differences over the device's working length [3].

The three basic components of a heat pipe are the working fluid, the pipe container, and the capillary structure (the wick). The driving force for liquid movement in a heat pipe is due to the different radii of curvature between the evaporating liquid in the evaporator section (heat source) and the condensing vapor in the condenser section (heat sink) of the pipe. These differing radii of curvature cause differing capillary surface tensions, which in turn give rise to a net pressure difference that provides the driving force needed to pump the condensate through the capillary structure and return it to the evaporator end of the heat pipe where the process is repeated. This process will continue as long as the flow passage for the working fluid is not blocked and sufficient capillary pressure is maintained [4]. The principles of heat transfer and fluid flow inside the heat pipe are illustrated in Fig. 2. Further details on the principles of operation of heat pipes are given in [5].

Design of the heat pipe thermal analyzer

The new thermal analysis system takes advantage of the heat pipe characteristics by adopting the technology to

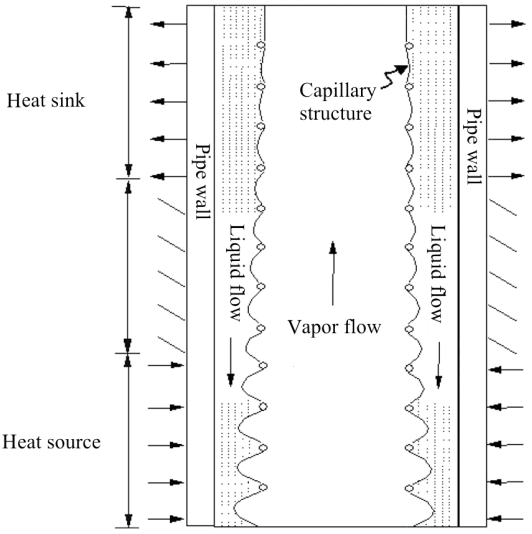

Fig. 2 Schematic diagram of the principles of heat transfer and fluid flow within a heat pipe

the thermal analysis of aluminium alloys. Several design considerations had to be taken into account in order for the new system to work satisfactorily. The most important being the choice of the working substance, cooling medium, and the materials, size and maximum heat transport capability of the pipe. The choice of the working substance was based on the desired thermophysical properties of the working substance and the chosen aluminum alloys. Potassium metal was used as the working substrate in the present study (boiling temperature = 1047 K). The body of the pipe consisted of two concentric tubes (Fig. 3). Since the sample being solidified is contained within the inner tube, the inner diameter of the inner tube dictated the size of the sampling region. The sample should be large enough to represent the whole batch of the melt, and at the same time, small enough so that it can be easily cooled and reheated.

The selection of material for the heat pipe vessel was governed primarily by the working environment

