Solidification Behavior of Water-Cooled and Subzero-Chilled Cast Iron on Mechanical Properties

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Hypoeutectic cast iron specimens cast using chills that are water-cooled and liquid-nitrogen-cooled (subzero chilling) were compared with specimens of the same chemical composition, which were sand-cast without any chill. The solidification behavior, number of eutectic cells, grain size, and effects of these on the mechanical properties such as strength and fracture toughness were recorded and analyzed in this paper. It is revealed from the above investigation that subzero and water-cooled chilled cast irons exhibit severe under cooling as compared to that of normal sand-cast iron. Thus, it is concluded from the investigation that nucleation conditions are completely altered during solidification, which is considered to be responsible for the variation in eutectic cell size, grain size, microstructure, and hence mechanical properties of the cast iron.

Keywords chill, solidification, subzero, water-cooled

1. Introduction

1.1 Chilled Cast Iron

Chilled cast iron belongs to a group of metals possessing high strength, hardness toughness, and wear resistance. For a metallurgist, there is sufficient information available on the solidification mode and cell size of ordinary cast iron cast in sand molds. However, there is a serious lack of information on the mode of solidification of chilled cast iron and its effect on cell size, grain size, and microstructure and the effects of these on strength and toughness. This prompted the author of this paper to embark upon a series of experiments to study the relationship between these parameters in the case of watercooled and liquid-nitrogen-cooled chilled cast irons. The reason behind the selection of this series of chilled cast iron for the present investigation is that a wide range of strength and fracture toughness values can be obtained with different cell sizes and microstructures.

1.2 Effect of Cooling Rate during Solidification

In general, the cooling rate during casting is largely governed by the design and thermal nature of the casting procedure, one significant factor being the mold material. Metal molds generally offer higher chilling action on the solidifying mass due to their heat diffusivity. Therefore, the influence of higher cooling rate is normally responsible for the superior properties of chilled castings. The influence of very high cooling rates in producing fine structures offers the possibility of future development of cast irons possessing high strength and fracture toughness. The undercooling of a melt to a lower temperature increases the number of effective nuclei for solidification relative to the growth rate, the latter being restricted by the rate at which the latent heat of crystallization can be dissipated. Conversely, slow cooling favors the growth from a few solidification nuclei and produces coarse grain structures. The refining effect of enhanced cooling rate applies both to the primary grain size and to the substructure, although in the latter case, the effect is on the growth process rather than on nucleation. Thus, there is a marked effect upon dendritic cell size, grain size, and microstructure over a wide range of cooling rates and, consequently, on the mechanical properties.

Copper was selected as an important alloying element for the hypoeutectic cast iron in view of its tremendous potential as a grain refiner. Keeping the above discussion in mind, the present research work was planned for the following purposes:

- to obtain experimental data for the eutectic cell count, grain size, and resulting microstructures with various cooling rates;
- to analyze the data in light of the solidification process; and
- to correlate ultimate tensile strength (UTS), fracture toughness, and pearlite composition with eutectic cell size and microstructure.

1.3 Past Research

Several properties of cast iron are of interest to operating foundrymen, including UTS, fracture toughness, and hardness. These properties are influenced mainly by chemical composition and microstructural features, including graphite type, graphite content, and cell count. As most foundry metallurgists know, the composition of the cast iron determines the quantity and character of the graphite, as well as the metallurgical characteristics of the metallic matrix for some specified set of cooling conditions.^[1]

Cooling curves of cast iron have been used quite successfully to explain the solidification behavior of gray cast iron (stable) and white cast iron (unstable), as well as the influence of alloying elements and cooling rate on the solidification of cast irons in general.^[2,3] Significant undercooling reflects an absence

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Table 1 Composition of cast iron tested

Element	С	Si	Mn	S	Р	Cu	Fe
%Composition	3.42	1.8	0.41	0.04	0.08	1.57	Balance

of nuclei for solidification, which can serve as eutectic cell centers.

