Chapter 2

Thermal Analysis of Ductile Iron Casting

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Additional information is available at the end of the chapter

http://dx.doi.org/10.5772/intechopen.72030

Abstract

Pure metals solidify with a solidification front that is very well defined and a clearly delineated solid-liquid interface. Ductile cast iron solidification is characterised by a very thin solidified skin and appearance of different phases. The outer skin is formed being very thin in ductile iron; the expansion occurs due to graphite nucleation into the casting forces to the mould walls. With proper care taken while designing and during melt processing stage, quality ductile iron castings can be produced with minimal risering. With recent developments in sensing and storing instruments, it is now possible to see and measure structural transformations within the solidification in ductile iron castings very precisely. The shape of a cooling curve measured by a thermocouple mounted on a thermal analysis sample cup reflects the solidification process of the melted cast alloy for the given solidification conditions. By analysing particular cooling curve, the solidification start, eutectic arrests, recalescence, amount of undercooling and end of freezing temperature temperatures are generated. The thermal analysis data so generated will be used to study composition, soundness, chill and microstructure by analysis of cooling curve. The cooling rates measured in degrees per second at different stages of solidification sequence will be analysed and correlated with the properties of the castings to be produced from the same melt.

Keywords: ductile iron, thermal analysis, solidification, cooling curves, microstructure

1. Introduction

Ductile iron possesses the processing advantages of grey iron, such as low melting point, good fluidity, castability and machinability, and engineering advantages of steel, including high strength, ductility and wear resistance. Achieving the desired quality consistently at low cost in a production foundry is, however, still a challenge. Addition of small amount of cerium or magnesium to molten cast iron changes the shape of graphite from laminar to spheroidal, giving rise to spheroidal graphite iron or ductile iron. The rapid growth in industrial applications of ductile iron (DI) is driven by its versatility and high performance at low cost. It offers a good combination of tensile strength and ductility. This allows designers to select ductile iron for a wide range of applications. Ductile iron also offers cost savings compared to steel and malleable iron castings through higher yield and thereby lower melting energy. Formation of graphite during solidification leads to lower volumetric shrinkage in ductile iron (compared to steel), necessitating smaller and fewer feeders to prevent the formation of shrinkage defects. Further cost advances can be achieved by eliminating heat treatment of as-cast DI parts.

The near-spherical shape of the graphite nodules distributed evenly in the matrix phase of ductile iron enhances its ductility and impact resistance along with tensile and yield strength equivalent to a low carbon steel. While ferritic ductile iron can be used as 'as-cast', it may also be annealed to increase its ductility and low-temperature toughness. The pearlitic ductile iron has graphite spheroids in a matrix of pearlite, resulting in high strength, good wear resistance, moderate ductility and impact resistance. The most commonly used ferritic-pearlitic ductile iron containing both ferrite and pearlite in matrix offers a good combination of tensile strength and ductility with good machinability and low production costs.

The ductile iron castings are produced in a wide range of weight, from a few grams to a hundred tons or more, greatly varying in shape and size depending on the applications (**Figure 1**). Many forged and fabricated steel components are getting replaced by ductile iron castings, owing to their good combination of mechanical properties such as strength, wear resistance, fatigue strength, toughness and ductility coupled with economic production. The specifications of ductile irons with ferrite/pearlitic matrix with different grades are shown in **Table 1**. They are used in safety parts in automobiles, armatures, pumps and machine tools. They are also used in parts subjected to high pressure, such as pressure containers and hydraulics. Many welded assemblies and forgings used in governor housings, armatures and car parts (like brake calipers and gear housings, hydraulic parts, crankcases and blower buckets) are being replaced by ferritic ductile iron castings.