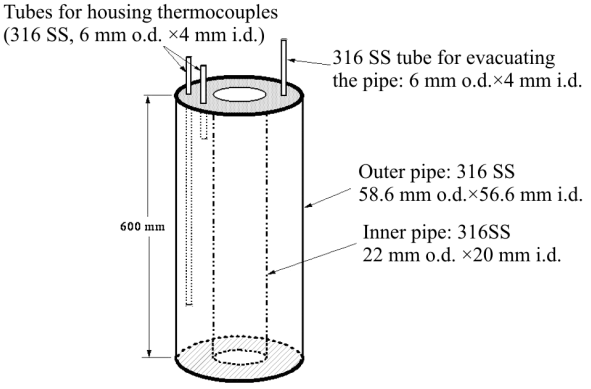

Fig. 3 Dimensions and materials of the laboratory scale heat pipe container

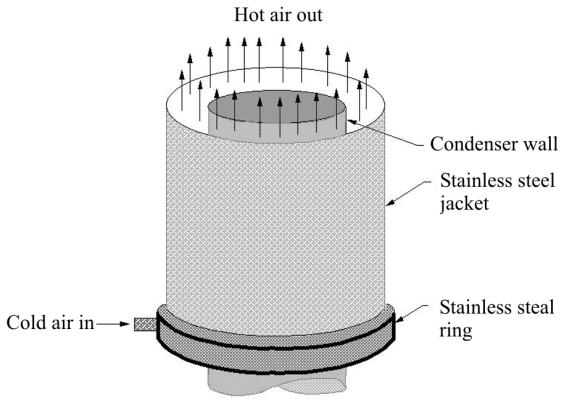

Fig. 4 A schematic of the condenser cooling arrangement

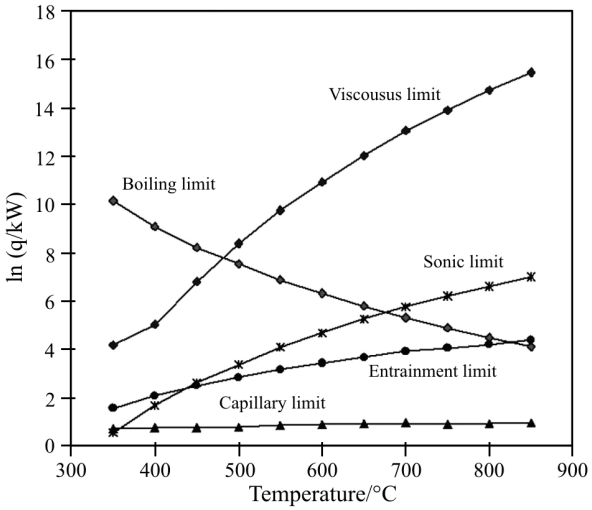

Fig. 5 Possible heat transfer limits according to the present heat pipe design

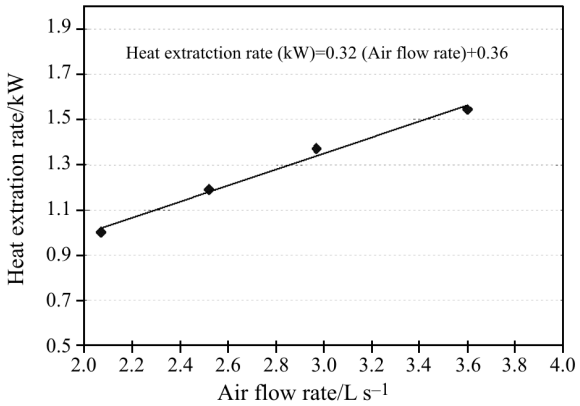

Fig. 6 Experimental results showing the overall heat extraction rate at steady state as a function of air flow rate

and cost. Type 316 stainless steel was deemed suitable for this application. The problem of stainless steel dissolution, when in contact with liquid aluminum, was overcome by using a graphite block (1 cm thick) to protect the evaporator outer surface, and by coating the inner surface with boron nitride. A 100-mesh stainless steel wick (type 304) was wrapped around the inner surface of the outer pipe and the outer surface of the inner pipe to form the capillary structure. Air was chosen as the cooling medium. As illustrated in Fig. 4, air was directed to pass through the cooling jacket designed to envelop the condenser section of the heat pipe.

Heat transfer considerations

The possible limits to heat transport within, and in and out of the heat pipe were determined to ensure that the pipe would not be hindered by these limits during its normal operation. These limits are given as a function of the operating temperature in Fig. 5. Although a first look at Fig. 5 may imply that the heat transport capacity of the present heat pipe would be limited primarily by the capillary limit, in reality this is not necessarily the case. That is, if the liquid is to be uniformly distributed over the entire inner evaporator surface and the pipe is in a horizontal position, then capillary limit would indeed be the limiting heat transport case. However, the present heat pipe was intended to work in a vertical position, and the steel mesh (wick) covered the whole interior surface. As the vapor condenses in the condenser section, the condensate that flows down the pipe will start to spread circumferentially before it enters the evaporator section. As the condensate enters the evaporator section it has already covered the whole wick surface. The capillary limit is not a 'real' limit for the present heat pipe configuration. Thus, it is of little concern for the present study.

