NAKAYAMA
Co.,Ltd.

NEW CE-Meter

A tool for characterization of cast iron melt

TEC-21

A "must" for thin-walled cast iron, complicated shapes, avoiding chilled layers, and high function materials.

Functions of TEC-21

Newly added functions

- **a** Estimation of nodularity(SG%)
- **b** chill
- **c** ASTM Flake graphite size
- **d** inoculant selection
- **e** shrink

All the values of a to e can be obtained within 3 to 4 minutes after sample pouring.

Purpose of development of TEC-21

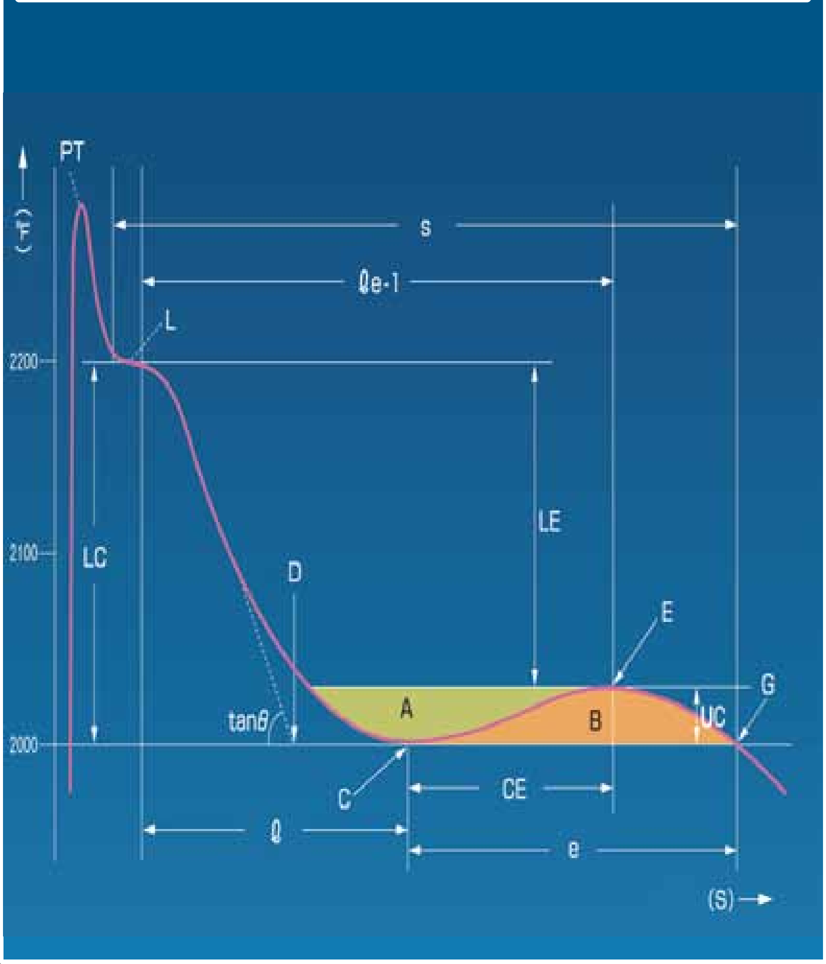
Quality control on the melting floor

Conventional CE-meters only determine chemical compositions and are unable to respond to advanced level of such technical requirements as thin-walled castings, complicated shapes, necessity of avoiding hard layers, or materials for high functions. This equipment was developed as a quality control tool on the melting floor that will greatly help production of castings for high technical requirements.

thin-walled castings	avoiding hard layer	»	better quality control
complicated shapes	high functions		

cooling curve for melt quality characterization in relation to phase diagram

a cooling curve and characteristic values



17characteristic values in a cooling curve	
1) primary temperature (liquidus)	L
2) eutectic solidification temperature (eutectic point)	E
3) primary solidification range	LC
4) solidification range	LE
5) primary solidification time	ℓ
6) eutectic solidification time	e
7) total solidification time	s
8) primary solidification temperature gradient	tan θ
9) primary solidification time ratio	ℓ /e
10) undercooling	UC=E-C
11) undercooling point	C
12) undercooling area	A
13) eutectic solidification area	B
14) pouring temperature	PT
15) time from primary to eutectic point	ℓ e-1
16) time from undercooling point to eutectic point	CE
17) time from eutectic point to solidification end	EG

Melt Quality Assessment Function

1) Reading off CE value (L)

When molten metal is cooled and solidified, there is always a solidification point or starting temperature of solidification. Solidification temperature of a cast iron melt is dependent on its chemical composition. From starting temperature of solidification, CE value (Carbon Equivalent) of the melt can be determined. There are several different definition of CE value: C+(1/3)Si, C+(1/3)Si+(1/3)P, C+(1/4)Si+(1/2)P, or C+0.3Si+0.3P. By examining quality control charts fitting to the conditions of your plant, you may be able to find out if you can use the CE value table of your CE-meter catalogue without correction, or you need to make some corrections to it.