Figure 1. Ductile iron castings used in automobile, agriculture and sanitation.

http://dx.doi.org/10.5772/intechopen.72030

Country/region specification	Minimum tensile strength/elongation (N/mm²/%)								
Europe EN-GS CEN 1563:1997	350-22	400-18	400-10	450-10	500-7	600-3	700-3	800-2	900-2
UK BS2789 1985	350/22	400/18	420/10	450/10	500/7	600/3	700/2	800/2	900/2
USA ASTM A356 1993[1]	60-40-18	60-42-10	65-45-12	70-50-05	80-55-06	80-60-03	100-70-03	120-90-02	
Japan JIS FCD G5502 1995	350-22	400-18	400-10	450-10	500-7	600-3	700-3	800-2	
International ISO 1083 1987	350-22	400-18	400-10	450-10	500-7	600-3	700-3	800-2	900-2
Typical hardness (HB)	<160	130–175	135–180	160–210	170–230	190–270	225–305	245–335	270–360
Typical structures[2]	F	F	F	F & P	F & P	F & P	P	P or T	TM

[1]60-40-18 refers to minimum tensile strength (lbf/in²)—minimum proof stress (lbf/in²)—% elongation.
[2]The structures are: F, ferrite; P, pearlite; T, tempered; TM, tempered martensite.

Table 1. Specifications for ductile cast irons [1].

2. Production of ductile iron

Production of thin wall ductile iron (TWDI) castings is challenging due to faster solidification rates in thinner sections, affecting the graphite nodules and matrix phases, which in turn affect the mechanical properties. Ductile iron castings for the applications mentioned earlier are normally produced by sand casting or shell moulding processes, with cores used to create hollow internal sections. Sand casting is the most common process, due to the relatively low cost of production as compared to other manufacturing techniques. The metal composition and micro-constituents in the phases, as well as the cooling rates, control the mechanical properties of these TWDI castings. While the casting wall thickness affects the amount of heat to be transferred, the mould wall thickness affects the rate of heat transfer and thereby the rate of metal cooling during casting solidification. Kasvayee et al. examined localised strain at the location of the first microcrack to form during testing using digital image correlation (DIC) [10]. It has been found that inoculation process influences the microstructure and the fatigue resistance of heavy section pearlitic ductile iron castings [11]. The graphite shape factors were studied on the metallographic samples and evaluated as a function of the chemical composition, and the solid fraction was analysed [12]. The sudden temperature drop of liquid iron shows increased cooling rate, which will affect the solidification and microstructure in thin-walled castings [13].

2.1. Micro-constituent phases

The solidification process in thin wall ductile iron casting involves nucleation of micro-constituent phases such as austenite, graphite and/or carbides. The formation of these phases depends upon the chemical composition of cast metal, inoculation processing and cooling rate. The chemical composition (especially carbon and silicon) decides whether the DI is hypoeutectic, eutectic, or hypereutectic in nature. The control of chemical composition and inoculation is essential to avoid nucleation of primary carbides. Faster cooling and solidification in thin sections lead to the formation of primary carbides along with austenite dendrites in the structure instead of graphite nodules. The trapping of carbon by formation of carbide restricts the formation of graphite. The inoculation provides more number of nucleating sites on which graphite can grow.

The chemical composition controls the amount of graphite, ferrite and pearlite in the structure. The silicon as a graphitiser facilitates the formation of graphite instead of carbides. The increase in carbon and silicon increases the carbon equivalent of the casting. Addition of elements like copper and manganese decides the amount of pearlite in the matrix. Silicon is a strong solid solution strengthener; it reduces undercooling and avoids carbide formation by nucleating graphite. It increases volume fraction of ferrite and nodule count. Copper is a strong pearlite promoter; its addition up to 1% converts ferritic structure into pearlitic. Arsenic, tin and antimony promote pearlite and carbides and are hence kept to lower limits; their effect can be counteracted by cerium additions [2]. Achieving the desired microstructure in thin castings necessitates controlling chemical composition and cooling rates during solidification.

2.2. Cooling rates

In ductile iron castings, the carbon equivalent should be high (hypereutectic) in the case of very thin-walled components to avoid formation of primary carbides [2]. Thin walls cool faster due to the larger surface area available for heat transfer through mould, influencing the microstructure and mechanical properties of the casting. Design parameters such as the area of the thin wall, its proximity to gate and the availability of an adjacent heavy section influence the process limits of achievable wall thickness [3]. An increase in the number of graphite spheroids can be expected to result in a corresponding increase in the amount of ferrite formed at a given cooling rate and for a given austenite composition. This effect is readily observed in ferrite-pearlite structures of ductile cast irons.