Figure 6 shows experimentally-determined heat extraction rates for the current system. As can be seen, the maximum heat extraction rate for a typical aluminum melt, at the highest air flow rate possible with this system (4 L s^{-1}), is about 1.7 kW. Thus, according to the present design parameters and the operating conditions, the operation of the heat pipe should not be hindered by any of the limits (Fig. 5).

TA tests using the heat pipe system

In the present system thermal analysis of melts is carried out without the need for physically removing a sample from the melt, as is the case with the conventional thermal analysis techniques. A typical thermal analysis test is performed by gradually lowering the evaporator portion of the heat pipe into the liquid bath to within 10–12 cm below the melt surface, where about 0.3% of the total melt mass (approx. 10 kg of an aluminum alloy) filled the sampling region (evaporator core) of the pipe. Figure 7 shows a schematic of an

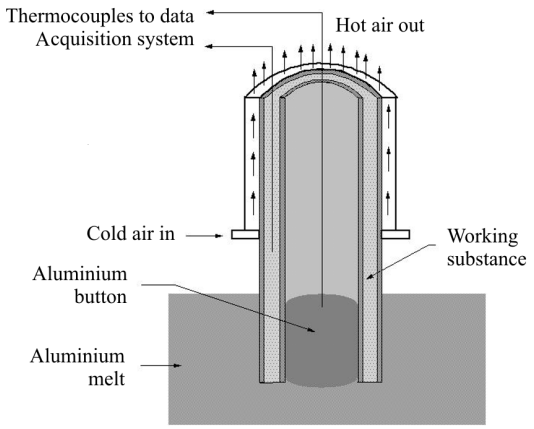

Fig. 7 The heat pipe thermal analyser is lowered into the liquid bath

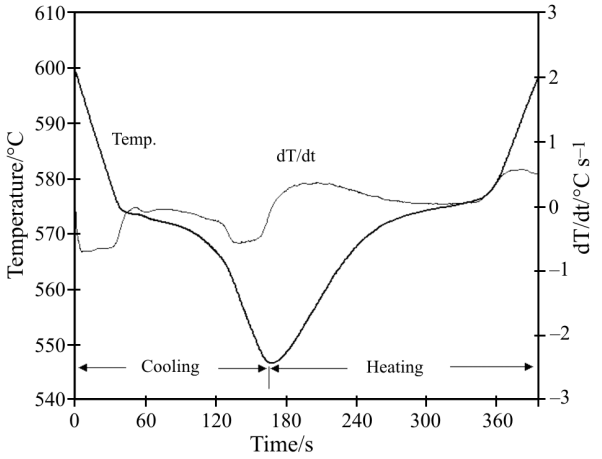

Fig. 8 The cooling-reheating pattern of A413 alloy sample inside the heat pipe

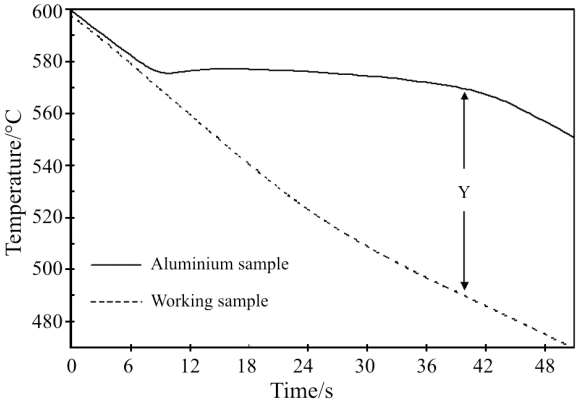

Fig. 9 Thermal behavior of the heat pipe during the cooling process

axial cross section of the heat pipe with its evaporator section inside the aluminum melt. The isothermal condition of the working substances is monitored by placing two thermocouples at both ends of the working substance chamber (Fig. 3). Once equilibrium is achieved between that part of the melt that now resides in the pipe (the sample to be solidified) and the rest of the melt, the system is then instructed to start cooling the sample. The amount of air flow into the system controls the cooling rate. Therefore, it is possible to have a range of cooling rates simply by changing the flow rate of air into the system. Once the cooling curve of the solidified sample has been recorded, the probe can be instructed to remelt the sample and await instructions for running another test. Thus, a thermal analysis cycle consisting of two stages (melting and cooling) can be obtained (Fig. 8).