In a study of the fracture mechanics of gray cast iron, Baker^[4] observed that both the UTS and fracture toughness decreased linearly with increasing eutectic cell size. Suzuki and Kayama^[5] concluded from their own investigations that graphite nucleation out of residual graphite is by means of eutectic cells and that eutectic cell number is an index to nucleation frequency. DeHoff and Rhines^[6] developed an equation to calculate the number of eutectic cells per unit volume, and this can be deduced using the number of eutectic cells. Fedrikson and Hillert^[7] studied the graphite precipitation process in an alloyed cast iron under conditions of controlled solidification at various rates.

Nucleation of the eutectic liquid can be a difficult process since considerable undercooling is often observed before solidification begins. If the stable eutectic is not nucleated, then the iron may undercool and solidify to form iron carbides mixed with austenite. The minimum eutectic temperature can be used as a measure of undercooling and reflects the amount of primary austenite precipitated and the chilling tendency of the cast iron. Austenite dendrite interaction has been shown to be a major factor in determining the UTS of irons.^[8]

The most important fact to recognize regarding cell structure is that it develops after the precipitation of austenite dendrites. Thus, cell growth conforms to and is superimposed on a dendritic structure that solidifies hosts of the cells and therefore can have a major effect on cell nucleation and growth.^[9] Ruff and Wallace^[10] showed that, by increasing the eutectic cell count, the effective span and stress concentration effect of the graphite was reduced and the UTS was improved. A suggested classification of cell size has been proposed by Dawson and Oldfield.^[11]

2. Experimental Procedure

2.1 Fabrication of Material

A cast iron alloy of the composition shown in Table 1 and cast at three different cooling rates was produced by casting at 1440 °C in the form of ingots. They shall henceforth be designated by specimen A (liquid-nitrogen-cooled with copper chill), specimen B (water-cooled with copper chill) and specimen C (sand-cast without any chill). Apart from the usual alloying elements such as Si, Mn, S, and P, copper was also added to improve merchantability as well as to act as a grain refiner.

2.2 Casting Procedure

Figure 1 shows a schematic diagram of the mold used for producing the various ingots cast with liquid-nitrogen-cooled and water-cooled chills (specimens A and B, respectively). (The



Fig. 1 Mold used for casting specimens

same type of mold was used to sand-cast specimen C, in which case no copper chill was used.) To make the mold for casting, a teakwood pattern of size 225 by 150 by 25 mm was employed with standard pattern allowances. The molds were prepared using silica sand with 5% bentonite as a binder and 5% moisture. The molten alloys were cast in the mold and were cooled from one end by the chill. In the case of water-cooled chilling and liquid-nitrogen-cooled chilling, arrangements were made in the copper chill to circulate water (at 23 °C) and liquid nitrogen (at -60 °C), respectively.

2.3 Specimen Testing

Tensile tests were carried out using an Instron testing machine and the tests were performed in conformance with American Foundryman's Society standards. Each result of the test recorded in this paper was an average of at least three repetitions.

Fracture toughness tests were carried out on ASTM standard specimens (with a chevron notch in the middle), all of which were selected along the length of the casting using the Materials Testing System. All tests were done in accordance with ASTM 399-1990 test standards. A three-point bend fracture toughness specimen was precracked by fatigue loading and finally fractured by static loading to find its fracture toughness. Finally, the fractured surface was studied using scanning electron microscopy (SEM).

Microscopic examination was conducted on all the specimens using SEM and a Neophot-21 metallurgical optical microscope. For this, various etchants were tried but 2% Nital proved to be the best and was therefore used.

Grain size measurements were done on polished specimens using ASTM index methods, and eutectic cell count measurements were carried out on polished specimens at low magnification (5×) using Steads reagent as the etchant.