Generally speaking, the amount of necessary correction is not too large.

2) Reading off eutectic point (E)

A cast iron melt has its solidification end point or eutectic solidification temperature as well as solidification start point. Among the two major alloying elements of cast iron melt C and Si, the latter has a large influence on change of eutectic temperature, where eutectic temperature is raised by increased Si content. Thus Si % can be estimated from the eutectic temperature.

Knowledge of eutectic temperature can be utilized for other purposes such as estimation of chill depth or shrinkage. Thus it can be said that eutectic temperature and primary solidification temperature are the two most important features of a cooling curve.

3) Reading off solidification temperature range (LE)

The temperature difference between the primary and eutectic solidification is called solidification temperature range LE. From this LE value of the melt, C% of the melt can be determined and mechanical properties of the castings from the melt such as strength or hardness can be estimated. ....This applies to irons without Tellurium.

4) Reading off undercooling(UC)

The phenomena of undercooling are observed more or less at primary and eutectic solidification. Undercooling appears when the balance between cooling rate of the melt and growth velocity of iron crystal is broken. Degree of undercooling (UC) has a close relation with the tendency of chill formation.

Generally speaking, a large undercooling is accompanied by deep chilling and severe segregation. Growth rate of graphite in cast iron is said to be proportional to square of degree of undercooling. Therefore, a small undercooling causes slow growth, and hence, thick graphite with large layer distances. A large undercooling enhances growth of primary dendritic austenite and reduces amount of eutectic cell formation in the interdendritic area.

A cup without Tellurium is used for the melt quality assessment.....Chill cup

5) Reading off time of passing the solidification range (time ratio of ℓ and e)

Cast iron characteristics are also estimated by examining the time from the primary solidification through undercooling and to eutectic solidification. Consider the ratio between the time from primary to undercooling (primary solidification time ℓ) and the time from undercooling to eutectic solidification (eutectic solidification time e). Melt quality can be largely different between those with a large ratio and those with a small ratio, even when the total time is the same.

Generally speaking, melts with a short primary solidification time and long eutectic solidification time are favored.

6) Reading off time from eutectic point to solidification end (inoculation effect)(EG)

A long time from the start of eutectic to the end of solidification (the range EG) indicates a good inoculation effect. .....graphite shape, eutectic cell number, graphite distribution.

7) Effects of soluble impurity in melt on cooling curves

Effect of 1% alloying element on solidification temperature of hypo-eutectic cast iron.(Results of three tests were averaged.)

alloying element	change in primary liquidus temperature, °F	Change in maximum eutectic temperature ,°F
Cr (Chromium)	-5	-10
Mo (Molybdenum)	-2	-18
NI	-3	+6
Cu	-5	+5
Mn	-5	-15

8) Difference between a cupola melt and electric furnace melt with the same CE value Differences:

1) Cupola melt exhibits less undercooling.
2) Cupola melt has shorterℓ and longer e.
3) Cupola melt exhibits better inoculation effect (Si%).
4) Cupola melt is better in chill tendency and shrinkage tendency.

Ⅰ, *Estimation of nodularity (SG%)*

functions

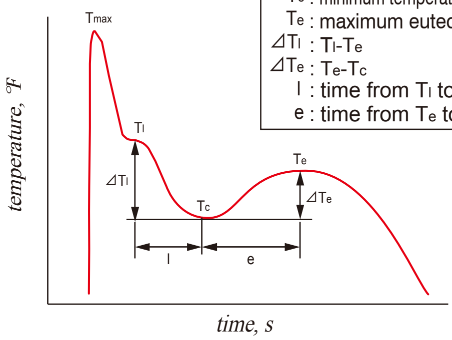
Once the melt is sampled on the melting floor, data collection for thermal analysis, calculations, and nodularity determination are performed automatically without human intervention