It should be noted that it is the working substance that is directly cooled by the cooling air and not the aluminum sample itself. Cooling the sample indirectly (through the working substance) allowed a better control over the heat extraction as opposed to the conventional methods of cooling. The relationship between the working substance temperature and that of the aluminum sample using the heat pipe thermal analysis system, Fig. 9, was described in a previous publication [6], and is provided here for the purpose of giving the reader a comprehensive description of the system. As can be seen, the difference, (Y), between the temperature of the working substance and that of the aluminum sample starts at a very small value and increases as metal solidifies. This difference in temperature creates a driving force allowing the sample to maintain steady cooling even at lower temperatures. Although one would expect higher cooling rates of the sample when the driving force becomes larger, this is not always the case in this system. This is due to the fact that when in the liquid state, the aluminum sample is in good contact with the heat pipe wall, and any small temperature changes in the system would affect the temperature of the sample. However, as the sample solidifies, it starts to shrink. The sample shrinkage gives rise to a thermal resistance between the sample and the heat pipe wall. If correctly controlled, this resistance compensates for the increase in the driving force, thereby, allowing the sample to cool at a relatively constant rate. As a result, unlike conventional technique, the cooling rate can be controlled at different stages during solidification of the sample.

Cooling curves produced by conventional and heat pipe TA methods

Figure 10 shows cooling curves of 413 aluminum alloy (a eutectic alloy with $T_{eut} \cong 577$°C). Figure 10a shows a typical cooling curve obtained by a conventional method (permanent mold casting), whereas Fig. 10b and c present two cooling curves acquired by the new

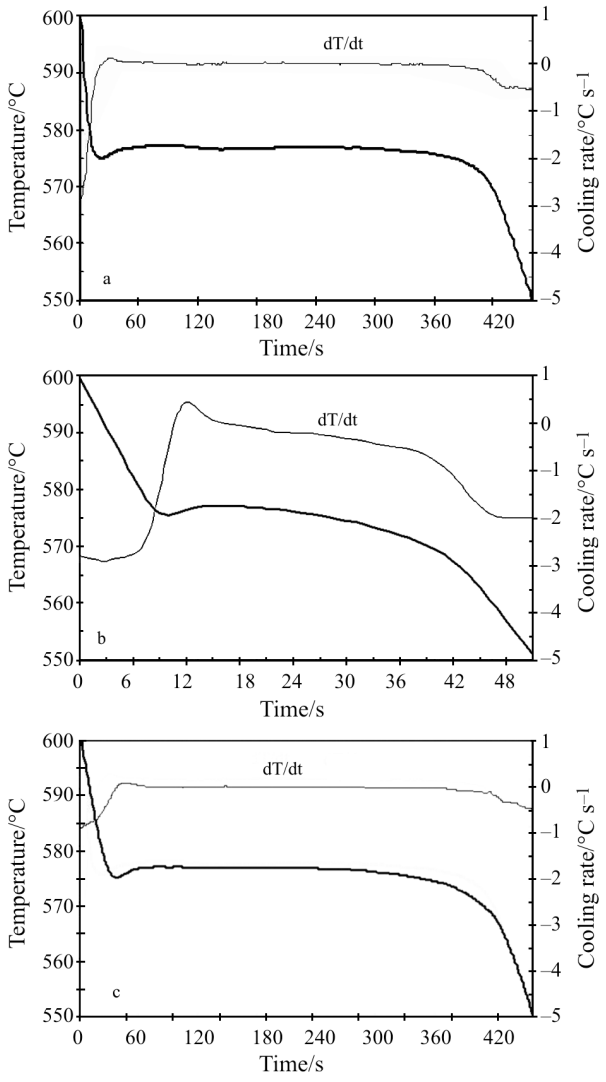

Fig. 10 The cooling curve of 413 aluminum alloy; a – obtained by the conventional method, b – obtained by the heat pipe probe at a high cooling rate and c – obtained by the heat pipe probe at a low cooling