The graphite nodule count and its size distribution are important in deciding the quality of the DI castings. The sufficient number of graphite nodules is required in order to avoid formation of carbides during solidification, especially in thin-walled castings because of the high solidification rate. The total number of particles decreases with increasing plate thickness. Austenite will contract during solidification, but this will be compensated by the expansion of graphite. It is especially important that there is sufficient graphite expansion in the last part of solidification when feeding from an external feeder normally is impossible. Sufficient graphite expansion in the latter stages of solidification is identified by nucleation of graphite nodules, giving many small graphite nodules in the microstructure [4].

The cooling rates and hence the solidification morphology in ductile irons depend on casting wall thickness. The ductile iron microstructures are very sensitive to actual solidification rate and inoculation processing in casting. The inoculation will provide nucleation sites (substrates) in the melt of specific size such that these nuclei will grow. The high nucleation rate in thin wall ductile iron (due to fast cooling) will thus show more number of graphite nodules in thinner compared to thicker sections. Further, higher growth rates in thicker sections will give less number of graphite nodules, but they will be coarser in size. The solidification rates are also controlled by heat transfer through mould; hence, the use of optimal sand wall thickness to achieve proper cooling rates becomes essential. Thus, the casting geometry (wall thickness), metal composition (especially micro-constituents) and mould configuration (cooling rate) together affect the solidification structure in ductile iron casting production.

2.3. Processing parameters

The properties of as-cast ductile iron are largely driven by liquid metal processing, including melt pretreatment, magnesium treatment and inoculation processing. The melt pretreatment involves controlling the initial sulphur content in the liquid metal so as to facilitate magnesium treatment. If the initial sulphur content is high, then it reacts with magnesium and forms magnesium sulphide, decreasing the effectiveness of magnesium treatment. Also, manganese reacts with sulphur and forms manganese sulphide; hence, Mn:S ratio is to be maintained to control the final properties of the ductile iron.

The magnesium treatment involves addition of magnesium and/or cerium in different forms to facilitate effective melt treatment and achieve spheroidisation of graphite during the solidification of the metal. The most common form is ferro-silicon-magnesium, which allows better magnesium recoveries (than other forms, owing to oxidising properties of magnesium). This helps utilise more amount of magnesium for spheroidisation purpose.

Mg treatment eliminates oxide bi-films and produces compact particles in ductile iron melt. Hence, it is usually followed by inoculation treatment, which increases the number of nucleation sites in ductile iron castings. Graphite nucleates on these particles, and its further growth is controlled by austenite dendrites. The shape and size of these graphite spheroids affect the mechanical properties of castings. The time span between inoculation treatment and pouring has a significant effect on elongation but less effect on the tensile strength and hardness of castings [5].

The ratio of ferrite to pearlite in the matrix and the morphology of graphite influence the mechanical properties of ductile iron castings. This also depends upon the cooling rate during solid-state (eutectoid) transformation, nodule count and alloying elements. The studies of nucleation and solidification help in controlling the final properties of varying thickness ductile iron castings. The ferrite being softer gives higher ductility but lower tensile strength than pearlite. Also, the graphite morphology plays an important role; deviation from spheroidal shape reduces the ductility and impact properties [6].

It is therefore important to understand how the different phases nucleate and grow during solidification in order to be able to control the casting process and achieve the desired set of mechanical properties in varying thickness ductile iron castings.