Specimen location	P	earlite content (%	%)
Distance from chill end (mm)	Specimen A	Specimen B	Specimen C
30	28	32	59
70	35	39	58
105	42	60	61
145	57	62	59
180	59	68	60
225	68	71	59

 Table 2
 Pearlite content of various specimens along the length of the casting

 Table 3
 Grain size of various specimens along the length of the casting

Specimen location	Number of grains/in. ² at 100× magnification			
Distance from chill end (mm)	Specimen A	Specimen B	Specimen C	
30	128	128	32	
70	64	64	32	
105	64	32	32	
45	32	32	32	
180	32	32	32	
225	64	64	32	

Table 4Eutectic cell count of various specimens along
the length of the casting

Specimen location	Number of eutectic cells/cm ²			
Distance from chill end (mm)	Specimen A	Specimen B	Specimen C	
30	185	165	75	
70	175	150	75	
105	105	105	75	
145	105	95	75	
180	95	95	75	
225	70	70	75	

3. Results and Discussion

Table 2 to 6 show the pearlite content, the grain size, the eutectic cell count, the UTS, and the fracture toughness, respectively, of the three specimens along the length of the casting.

3.1 Eutectic Cell Count

Solidification of hypereutectic cast iron begins with the crystallization of primary graphite. The primary graphite develops as straight graphite plates with some branching growing while totally surrounded by liquid. The composition of the remaining liquid shifts toward the eutectic where the liquid is believed to solidify in a manner similar to that of a eutectic cell, although the primary graphite may influence the size of the eutectic cell and the distribution of the eutectic graphite.

Table 5UTS various specimens along the length ofthe casting

Specimen location		UTS (MPa)	
Distance from chill end (mm)	Specimen A	Specimen B	Specimen C
30	488.67	467.33	282.55
70	381.44	328.34	282.43
105	372.86	361.43	287.53
145	272.43	279.31	281.54
180	268.32	282.67	279.75
225	273.64	281.54	282.65

 Table 6
 Fracture toughness of various specimens along the length of the casting

Specimen location	Fracture toughness (MPa \sqrt{m})			
Distance from chill end (mm)	Specimen A	Specimen B	Specimen C	
30	33.9	32.1	22.0	
70	31.2	28.4	21.3	
105	29.1	26.8	22.5	
145	28.7	24.1	20.8	
180	24.7	23.8	22.4	
225	23.6	23.0	22.4	

Since it is generally accepted that the eutectic cell count is an indication of the number of nuclei on which solidification has taken place, a special experiment was designed in order to compare the eutectic cell counts of water-cooled and liquidnitrogen-cooled cast iron with ordinary sand-cast iron. Pearlite content, UTS, and fracture toughness were plotted against eutectic cell count for various specimens along the length of the casting, as in Fig. 2 to 4, respectively. Some typical photomicrographs of eutectic cells for sand-cast, water-cooled, and liquidnitrogen-cooled chilled cast iron are shown in Fig. 5 to 10. It can be seen from Fig. 3 and 4 that the UTS and fracture toughness increase as the eutectic cell count increases until the cells become compacted (as shown in Fig. 5 to 10). It can be seen that the difference between the eutectic cell counts for water-cooled and liquid-nitrogen-cooled chilled cast iron is small, whereas that between the eutectic cell counts for chilled cast iron and sand-cast iron is very large. Typical values are 185 cells/cm² for liquid-nitrogen-cooled (30 mm from chill end), 165 cells/cm² for water-cooled (30 mm form chill end), and 75 cells/cm² for sand-cast iron without chill.

It follows that the number of nuclei available for solidification of liquid-nitrogen-cooled chilled cast iron is slightly higher than the number of nuclei available for solidification of watercooled chilled cast iron. However, there are significantly fewer nuclei for solidification in the case of sand-cast iron without a chill. This is due to the undercooling effect caused by chilling. Perhaps the most important fact to recognize regarding cell structure is that it develops after the precipitation of austenite dendrite structure, which solidifies first. The dendritic structure hosts the cells and therefore can have a major effect on cell nucleation and growth.