probe at high and low cooling rates, respectively. As can be seen, curves acquired by the new probe compare very well with those obtained by the conventional method. However, in terms of cooling rates, these curves are quite different. Comparison of Fig. 10a with Fig. 10b shows that although the two curves have approximately equal initial cooling rates (≅ 3°C s^{-1}), these cooling rates become quite distinct at later stages of solidification. In the conventional case the cooling rate decreases from 3°C s^{-1} at the beginning of solidification to 0.6°C s^{-1} at the end of the eutectic plateau. However, Fig. 10b shows that the difference in cooling rate from start to end of solidification is only about 1°C s^{-1}. Moreover, comparison of Fig. 10a and c shows that although the initial cooling rates are different in the two cases, these rates become approximately equal as the temperature of the alloy falls below the eutectic temperature. The cooling curves show that unlike conventional technique, a constant cooling rate can be achieved with the new system.

The control of cooling rate during thermal analysis is of paramount importance, as the structure of cast metal is a sensitive function of the cooling rate during solidification. For example, control of cooling rates can be an asset in cases where the intermetallic reactions that are detrimental to the final cast product are to be detected, and if possible, depressed by choosing the appropriate rate. Since the detection limit of these reactions is a strong function of the cooling rate, then a system that could be set to give a predetermined cooling rate is vital. In this work, a cooling-rate control scheme has been devised, which is capable of changing the solidification rate at any time or temperature during the thermal analysis process. The method allows an aluminum sample, residing in the core of the pipe, to be cooled at a high cooling rate until a predetermined temperature range, at which certain reactions are believed to occur. The cooling rate can then be lowered to allow for the easy detection of any possible reactions. Once the temperature drops below the lowest temperature of this range, the cooling rate of the sample can then be increased again. This procedure allows a shorter time for each thermal analysis, and gives more flexibility over the control of the solidification process. The reader is referred to [7] for a detailed description of the control scheme in the heat pipe thermal analysis system.

As mentioned above, the freezing rate of the sample inside the pipe is determined by controlling the amount of air introduced into the system per unit time. The air flow rate is, in turn, controlled by a motor-driven metering valve. The valve is controlled by providing a control signal to a controller unit. The controller has two inputs, control and target. During operation, the controller will attempt to position the valve such that the control voltage is equal to the target voltage. The target voltage can be supplied either through the connections on the controller unit, or generated internally. The target value, X_T, can be used to control any of the acquired data (X_I), where X can be the cooling rate of the sample or working substance, air flow rate, etc. Based on the sample mass inside the pipe (≈105 g), the heat extracted during the cooling process was calculated (assuming a constant heat capacity of the sample of 963 J kg^{-1} K^{-1}) and the net heat extraction was correlated to the air flow rate (Fig. 11). Figure 12 shows a cooling curve of aluminum A319 alloy (Al – 7%Si – 3.5%Cu), where a predetermined cooling rate of 2.1°C s^{-1} (air flow rate of 17.6 L s^{-1}) was used.

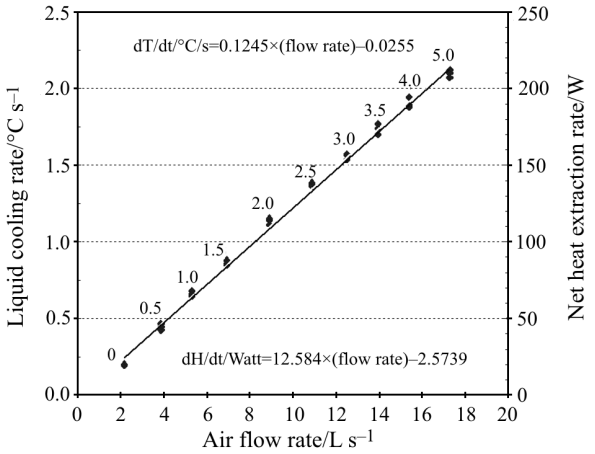

Fig. 11 Liquid cooling rate *vs*. air flow rate (the numbers 0-5 indicate the instructed input controller signals)