3. Thermal analysis

Recent developments in thermal analysis instruments make it possible to precisely measure and visualise the events within the solidification of iron samples. Data generated from thermal analysis can be used to study composition, soundness, chill and microstructure. The shape of the cooling curve measured by a thermocouple mounted in a thermal analysis sample cup reflects the solidification process of the melted cast alloy for the given solidification conditions. The cooling rates measured in degrees per second at different stages of the solidification sequence can be analysed and correlated with the properties of the castings to be produced from the same melt. Chronologically, the major parts of the curve are pre-liquidus, austenitic arrest, dendritic growth, eutectic solidification, end of freezing and the austenite transformation region. The normal cooling curve gives basic information about the solidification. Additional information can be obtained from the first derivative (DT/dt) of the curve. The cooling rate which is the first derivative calculated by change in temperature per unit time (dT/dt) in °C/s. The horizontal line is the zero line. If the first derivative is zero, it indicates that heat transferred from the solidifying casting equals the heat generated (evolved) due to phase transformation.

A method of thermal analysis of cooling curve is studied by different researchers. Carbon and silicon content can be estimated from cooling curves when iron is solidified in a tellurium-coated sand cup (the tellurium causes the iron to solidify as white iron rather than grey iron). Silicon is a major alloying constituent in cast irons, which raises the graphite eutectic solidification temperature and lowers the carbide eutectic range. To calculate C% and Si%, the austenite liquidus temperature (TAL) of the ductile base iron and the carbide eutectic temperature (CET) arrest temperatures are used. Once the eutectic composition is determined, the silicon and carbon compositions and carbon equivalent (CE) can be obtained by the following equations [7]:

$$\mathrm{TAL} = 0.556\,(2962 - 212.3\ \mathrm{C}\,\% + 0.25\ \mathrm{Si}\%) \tag{1}$$

$$\mathrm{CET} = 2085.4 - 22.7\ \mathrm{Si}\% \tag{2}$$

The cooling curves generated during solidification of alloys can be further analysed by the use of first-order and second-order derivatives to find out the temperature arrest points. The thermal analysis cooling curves can be used for optimisation of inoculation in ductile iron [8]. The minimum eutectic temperature (T_{min}) should be greater than 1140°C to avoid primary carbides in ductile iron, and the angle at the end of solidification in cooling rate curve (VPS) should be between 25 and 45°C to avoid secondary carbides. Furthermore, computer-aided cooling curve analysis can be used for evaluating latent heat evolved during solidification. Iron castings produced with identical chemical composition can have considerable variations in mechanical properties. With thermal analysis, it is possible to predict such variations and correct the melt before pouring.

The thermal analysis studies explore the effect of inoculation on holding time and fading of graphite nodules in ductile iron castings. The undercooling during solidification known as recalescence ($T_{max} - T_{min}$) depends on inoculation rate. High T_{min} indicates high inoculation effect and indicates that more graphite is precipitated. High recalescence is an indication of poor inoculation [9]. A lower VPS angle (**Figure 2**) indicates better protection against micro-shrinkage tendencies; however too low VPS angle will produce flake graphite. To compare two cooling curves, the separating distance of the two curves as well as the shape similarity of the curves should be considered. The segment of the thermal analysis curve from the liquidus temperature to the end of the eutectic solidification represents the entire solidification range.

3.1. Solidification cooling curves

The composition of the melt and its processing sequence will reflect in the cooling curve; in other words, cooling curve is a fingerprint of the melt. To predict the final microstructure and mechanical properties, the cup thermal analysis was adopted. Cups of the standard shape made in shell mould are used to pour the melt and generate cooling curves with the aid of small K-type thermocouples inserted in glass tubes at the centre of cup cavity. The contact block of cup stands has points which connect thermocouple wires in the cup after fixing the cup. The extension wire made of NiCr and NiAl connects to the positive and negative ends of the thermocouples, respectively, as shown in **Figure 3**. The extension wires are connected to data-logger instrument to store the time temperature history of the solidifying metal as shown in **Figure 3**. The stored data can be retrieved later to generate the cooling curve.

The melt samples of base iron and inoculated molten iron were tested every hour during the day shift in ductile iron production foundry by pouring cups. It is important to note that the use of identical base metal chemistry is required to compare the efficiency of different amounts of inoculants. The plain test cups without tellurium (Te) are used for pouring the test samples. The samples are poured within 5–7 min after inoculation to minimise variations due to fading. In a series of melt processing trials, particle size and method of addition of inoculant were kept constant; the only varying factor was the amount of inoculant.