Fig. 2 Plot of pearlite content vs eutectic cell count for various specimens along the length of casting

3.2 Rate of Cooling

The differences in associated graphite, its randomness, length (as shown in Fig. 11 to 13), and matrix structure (as shown in Fig. 14 to 16), for the various specimens cast were analyzed and correlated to the temperature of eutectic formation. Faster cooling produces fine, highly oriented dendrites, while slow cooling produces large, coarse dendrites. Solidification over a temperature range is the primary requirement for dendrite growth. Primary austenite dendrites readily grow from the liquidus down to the eutectic temperature. Growth of dendrites may also continue concurrently with the eutectic as the temperature decreases through the eutectic range to the solidus. Thus, undercooling in the case of water-cooled and liquid-nitrogencooled chilled cast iron may lead to longer dendrites and higher interaction. Hence, the eutectic cells solidify around these austenite dendrites, and in this manner the entire microstructure is affected by the number and size of the dendrites.

Therefore, the general picture of solidification of chilled



Fig. 3 Plot of UTS vs eutectic cell count for various specimens along the length of casting

cast iron (water-cooled and liquid-nitrogen-cooled) with various eutectic cells, graphite shapes, and matrix structures (as shown in Fig. 5 to 16) can be summarized as follows.

The solidification of flake graphite iron resulting from sand casting without a chill starts from a small number of nuclei as compared with chilled (water-cooled and liquid-nitrogencooled) cast iron, a phenomenon indicated by the eutectic cell count. Since the growth conditions in the liquid are favorable, these nuclei start growing as soon as the temperature is below the equilibrium temperature in the case of sand-cast iron and at a maximum under cooling in the case of chilled cast iron, whether water-cooled or liquid-nitrogen-cooled.

3.3 Pearlite Content

Also, if the results shown in Table 2 to 4 are compared with one another, it can be seen that there exists a straightforward



Fig. 4 Plot of fracture toughness vs eutectic cell count for various specimens along the length of casting



Fig. 5 Eutectic cell structure of sand-cast specimen, etched with Steads reagent (75 cells/cm² at $5 \times$ magnification)

relationship between pearlite content and eutectic cell count taken along the length of the casting for the various specimens cast. This relationship is plotted in Fig. 2. Comparing Table 2 and 6, it can be deduced also that fracture toughness decreases with an increase in pearlite content and *vice versa*. This too agrees with Baker's findings.^[14] It is noteworthy that specimens A and B contain cementite in pearlite matrix, whereas specimen C contains ferrite in pearlite matrix (Fig. 14 to 16).



Fig. 6 Eutectic cell structure of water-cooled chilled specimen 30 mm from the chill end, etched with Steads reagent (165 cells/cm² at $5 \times$ magnification)



Fig. 7 Eutectic cell structure of water-cooled chilled specimen 225 mm from the chill end, etched with Steads reagent (70 cells/cm/² at $5 \times$ magnification)

3.4 Dendrite Formation

Results of the investigation indicate that the minimum eutectic temperature can be used as a measure of undercooling and reflects the number of cells precipitated and the chilling tendency of the iron. The effects of eutectic cell count on UTS and fracture toughness of the specimens tested in this investigation are shown in Fig. 3 and 4. Higher dendritic interaction areas in the case of chilled cast irons reflect the interweaving of dendrite through eutectic cells that effectively tie the eutectic cells together. Since dendrites are formed from primary austenite, they have a higher tensile strength than eutectic carbon. Here, due to chilling, dendrites directly form austenite and do not contain flake carbon. Instead they contain carbides (cementite) in the pearlite matrix, as shown in Fig. 15 and 16. Baker^[4] in his investigation on fracture mechanics of gray cast iron observed that both tensile strength and fracture toughness



Fig. 8 Eutectic cell structure of liquid-nitrogen-cooled chilled specimen 30 mm from the chill end, etched with Steads reagent (185 cells/ cm^2 at 5× magnification)



Fig. 10 Eutectic cell structure of liquid-nitrogen-cooled chilled specimen 225 mm from the chill end, etched with Steads reagent (70 cells/ $cm/^2$ at 5× magnification)



Fig. 9 Eutectic cell structure of liquid-nitrogen-cooled chilled specimen 105 mm from the chill end, etched with Steads reagent (105 cells/ cm^2 at 5× magnification)

decreased linearly with increasing eutectic cell size (smaller number of cells/cm²), and this agrees well with the results of the present investigation, which indicate that UTS and fracture toughness increase as the cell size decreases (larger number of cells/cm²).