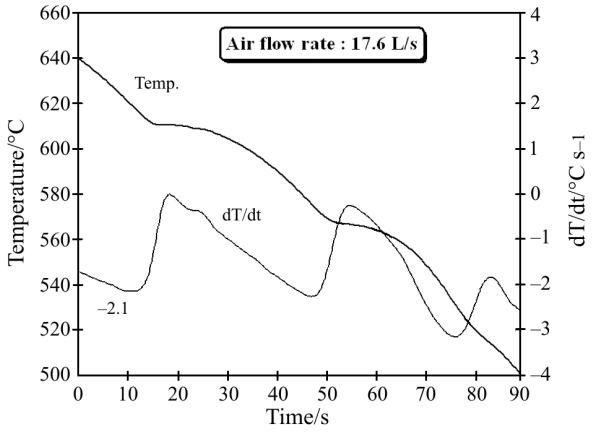

Fig. 12 A cooling curve of A319 alloy acquired at a controlled air flow rate

Formation of intermetallics

Iron is one of the most harmful impurity elements in aluminum alloys. Iron contents of more than 0.7% [8, 9] result in the precipitation of thin, coarse needles referred to as the β-phase (Al_5FeSi). This intermetallic compound is detrimental to ductility, fracture toughness and fatigue properties [9]. It is, therefore, of interest to detect the precipitation of Al–Fe–Si intermetallics during solidification. The heat pipe thermal analysis system has been used to detect, and consequently suppress, the formation of the above intermetallic compounds. Figure 13 shows a cooling curve of aluminum A319 alloy (containing 0.65% iron) where the control system was used to vary the cooling rate in order to detect the intermetallic reaction leading to the formation of iron-bearing β-phase. As seen, the flow rate was initially set at 10.6 L s^{-1}, and then decreased to 4 L s^{-1} in the temperature range 612.4–574.3°C. At 574.3°C the rate was raised again to 10.6 L s^{-1}. Changing the cooling rate during the solidification process from a high to low value allowed

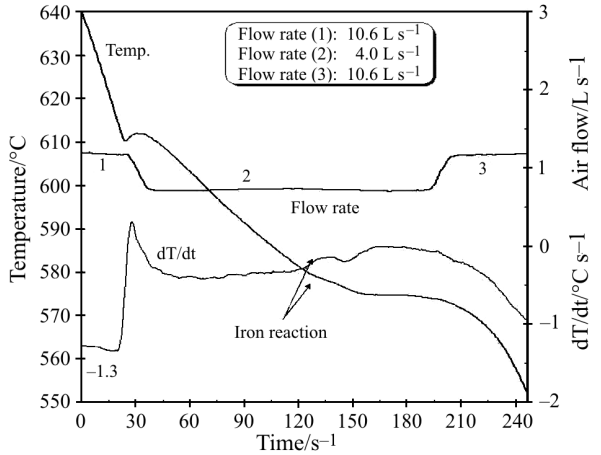

Fig. 13 The effect of cooling rate variation on the detection of iron-bearing reactions

the detection of the β-phase reaction, and decreased the time required to acquire the cooling curve. Once the temperature range at which this reaction takes place is known, the cooling rate can be adjusted in such a way that the formation of this phase is depressed.

Degree of grain refining and eutectic modification

The capability of the new thermal analysis system to detect the degree of grain refining and the level of eutectic modification of aluminum–silicon alloys was also investigated. In an article previously published on this subject [10], it was illustrated that the system showed excellent sensibility to detecting the degree of grain refining and the level of eutectic modification. In the case of grain refining it was shown that the heat pipe system was able to detect the change in grain size with an addition of the grain refining agent as low as 0.08%.