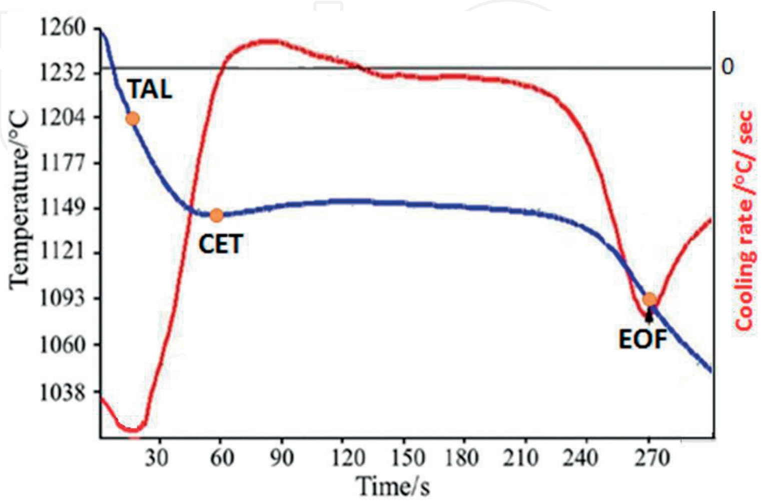

Figure 2. Typical cooling curve and cooling rate in ductile iron [8].

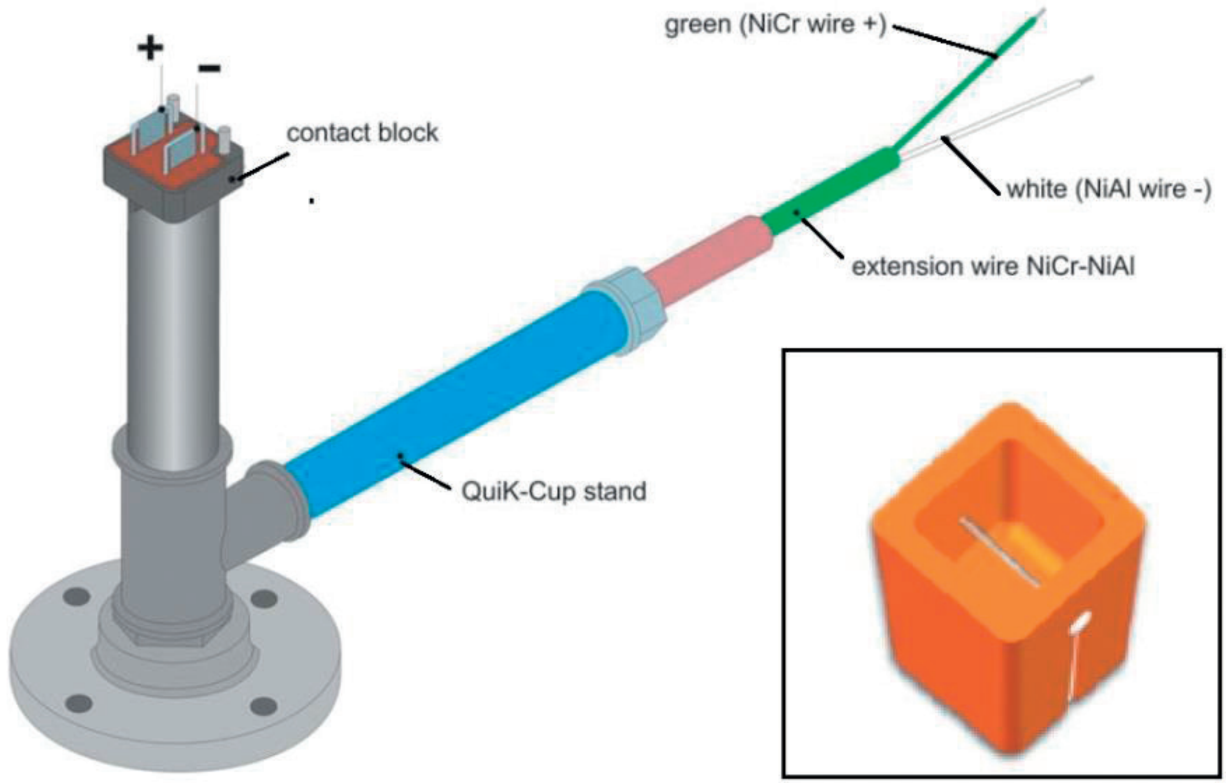

Figure 3. Thermocouple cup holder with extension wires and cup (bottom-right corner).