Aim of development

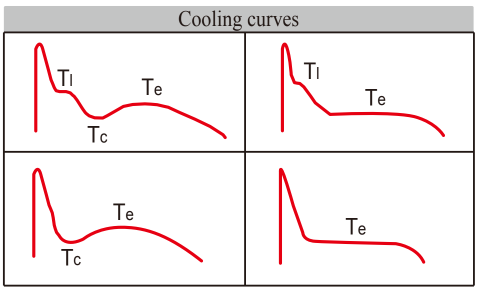
1. Nodularity is determined within 3 minutes after melt treatment.
2. Nodularity determination is performed automatically by the instrument avoiding human power and personal errors.

1. A schematic cooling curve and characteristic points

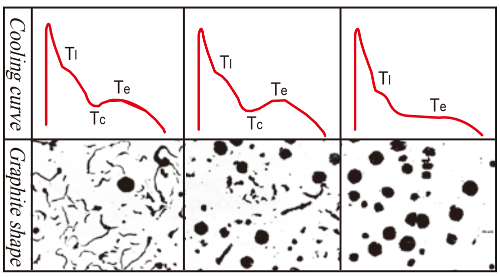
- T_{max} : maximum attainable temperature
- T_l : primary temperature
- T_c : minimum temperature at eutectic undercooling
- T_e : maximum eutectic temperature
- ΔT_l : T_l-T_e
- ΔT_e : T_e-T_c
- l : time from T_l to T_e
- e : time from T_e to T_c



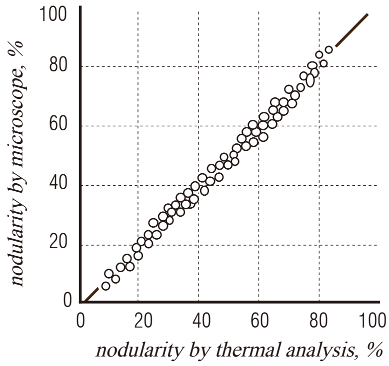
2. Types of cooling curve



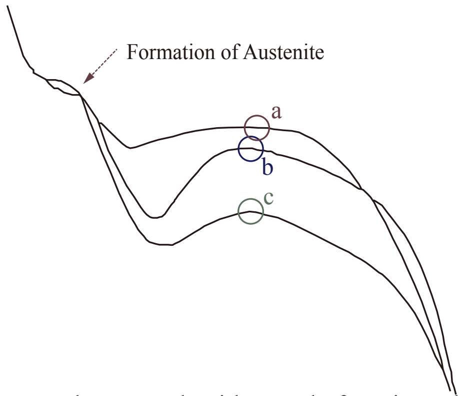
3. Relation between cooling curve and graphite shape



4. Correlation between nodularity as determined by microscope and that estimated from thermal analysis



5. Schematic cooling curves for gray, Ductile and vermicular graphite irons.



a. Flake	1154°C	2109°F
b. Vermicular	1150°C	2102°F
c. FCD	1137°C	2079°F

In melts treated with Mg before inoculation, if the eutectic solidification temperature is found above 2102 degree F, graphite spheroidization is almost always unsuccessful, whereas if the eutectic solidification temperature is between 2066 and 2075 degree F, spheroidization is successful.

※Test results・・・・・Results of more than 200 tests indicated that melt treatment with SiC as the Si source contributes to the improved nodularity and ease of spheroidization treatment in terms of treatment temperature, Mg yield, effects of spheroidizers and inoculants.

A calibration curve for nodularity is necessary for each material. Proprietary curves should be prepared at each foundry.

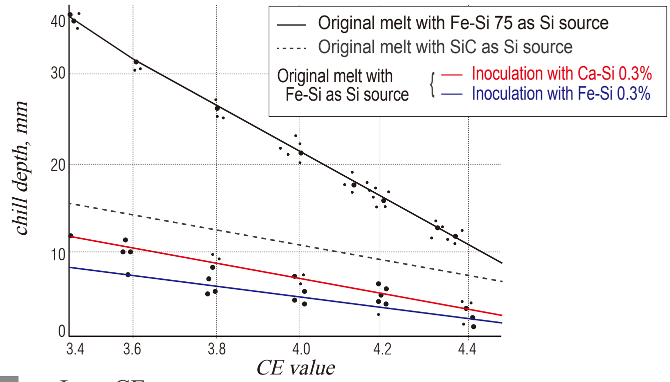
Experimental method

In both FCD and CV irons, melts after spheroidization treatment were sampled with a ceramic fiber spoon and poured in the specified mold for cylindrical bar of 20 mm in diameter and a shell cup.

