Heraeus

Thermal Analysis of Cast Iron



Electro-Nite

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1. PREFACE

Thermal analysis is a well-proven method commonly used in the iron foundry for a rapid, accurate, reliable and low cost control on the shop floor. It enables to determine carbon equivalent (CEL), saturation degree (Sc), carbon and silicon content in most iron grades. Further applications are the determination of the eutectic undercooling and control of the inoculation degree. It can also predict some mechanical and physical properties, such as tensile strength (Rm), Brinell hardness (HB), tensile strength/hardness quotient (Z/H), graphitization factor (K) and quantity of eutectic graphite (MEG) in unalloyed lamellar iron castings.

The aim of this booklet is to give a clear overview of typical thermal analysis applications in cast iron, to indicate the most appropriate tools (different cups and instrumentation) for obtaining optimal results and to pass some tips and recommendations to get the utmost out of this control method.

For in-depth metallurgical explanations of transformations and phenomena occurring during cooling down a liquid iron sample to room temperature, a large number of specific publications can be consulted.

2. BASIC PRINCIPLES

Thermal analysis is based on the interpretation of the cooling trace – temperature versus time – of a solidifying metal alloy showing some particularities (change of slope, undercooling, recalescence, thermal arrests) which correspond with transformations of the alloy during solidification.

For better understanding the basic principles, let us consider a typical binary phase equilibrium diagram (fig. 1 left). It shows the temperature/composition diagram of an alloy consisting of two elements A and B which are mutually soluble in the liquid state and completely insoluble in the solid state. TA and TB are the melting temperatures of pure element A and pure element B.



Fig. 1 – Complete solubility in the liquid state and complete insolubility in the solid state

Four zones can be distinguished in this binary phase diagram:
zone 1: above the top curve TAE - ETB (called liquidus curve), the system consists of a solution of liquid element A and liquid element B
zone 2: crystals of element A in equilibrium with liquid alloy
zone 3: crystals of element B in equilibrium with liquid alloy
zone 4: solid state, mixture of crystals of pure A and pure B

When cooling down a liquid metal alloy consisting of x% A and y% B, solid crystals B in equilibrium with liquid alloy (composition given by the liquidus curve) start to precipitate at TL (liquidus temperature) and a change of slope in the cooling curve appears (fig. 1 right) due to the release of solidification heat of crystals B. At the eutectic temperature TE a thermal arrest, called eutectic arrest, will occur. Indeed, according to Gibbs' phase rule F' = C - P + 1 (adapted form for metallurgical systems under normal atmospheric conditions), in which F' is the number of degrees of freedom, C is the number of components (A and B) and P is the number of phases (liquid alloy, crystals A and crystals B), the system is invariant at TE. Temperature will remain constant until one of the three phases disappears, in this case the liquid phase. Obviously, the length of the eutectic arrest will depend from the alloy's composition and will reach a maximum at the eutectic composition corresponding with point E in fig. 1 left.

3. THERMAL ANALYSIS OF UNALLOYED GREY CAST IRON

3.1. Introduction

Cast iron is by definition an Fe-C alloy containing also Si, Mn, P and S in variable amounts, and in which carbon content is sufficiently high so that graphite in the stable system (broken lines in the Fe-C diagram fig. 2) or cementite in the metastable system (full lines) are formed on solidification.



Fig. 2 – The pure Fe-C diagram

The Fe-C diagram clearly shows the unique relation between the liquidus temperature and the carbon content. However, the pure binary Fe-C diagram is not usable for thermal analysis of cast iron due to the important influence of mainly Si and P on the location of the characteristic points C' (or C) and E' (or E), as well as on the liquidus (TL) and solidus (TS) temperatures. The knowledge of the quaternary Fe-C-Si-P diagram would be necessary.

Initially, it was estimated that for cast iron the eutectic composition does not occur at about 4.30% of carbon (such as in the pure binary Fe-C system), but at a lower carbon content given by the simplified equation:

%C = 4.30 - 1/3 (%Si + %P)

The influence of Si and P can be expressed in terms of carbon, hence the notion of Carbon Equivalent Value:

CEV = %C + 1/3 (%Si + %P)

The CEV of a cast iron gives an idea about how much its composition differs from the eutectic composition. A cast iron is considered to be hypoeutectic, eutectic or hypereutectic when the carbon equivalent is lower than, equal to, or higher than 4.30 %.

In stead of CEV, the influence of Si and P is sometimes expressed by the saturation degree (Sc), comparing the total carbon content of the iron with the carbon content of the strict eutectic composition:

$Sc = %C_{total} / [4.30 - 1/3 (%Si + %P)]$

If Sc = 1, it means that the iron composition corresponds exactly with the eutectic composition. Values lower or higher than 1 correspond respectively with hypoeutectic or hypereutectic cast iron.

Fig. 3 illustrates a typical cooling trace of hypoeutectic grey cast iron solidifying in the stable system, showing the liquidus arrest temperature TL and eutectic undercooling.

Fig. 4 illustrates a typical cooling trace of hypoeutectic white cast iron solidifying in the metastable system, showing the liquidus arrest temperature TL and a well defined white eutectic arrest without undercooling, the so called solidus temperature TS.