After testing the inoculated iron, the cooling curves were plotted to analyse the effectiveness of inoculation. The cooling curve analysis includes the liquidus temperature, the eutectic undercooling, the recalescence, the total eutectic freezing zone and the end of freezing. For achieving a smooth and accurate cooling curve, the data is averaged from five consecutive readings. Using this curve, the next data is generated by taking the first derivative. The first derivative is a picture of the actual solidification, and the area it contains is the energy being released by the crystallisation process. The second derivative (if needed) can be used to magnify and detect important events on the overall curve which are indicative at the start or end of a nucleation of a phase in the cooling curve.

The type (inoculant base) and amount of inoculation are varied so as to study its effect on microstructure and properties of ductile iron. By analysing the data generated in the above experiments, the response of these variables on final microstructure of the poured casting can be determined. In the data-logger, the data of the actual temperatures at the tip of thermocouple placed in solidifying metal is stored. The stored data is then retrieved for plotting of time-temperature curve, i.e. cooling curve. The phase transformations during solidification will reflect in the cooling curves and can be identified by calculating a change in the slope by plotting the first derivative of the cooling curve (dT/dt) referred as cooling rate.

The thermal analysis is an effective tool in controlling the melt quality before pouring into actual ductile iron castings. The cooling curve from cup analysis has unique features, affected by melt processing. Five distinct points can be visualised: liquidus temperature (T_{liq}),

http://dx.doi.org/10.5772/intechopen.72030

minimum eutectic temperature (T_{min}), maximum eutectic temperature (T_{max}), recalescence ($\Delta T = T_{max} - T_{min}$) and end of solidification temperature (TES) and solidification time.

The cooling rate curve with increasing trend passes through zero, marked as eutectic minimum (T_{min}), and, after attaining a peak value, follows a decreasing trend and again passes through zero, noted as maximum temperature (T_{max}). The end of solidification is TES, and cooling rate curve angle at solidus is called VPS. The first peak in cooling rate curve is noted as liquidus temperature (T_{liq}). This is a function of the active carbon equivalent, which shows the integrated effect of all elements that influence solidification process. It indicates the effect of elements which are present in melt, for example C, Si and P, and also some other dissolved elements such as oxygen. It gives more information than carbon equivalent (CE) calculated from chemical analysis. To achieve stable properties in the ductile iron, it is essential that the CE value should be maintained consistently. For hypoeutectic composition (CE < 4.33), the amount of primary austenite is directly proportional to the difference in CE values of eutectic (4.33) and actual melt. Hypereutectic (CE > 4.3) iron will solidify with precipitation of primary graphite (kish graphite) which can give surface defects if proper inoculant is not added. Consequently, this technique can also reveal microstructural information that could not be obtained from the standard metallographic techniques. For example, the amount of austenite cannot be easily determined through classic metallographic techniques because of the solid-state transformations that occur.

The thermal analysis of the ductile iron melts at different processing stages was conducted to study the effect of processing on solidification parameters. The cups were poured with base metal, after addition of FeSiMg alloy in the melt and inoculation processing. Cooling curves were plotted as shown in **Figure 4**. It is observed that inoculation processing increases the overall eutectic temperature and minimises undercooling, i.e. the difference between the minimum and the maximum eutectic temperature ($T_{max} - T_{min}$). The inoculated metal shows a wide range of eutectic freezing.