Then cooling curve was recorded and nodularity was measured by the automatic image analysis. The cooling curves and nodularity thus obtained were correlated. Experiments were repeated until the following degree of correlation was confirmed: above 75% at the first step, above 85% at the second step, and above 95% at the third step. Finally the precision of ±5% was established in the estimation of nodularity by the cooling curve method.

Ⅱ, *Chill*

(a) CE value (carbon equivalent) and chill depth



Causes of chill

a. Low CE

b. Large undercooling in the cooling curve

c. Large l/e ratio in the cooling curve (The time from undercooling to solidification end (G point) is less than the time from primary point to undercooling point)

d. High oxygen content

Countermeasure

See the graph above

(By the use of SiC as the Si source for the original melt, melt treatment becomes easier; less amount of necessary inoculant, and longer effective time.)

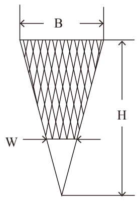
Sampling time:

The original melt, 5 minutes, 10 minutes, 15 minutes after inoculation

Dimensions of wedge test pieces (mm)

No.	B	H	length
W1	5.0	25.0	100.0
W2	10.0	30.0	100.0
W3	20.0	37.5	100.0
W4	25.0	45.0	125.0
W5	30.0	50.0	150.0

Wedge test piece pattern



Chill depth is measured to the unit of 0.1mm.

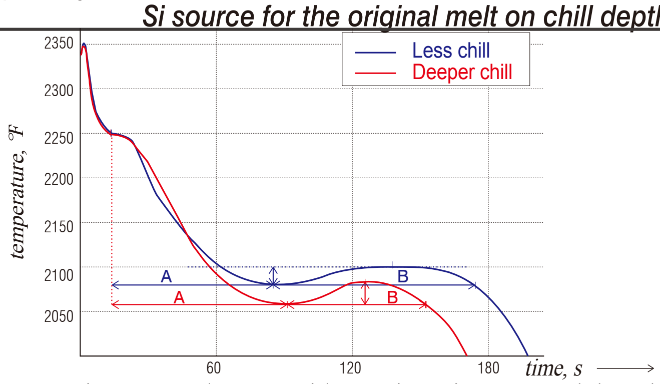
Wedge-type chill test

Different test methods have been known: Japan Gakushin method, Meehanite method, or ASTM method. Currently the ASTM method seems to be almost universally used. Its pattern shape is shown above.

Dimensions of wedge test pieces (mm)

No.	B	H	length
W1	6	25	100
W2	12	32	100
W3	20	38	100
W4	32	50	150
error allowance	0.8	0.8	3.2

(b) Comparison of effects of Fe-Si and SiC as Si source for the original melt on chill depth.



Comparing two melts, one with Fe-Si as Si source and the other with SiC as Si source, the latter exhibits much less chill depth, as measured by difference curve in cooling and by microstructural observation.

Example

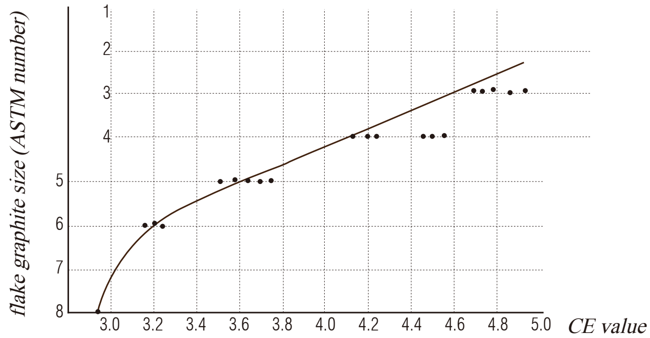
1) The curve with a longer vertical arrow corresponds to deeper chill.

2) The curve with a longer horizontal arrow B than A corresponds to less chill depth, assuming A+B is constant.