Fig. 4 – Typical cooling trace of white solidified cast iron

3.2. Determination of carbon equivalent / saturation degree

As mentioned before, the pure binary Fe-C diagram can not be used as such for thermal analysis of cast iron. In order to evade the complexity of quaternary diagrams, statistical studies have been made by several authors to find a direct correlation between the liquidus temperature and the C, Si and P contents.

By multiple regression analysis, and using Heraeus Electro-Nite's test cups, following equation has been found:

TL (in °C) = 1623.60 - 112.36 (%C + %Si/4 + %P/2)

in which CEL = %C + %Si/4 + %P/2

CEL is the so called Carbon Equivalent Liquidus. It is recommended to use this improved definition of carbon equivalent, in particular when it is directly calculated from the liquidus temperature measured by means of thermal analysis of the iron.

From above equation, following formula is deducted for CEL calculation:

 $CEL = 14.45 - 0.0089^{*}TL$ (1)

This formula is graphically represented in fig. 5.

Standard error is +/-0.047 CEL and correlation coefficient r = 0.987





Similar studies as made for CEL, have led to following formula for Sc:

Sc = 3.674 - 0.0023 * TL (2)

Determination of CEL by thermal analysis shows however some important restrictions. With increasing CEL values, the liquidus arrest approaches the eutectic arrest and, finally, at the eutectic composition the liquidus arrest coincides with the eutectic arrest. The solidification of hypereutectic grey iron, starting with the precipitation of graphite (stable system), does not give rise to a clearly recognizable thermal arrest due to the small amount of heat liberated by this crystallization.

To extend the measurement range of CEL, several methods have been tried out. The best method consists in making solidify the potentially grey iron as a white iron by adding a strong carbide forming element such as tellurium. Fig. 6 shows on an enlarged scale how the grey eutectic point C' is shifted when the iron solidifies white (metastable system). A hypereutectic grey iron (vertical line) can become now a hypoeutectic white iron in the metastable system.



Fig. 6 - Extension of the measuring range of CEL values by white solidification

Tests made by the British Cast Iron Research Association (BCIRA) have revealed that by using this method TL and consequently CEL measurements are possible for iron compositions corresponding to the following equation (TL coinciding with TS in the case of white solidification):

 $%C + \%Si/9 + \%P/3.5 \le 4.30$

Besides the extension of the CEL measurements' range, the forced white solidification has the big advantage that in contrast with grey solidification (see fig. 3) no undercooling appears, which means that TS is simply and correctly to determine.

As said before, through a CEL or Sc measurement it can be seen how much the composition of the concerned cast iron differs from the eutectic composition. Moreover, tensile strength Rm (standard tensile strength in a \emptyset 30 mm test bar) can be directly deducted from Sc for unalloyed lamellar iron grades (see further).

3.3. Determination of carbon content

It has been found that with each pair of TL and TS values measured in the metastable system, only one carbon content corresponds. This relation is given by the following equation:

%C = -6.51 - 0.0084*TL + 0.0178*TS(3)

This formula is graphically represented in fig. 7.

Standard error is +/-0.039 C and correlation coefficient r = 0.991



Fig. 7 – Relation between %C, TL and TS

3.4. Determination of silicon content

It is well-known that in the metastable system (white solidified iron), the eutectic or solidus temperature decreases with increasing silicon contents. This behaviour is characteristic for all graphitizing elements such as P, Al, Si, Cu and Ni.

For Si values between 1 and 3% and P < 0.2%, a polynomial regression shows that the best correlation is found between TS and Si + 4P:

 $TS = 1138.2 - 6.4 (Si + 4P) - 1.65 (Si + 4P)^2$

Standard error is +/-0.10 (Si + 4P) and correlation coefficient r = 0.981

For practical use however, this formula can be simplified to:

Si = 78.411 - 4.28087 Si - adj. - 0.06831 TS (4)

in which Si-adj. is a correction factor, mainly depending on the P content of the cast iron.

This formula is graphically represented in fig. 8.



Fig. 8 - Relation between %Si and TS for different P contents

From above formula it is clear that in contrast with CEL and C calculation, thermal analysis provides only a rough estimation of the Si content, even when the P content is very low (in that case the Si-adj. correction factor equals more or less the P content of the iron). Indeed, 1 °C difference on TS measurement already causes a difference of 0.07% on calculated %Si. Hence, frequent calibration of the measuring instrument is necessary and measurements should not be taken before the instrument has reached thermal stability.

To find the proper Si-adj. value for a particular iron grade, the following procedure can be followed:

- measure TS by means of the appropriate thermal analysis equipment and compare it with the Si analysis of a simultaneously taken spectrographic sample
- knowing TS and Si, the correction factor can be determined either by means of existing thermal analysis tables or by means of above Si formula
- repeat this five times for the same iron grade
- the average value of the so determined correction values will be the most adequate Si-adj. value to be selected each time this particular iron grade is cast

3.5. Undercooling of the eutectic temperature

3.5.1. Definition

Several authors, among them Prof. Czikel and Dr. Hummer, consider the difference between the maximum and minimum eutectic temperature as the eutectic undercooling (Δ TM in fig. 3). This difference is also known as eutectic recalescence.