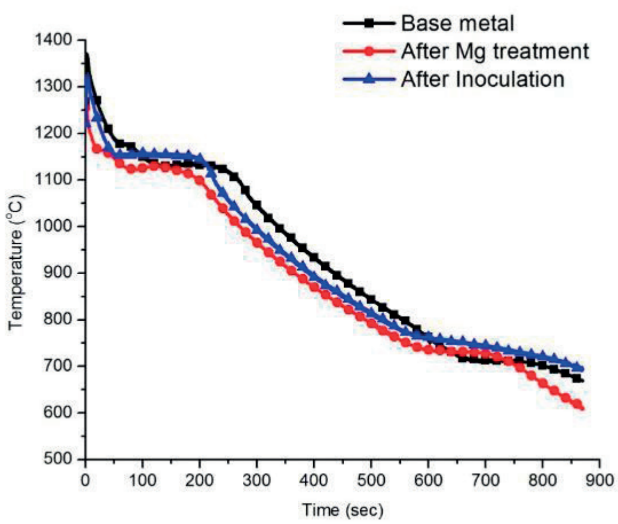

Figure 4. Cooling curves showing effect of melt processing in ductile iron.

Sr. no.	Inoculant type	Si%	Ca%	Ba%	Al%	Zr	Ce%	Fe
1	Ca based	73.34	2.46	–	1.26	1.32	–	Balance
2	Ba based	73.89	1.13	2.84	1.40	–	–	Balance
3	Ce based	72.91	0.96	0.8	0.82	–	1.95	Balance

Table 2. Typical chemical compositions of ductile iron inoculants.

3.2. Effectiveness of inoculation

Ductile iron melts can be inoculated by several methods such as ladle inoculation, in-stream inoculation or mould inoculation. In ladle transfer method, the inoculant is added to the metal as it is transferred from the furnace to the pouring ladle. The turbulence quickly dissolves the inoculant and evenly disperses it throughout the molten bath. For better recovery of the magnesium, inoculation is done in the stream or in the mould. The different nucleation mechanisms are active during the solidification of ductile iron. The solidification behaviour after pouring the metal into the mould can be studied in terms of the heat transfer mechanism in the solidifying metal. The transient temperature data of solidifying metal will be recorded by inserting thermocouples in the casting cavity. These thermal readings are further processed to plot the cooling curves. The analysis of cooling curves will be useful in studying small events occurring during solidification.

For effective and well-controlled inoculation, ferrosilicon of controlled chemical composition is usually used. Active inoculating elements are Ca, Al, Ba, Sr, Zr and Ce; some others are deliberately added in the ferrosilicon alloy. The sample chemical composition of calcium, barium and cerium-based inoculants is shown in **Table 2**. These inoculants were added in varying amounts while transferring the melt from treatment ladle to pouring ladle.

By conducting various trials with different inoculants as mentioned in **Table 2**, the best possible combination could be set for particular foundry processing to determine the optimum addition rate of particular inoculant. The amount of inoculant added could be from 0.05 to 1% of the total weight of liquid metal. With different combinations of melt processing, the cups should be poured to generate the time temperature data for base metal and after inoculation processing. The metal is poured into the cups instrumented with built-in thermocouples. Initially, base metal (without inoculation processing) cup must be poured.

4. Conclusions

The solidification cooling curve, its derivatives, related temperatures and calculated indices are patterns that can be used to assess the melt processing and predict the microstructure and mechanical properties of ductile iron. Inoculation raises both the maximum and minimum eutectic temperatures. The inoculation acts as a deoxidiser, and hence the liquidus temperature of an iron that contains a high amount of oxygen can be reduced by

8–10°C. Both the maximum and minimum eutectic temperatures rises compared to the base metal. The maximum recalescence reduces with an increase in the amount of inoculant. Well-inoculated ductile iron typically shows a recalescence in the range of 2–4°C. Higher recalescence requires more inoculation. Increase in the amount of inoculation decreases the undercooling with an overall increase in the total solidification time. Lower angle of the first derivative at the solidus point indicates high nodule count. It represents the speed with which the cast iron crosses the zone of complete solidification, indirectly measuring the thermal conductivity.

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