Ⅲ, *ASTM Flake graphite size*

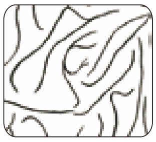
(a) Carbon equivalent and flake graphite size

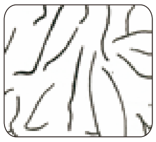
Carbon equivalent and flake graphite size at the center of a 30 mm diameter bar.

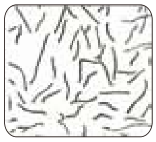


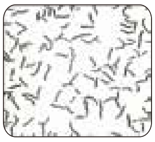
(b) Flake graphite size (mm) and ASTM number (microscope magnification 100×)

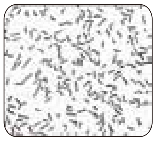

Length: 101.6 mm or longer

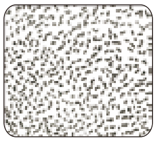

Length: 101.6 ~ 50.8 mm

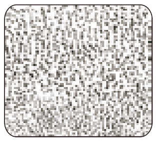

Length: 50.8 ~ 25.4 mm
CE value: 4.60±0.1


Length: 25.4 ~ 12.7 mm
CE value: 4.10±0.1


Length: 12.7 ~ 6.3 mm
CE value: 3.60±0.1


Length: 6.3 ~ 3.2 mm
CE value: 3.20±0.05


Length: 3.2 ~ 1.6 mm
CE value: 3.0±0.05


Length: 1.6 mm or smaller
CE value: 0.95±0.02

Causes of NG

a. The width of undercooling is large. (The less the undercooling and the longer the eutectic solidification time, the more A-type graphite is observed.)

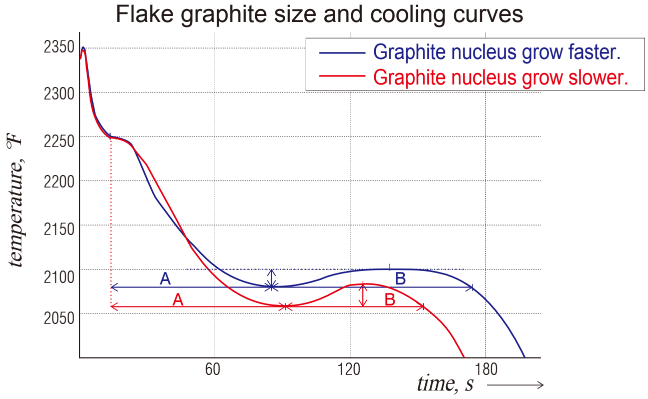
b. Oxygen too much.

c. S% too low (0.09 to 0.095% is better desirable).

d. Inappropriate choice of inoculant (check if melt treatment is appropriate).

(c) Adjustment of CE value

Flake graphite size and cooling curves



Growth rate of graphite is said to be proportional to the square of undercooling. Therefore, when undercooling is small, graphite growth rate is small, and graphite layers develop with larger mutual distances. When undercooling is large, primary precipitation of dendritic austenite is likely to occur, and hence, eutectic cell formation in the spacing between dendrite is limited. Larger inoculation effect and less fading are expected by using SiC as Si source than using Fe-Si.

Example

1) Flake graphite size becomes smaller at lower CE value.
2) Growth of flake graphite nucleus is illustrated in the graph in a separate sheet.
3) More graphite is observed with shorter vertical arrow.
4) Faster growth of graphite nucleus is observed when the horizontal arrow B is longer than the arrow A.

Ⅳ, *Choice of inoculant in relation to cooling curve*

(a) Effect of Fe-Si and SiC as Si source on melt quality (cooling curve).

Effects of different inoculants in melt whose Si and C adjustment has been made with Fe-Si or SiC.

1. Effect of S% in the melt on inoculation effect.
2. Comparison of inoculation effect in melts whose Si addition has been made with SiC or Fe-Si.
3. Comparison of inoculation effects in melts of different conditions.