Reproducibility of results with the heat pipe thermal analyzer

One of the inherent characteristics of the conventional thermal analysis technique is its poor reproducibility of results. The temperature of the solidifying sample is dependent on the volume of the sample (and the sampling cup), and since exact volume control is often difficult, test accuracy can vary. For each set of experiments conducted using the new thermal analysis system, an average of three experiments were conducted in order to check the repeatability of the results. In order to demonstrate reproducibility, the results of a series of experiments done on different days are highlighted. The results shown in Table 1 relate to the applicability of the new system to detect the effect of strontium modification on the eutectic temperature of aluminum 413 alloy (at two different air flow rates of 7

Table 1 Repeatability of results

Day No.	Air flow rate/ L s^{-1}	Sr/ppm	Eutectic Temp./°C	Day No.	Air flow rate/ L s^{-1}	Sr/ppm	Eutectic Temp./°C
1	7	0	577.4	1	17	0	576.1
1	7	50	577.0	1	17	50	575.9
1	7	100	574.2	1	17	100	571.8
1	7	200	573.4	1	17	200	569.2
2	7	0	577.1	2	17	0	576.3
2	7	50	576.8	2	17	50	576.2
2	7	100	574.0	2	17	100	571.6
2	7	200	573.1	2	17	200	569.0
3	7	0	577.1	3	17	0	576.0
3	7	50	576.7	3	17	50	575.7
3	7	100	573.9	3	17	100	571.4
3	7	200	573.1	3	17	200	568.9

and 17 L s^{-1}). Taking into account a thermocouple accuracy of about ±2°C, the repeatability of the results can be considered to be very good. The main factor for the excellent reproducibility of the new system is due to the fact that the amount of heat extracted form one test to another can be kept constant.

Conclusions

In order to enable the foundryman to have better control over the properties of the cast product, an intelligent method of performing thermal analysis of aluminum alloys has been devised. The method, based on heat pipe technology, was designed to replace the conventional methods of thermal analysis. The new thermal analysis system can be characterized by being semi-continuous (as opposed to the batch process of conventional thermal analysis), in-situ, easy to use, environmentally friendly, and has excellent reproducibility. This intelligent system can perform thermal analysis tests at pre-determined and preset cooling rates. Coupled with a control scheme, the new thermal analysis system has been able to produce controlled cooling rates in the range of 0.1 to 4°C s^{-1}. Moreover, the new system allows a change of the cooling rate during solidification. This allows focusing on a temperature range of interest. And hence cooling time can be greatly reduced.

In terms of applicability, the new system has been shown to be applicable to the detection of important melt treatment parameters used in the aluminum foundry to improve the quality of the cast product. The system is able to detect the extent of grain refining and the degree of silicon modification. It can detect the liquidus and solidus arrests for inoculation and modification control and, in addition, it has the sensitivity to detect reactions leading to the formation of iron, magnesium, and copper bearing intermetallics [10]. The new device can be used to quantify the amount of iron in aluminum melts. The probe is capable of providing semi-continuous iron analysis in foundry melts which can be of use in processes such as low pressure casting where a cast iron feed tube leads to continuous increases in melt iron levels.

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References

1 M. N. Bahadori, AFS Cast Metals Research Journal, 6 (1970) 38.
2 J. Steininger and T. B. Reed, J. Cryst. Growth, 13 (1972) 106.
3 H. Imura, K. Sasaguchi, H. Kozai and S. Numata, Intern. J. Heat Mass Transfer, 11996.983, Vol. 26, No.8, p. 1181.
4 K. J. Wells, G. T. Colwell and J. T. Berry, AFS Transactions, 95 (1984) 429.
5 S. W. Chi, 'Heat Pipe Theory and Practice', Hemisphere Publ. Corp., Washington D.C., 1976.
6 M. Mahfoud, F. Mucciardi and J. E. Gruzleski, Proceedings of Light Metals, Montreal PQ, Canada, (1996) 161.
7 J. E. Gruzleski, M. Mahfoud and F. Mucciardi, Intern. J. Cast Metals Research, 10 (1998) 191.
8 S. G. Shabestari, Ph.D. Thesis, McGill University, 1994.
9 G. K. Sigworth, AFS Transactions, 98 (1989) 811.
10 M. Mahfoud, F. Mucciardi and J. E. Gruzleski, AFS Transactions, 105 (1997) 473.

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