3.5 Grain Size

Table 3 shows the results of microscopic studies on grain size. It can be seen that the grains in the case of chilled cast iron were fine but the grains were very coarse in the case of sand-cast iron cast without a chill. The fine grain size in the case of chilled cast iron results in the soundness of the casting and hence its high strength and toughness. In water-cooled and liquid-nitrogen-cooled chilled cast iron, the experimental data show that dendrite morphology is the principal factor in



Fig. 11 Microstructure of sand-cast specimen 30 mm from the chill end, unetched (type A graphite, $250 \times$ magnification)

determining the strength and toughness but not the graphite morphology, an observation supported by Read Hill.^[12]

3.6 Mode of Fracture

As expected, the SEM photographs taken (Fig. 17 to 19) of the fracture surfaces of specimens revealed a brittle mode of fracture near the chill end. Looking at Table 5 and 6, it can be seen that the parameters causing more significant effects on the mechanical properties are the rate of chilling and the specimen location. It is observed that, if all other factors are kept constant, specimen C has the lowest UTS and fracture toughness, followed by specimens B and A in that order. This means that an increase in the rate of chilling results in an increase in UTS and fracture toughness of the material.

Moreover, the further away form the chill the specimen is taken, the lower the UTS and fracture toughness.^[13] This is obviously because the further away form the chill the specimen



Fig. 12 Microstructure of water-cooled chilled specimen 30 mm from the chill end, unetched (type D graphite, $250 \times$ magnification)



Fig. 15 Microstructure of water-cooled chilled cast iron specimen 30 mm from the chill end, etched with Nital (cementite in pearlite, $500 \times$ magnification)



Fig. 13 Microstructure of liquid-nitrogen-cooled chilled cast iron specimen 30 mm from the chill end, unetched (type D graphite, $250 \times$ magnification)



Fig. 16 Microstructure of liquid-nitrogen-cooled chilled cast iron specimen 30 mm from the chill end, etched with Nital (massive cementite in pearlite plus some ledeburite, $500 \times$ magnification)



Fig. 14 Microstructure of sand-cast specimen 30 mm from the chill end, etched with Nital (ferrite in pearlite, $500 \times$ magnification)



Fig. 17 SEM fractograph of sand-cast fracture toughness test specimen 30 mm from the chill end, indicating cleavage fracture of graphite flakes



Fig. 18 SEM fractograph of water-cooled fracture toughness test specimen 30 mm from the chill end, indicating dimpled rupture of graphite flakes



Fig. 19 SEM fractograph of liquid-nitrogen-cooled fracture toughness test specimen 30 mm from the chill end, indicating dimpled rupture of graphite flakes

is, the lower is the rate of chilling. This agrees with the deductions made earlier that increasing the rate of undercooling due to chilling tends to result in an increase in UTS and fracture toughness of the material.

4. Conclusions

Experiments have been performed using the number of eutectic cells as an index of graphite nucleation and the effect of these on structure and properties, since these cells exist in molten cast iron to act as nuclei of graphite during solidification. The following conclusions were made.

- The number of eutectic cells is significantly larger in the case of chilled cast iron than in the case of sand-cast iron cast without a chill.
- It has been established that the number of eutectic cells affects both the structure and properties of the material. The eutectic cell count was shown to be a major factor that affects UTS and fracture toughness. In the cast iron studied, the larger the number of eutectic cells, the greater will be the UTS and fracture toughness.
- Data on irons chilled using water-cooled and liquid-nitrogen-cooled chills indicated that the cooling rate has a marked effect on eutectic cell size, grain size, microstructure, and mechanical properties.

A straightforward relationship exists between pearlite content and the eutectic cell count.

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