		Si addition by Fe-Si	Si addition by SiC
Melting furnace Tapping temperature; 1525±10℃ Weight of one tap; 250kg Amount of inoculant; 0.3%		500kg high frequency induction furnace	same as the left column
Melting stock		Steel plate 50% Automobile steel plate with Cr, Cu 50%	same as the left column
additives for composition adjustment	Si addition	・Fe-Si (75%Si,0.43%Al, 0.06%Cu)	・SiC for Si addition(66%Si,22%C)
	C addition	artificial graphite(96 to 97%C, 0.26%S)	・same as the left column
	others	・Fe-Mn ・iron sulfide (50%Fe, 50%Fe)	・Fe-Mn ・same as the left column
inoculant used		see table below	
sampling		1) see the separate sheet 2 for the target composition 2) samples i. tensile test bar (two) ii. chill depth (W-2, W-3, W-4, W-5) iii. hardness test piece from tensile bar iv. microstructure test piece from tensile bar	
1. Chill suppression by inoculant		i. No inoculant was effective for melt of S content 0.032% to 0.040%. ii. For S 0.032%, some effect was observed within 5 minutes after inoculation. The order of effectiveness was No.2, the best, No.4, the next, and No.3, the last. Effects were lost with time passed 10minutes and 15 minutes. iii. For S 0.032%, No.1 inoculant had no effect. iv. For 0.089%S, No.1 and 2 had chill suppressing effect within 5 minutes, where the effect was faded at 10 minutes after inoculation. v. For 0.089%S, No.3 had the best chill suppressing effect.	i. For 0.032%S melt, no inoculant was effective. ii. For melt treated with SiC, No.1 was effective, whereas for melt treated with Fe-Si it was not effective. iii. With inoculant No.2 containing Ba and Mn, chill depth was 0mm in 8mm thickness after 5 minutes. Chill depth was 0mm in 9.6mm thickness after 10 and 15 minutes. No.11 inoculant had the same effect in 5 minutes (0mm chill in 11.2mm thick.), and the same at 10 minutes and 15 minutes. iv. No.3 and 4 exhibited obviously inferior effectiveness. v. For 0.096%S, all the inoculants were effective. vi. By using inoculant No.1, chill depth of 14mm without inoculant was changed to 0mm. After 5 minutes, still 0mm chill depth was observed in 6.4mm thickness. vii. No.2 has chill suppressing effect, though it decreases after 15 minutes. viii. No.3 has the most powerful chill suppressing effect, though its deterioration is fast.
		From the results; 1) Inoculant No.1 containing Sr. * This has the most powerful chill suppressing effect in melts of high S% (0.09%) which was treated with SiC for Si addition. Its effect is lost in other conditions. 2) Inoculant No.2 containing Ba and Mn. * This exhibits a stronger chill suppressing effect in melts treated with 88%SiC than in those treated with Fe-75%Si. 3) Inoculant No.3 with 0.97%Ca and 75%Fe-Si. * This has a chill suppressing effect in melts treated with SiC, though the effect is faded faster than No.1 with Sr.	

Inoculant used:

inoculant / grain size	Si%	Aℓ%	Ca%	Ba%	Sr%	Mn%	Ti%
1 (1-5mm)	48.0%	0.3%	0.06%	—	0.72%	—	—
2 (1-5mm)	63.0%	1.00%	1.60%	6.00%	—	9.90%	—
3 (1-5mm)	75.0%	—	1.00%	—	—	—	—
4 (1-5mm)	52.0%	—	5.00%	—	—	—	9.09%

	Si addition by Fe-Si	Si addition by SiC
2.Effect of inoculation on tensile strength	i. Tensile strength decreases with increasing S%. ii. With inoculant No.1 and with 0.023%S and 0.032%S melt, tensile strength in the original melt decreases within 10 minutes after inoculation. Similar decrease is observed with 0.089%S at 15 minutes after inoculation. Inoculation No.4 exhibits a similar tendency. iii. With No.3 inoculant containing a small amount of Ca, tensile strength increases drastically.	i. Like the melt treated with Fe-Si, tensile strength increases only a little by inoculation at low S%. Tensile strength is decreased below that of the original melt at 10 minutes after inoculation of No.1 and 4 inoculants. ii. In the melt of 0.044%S, tensile strength decreases greatly with time by all the inoculants. iii. In the melt of 0.096%S, strength decrease after inoculation is very small. It is smaller than in the melt treated with Fe-Si.
	From the results; 1) At low S%, little increase in tensile strength with inoculation is observed in both melts treated with Fe-Si and SiC. 2) At high S%, strength decrease after inoculation is much smaller in the SiC treated melt than in the Fe-Si treated melt.	
3.Brinnel hardness	No effect of inoculation was confirmed. Hardness decreased with time after inoculation in all the specimens.	
4. Microstructure and eutectic cell	i. Graphite shape changes from A-type to D or E-type and graphite size decreases with increasing S% or increasing time. ii. Ferrite % in the matrix is much larger with inoculant No.3 and 4 than with other inoculants. iii. Eutectic cell increases with increasing inoculation effect, though it is decreased by inoculant No.1 containing Sr when the original melt contains Ca. iv. In low S% melts, eutectic cell number reaches its maximum by inoculant No.3. Cell number decreases with increasing S% or time.	i.Fe3C decreases by increasing S to 0.095%. ii. Eutectic cell umber in inoculated melts decreases with increasing S%. iii. Inoculant No.4 containing 9%Ti and 5%Ca has a strong ferritizing effect in low S% melts. iv. Inoculant No.3 with 75%Si and 1%Ca has a strong ferritizing effect in high S% melts.

Conclusions

1. Effect of inoculants on chill suppression depends much on S% of the melt.
2. When SiC is used as a Si source and S content of the melt is 0.096%, chill is effectively suppressed by inoculation.
3. If S% of the original melt is raised higher, tensile strength is decreased.
4. Decrease of tensile strength by fading of inoculation effect is prominent in melts with Fe-Si as Si source and is least observed in melts with SiC as Si source.
5. When using inoculant No.1 containing Sr in melts containing Ca, quick pouring after inoculation is necessary, because fading of inoculation effect is extremely fast.
6. No.2 (5% Ba , 10% Mn) is a well-balanced inoculant and is effective in either melt with Fe-Si or SiC as Si source even at low S or high S.
7. No.3 inoculant (1.0% Ca, 75% Fe-Si) gave good results at medium to high S%.
8. No.4 inoculant (5.0%Ca, 9%Ti) has a strong chill suppression effect in low S% melts.
9. In melts with SiC as Si source, effective time after inoculation is much longer and decrease in tensile strength is smaller than in melts with Fe-Si as Si source.
10. Larger inoculation effect and less fading are obtained by using SiC as the Si source to the melt than by using Fe-Si.

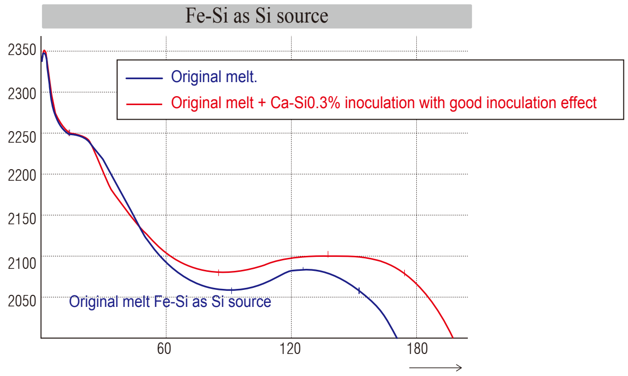
Causes

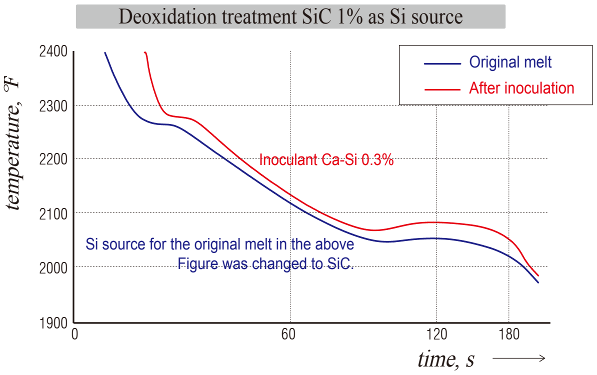
a. Enhanced chilling is observed when undercooling in the cooling curve is large.

b. In melts with long time interval between point E and G, better graphite growth is observed.

c. Favorable melt quality is expected when ℓ is short and e is long, assuming ℓ+ e is constant.

(b) Choice of inoculant and cooling curve with effect of minor elements (S, O_2).





Example

If a cooling curve like the blue one is obtained, inoculation effect is confirmed.

Behavior of S% with regard to cooling curve is similar to that of O_2 (undercooling is increased).

(c) Comparison of Fe-Si 75% and SiC as Si source Microstructure and eutectic cells

1. Effect of inoculants on chill suppression depends much on S% of the melt.
2. When SiC is used as a Si source and S content of the melt is 0.095%, chill is effectively suppressed by inoculation.
3. If S% of the original melt is raised higher, tensile strength is decreased.
4. Decrease of tensile strength by fading of inoculation effect is prominent in melts with Fe-Si as Si source and is least observed in melts with SiC as Si source.
5. No.2 (5% Ba , 10% Mn) is a well-balanced inoculant and is effective in either melt with Fe-Si or SiC as Si source even at low S or high S.
6. No.3 inoculant (1.0% Ca, 75% Fe-Si) gave good results at medium to high S%.
7. No.4 inoculant (5.0%Ca, 9%Ti) has a strong chill suppression effect in low S% melts.
8. In melts with SiC as Si source, effective time after inoculation is much longer and decrease in tensile strength is smaller than in melts with Fe-Si as Si source.
9. When carbon addition is made together with SiC, yield of carbon, and hence, economy are better than when carbon is added together with Fe-Si.
10. Excessive inoculation should be avoided.; Excessive inoculants may produce too many eutectic cells as never seen before and lead to large shrinkage formation caused by external mold wall movement.

(d)Countermeasures against NG, or unsatisfactory results.

As a reference in choosing an inoculant, on-floor study was made using a high frequency furnace on the influence of various factors on inoculation effects. Inoculation effect was evaluated by observing 1) suppression of white iron formation, 2) tensile strength, 3) increase of eutectic cell number, and 4) improvement of graphite shape. Factors studied were:

1) Melt S% affecting inoculation effect (S%=0.05 and 0.09).
2) SiC and Fe-Si as Si source for the melt as affecting inoculation effects.
3) Comparison of inoculation effects and fading of commercially available inoculants: Si-type, Ba-Mn type, Ca type, and Ti-Ca type.

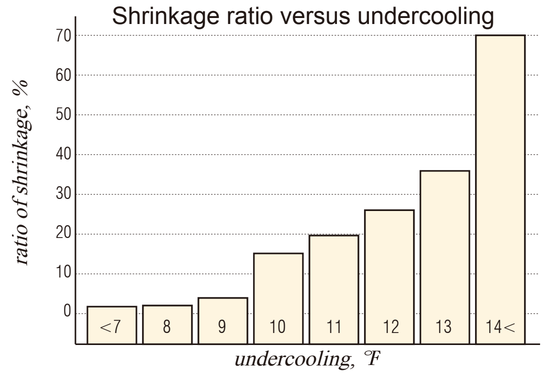
a. When melt S% is low, white iron suppression by inoculation can not be expected, and hence, at least 0.09%S is desired. At a high S%, for instance 0.096% instead of S% as low as 0.035 to 0.045%, mechanical properties and microstructures are vastly improved by inoculation, and chill formation is suppressed. Therefore, appropriate amount of S should be added to melts produced by electric furnace.

b. White iron suppression effect is very large when using SiC as Si source.

c. Effects of inoculants depend much on the melting conditions. For instance, Si-type inoculants are not effective in melts of low S or melts containing Ca. Otherwise, they have the most effective white iron suppression power. Regarding tensile strength, Ca-type inoculants are most powerful.

V , *shrink*

Relation between external shrinkage and undercooling



Causes	1) S% in melt is too low. 2) O% in melt is too much
Countermeasure	① Add sulfur to reach the range of 0.09 to 0.095%S. ② Deoxidation by adding SiC. Note: Both ① and ② are done to the original melt.

We are prepared to perform calibrations of melt quality assessment functions for you.
Inquire the programmer Higuchi Ayumu for details.

Set up of the apparatus

consumables

Technical support

Off-set setting

We can offer assistance to preparation of calibration lines (to be charged).

Free rental for demonstration

Free rental with a cup stand (two~three days).

Maintenance

Free maintenance for the initial one year. Paid maintenance after the initial period.
Cost of repair of physical damages by accident must be paid by the customer, even though it may be within the period of free guarantee.

Technical note

A booklet is provided describing details of countermeasures.

Temperature sensor

Temperature calibration and paper preparation after ISO standard (to be paid).

Specification of the apparatus

Outside dimension	H440 × W590 × D175 mm
Monitor	21 inch touch panel (supersonic type)
OS	Windows Embedded
Storage period	Three years
Power source	AC 100 to 240 V

Temperature convertor	JIS-K measurement range: 0 to 1340 C
Interface	USB×1 VGA×1 RS232×1 LAN×1 (option)
Cup stand	two sets (5m)
Weight	15 kg

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