Others like Dr. Ing. Caspers, define undercooling as the difference between a reference temperature of 1150 °C (+/- the eutectic equilibrium temperature in the Fe-C diagram) and the minimum of the eutectic temperature (ΔT in fig. 3).

Measurement of undercooling, whether or not in combination with TL, enables to control inoculation and to predict certain mechanical properties of the cast iron such as tensile strength (Rm) and Brinell hardness (HB).

3.5.2. Inoculation control

By inoculation is meant the addition to the molten iron just before casting of products based mainly on graphite, ferrosilicon or silicon calcium, in order to increase the nucleation potential of the melt for graphite. Inoculants strengthen the tendency to grey solidification, affect the graphite shape and increase the number of eutectic cells.

It is well-known that inoculation considerably reduces eutectic undercooling. Dr. Ing. Caspers has introduced the notion of undercooling quotient based on his definition of undercooling. The undercooling quotient is the quotient of the measured undercooling before and after inoculation. The result of extensive research made by him for iron with CEL between 3.86 and 4.08% is shown in fig. 9. The diagram shows the relation between the undercooling quotient, the intensity of inoculation and the number of eutectic cells. As it can be seen, the undercooling quotient is a practicable aid to control inoculation.



Fig. 9 – Relation between the undercooling quotient, the intensity of inoculation and the number of eutectic cells

3.5.3. Undercooling and its relation to mechanical properties

The standard tensile strength Rm in a \emptyset 30 mm test bar cast in sand depends on the composition and nucleation degree of the molten iron.

Based on statistical studies, Heller and Jungbluth found that the relation between the standard tensile strength and the composition of unalloyed iron grades can be expressed by following formula:

$Rm (in N/mm^2) = 9.81 (102 - 82.5 * Sc)$

Standard error is +/- 25 N/mm²

It was also determined that within the range of 150 to 400 N/mm², following relation between tensile strength and Brinell hardness (HB) is valid:

HB = 538.6 - 354.75*Sc

However, these equations can only be used in case graphite is mainly of type A. Consequently, these relations are not valid for thin wall castings (wall thickness < 15 mm).

The spread on the results obtained with the above Rm formula can be explained by the fact that the nucleation degree is not taken into account.

Tensile strength and hardness of cast irons having the same carbon equivalent (or Sc) can vary widely. Indeed, it has been found that in case undercooling remains low for a particular iron composition, tensile strength will tend to high values, whereas hardness will tend to low values. However, when undercooling becomes important, tensile strength will decrease and hardness will increase slightly.



Fig. 10 – Quality diagram for grey iron based on results obtained by thermal analysis (J. Czikel and R. Hummer)

Studies made by J. Czikel and R. Hummer prove that tensile strength and hardness in the Ø 30 mm test bar can be determined more accurately if besides TL (which is related to CEL or Sc, and so to the iron composition), also ΔTM (which is related to the nucleation degree) are taken into account. Both, TL and ΔTM , are easily measured by thermal analysis.

Their so-called Quality Diagram shown in fig. 10, correlates tensile strength and hardness of various iron compositions characterized by their liquidus temperature in function of different undercooling (recalescence) ranges.

It proves that thermal analysis enables identification of high quality cast iron.

3.6. Calculation of tensile strength / hardness quotient, graphitization factor and quantity of eutectic graphite

Thermal analysis also enables calculation of following quality characteristics of grey iron:

Z/H (Zugfestigkeit/Härte in German) is the quotient of the standard tensile strength (Rm) and Brinell hardness (HB). It gives an idea about the machinability of a particular iron grade. Cast irons having the same tensile strength can vary widely in hardness (see in this respect fig. 10). For equal tensile strength, machinability will decrease with increasing Brinell hardness.

K is the so called graphitization factor which expresses the influence of the composition on the structure of unalloyed grey iron grades. Both the cast iron composition and the cooling rate (wall thickness of the casting) determine the tendency to grey solidification. With regard to the influence of the composition of low phosphorous cast iron grades, mainly the carbon and silicon content are important. Within their usual limits in cast iron, variations of manganese and sulphur have no notable effect. For equal wall thicknesses, grey solidification tendency will increase with increasing K-values. K is determined by following formula:

MEG (Menge des eutektischen Graphits in German) is the portion of carbon precipitated as eutectic graphite on solidification of cast iron. It is expressed by following formulas:

MEG = %C - 1.3 + 0.1(%Si + %P)	for $Sc \leq 1$
MEG = 2.93 – 0.22(%Si + %P)	for $Sc > 1$

The precipitation of graphite in grey cast iron leads to a volume increase which works against shrinkage and shrinkage formation. The higher the quantity of precipitated eutectic graphite, the lower will be the shrinkage formation.

4. THERMAL ANALYSIS OF DUCTILE IRON

Similar to unalloyed lamellar iron, CEL, C and Si can be determined in ductile iron (also called S.G. iron or nodular cast iron) by means of thermal analysis.

As seen before, tellurium is added to the test sample (QuiK-Cup[®] thermal arrest cup) to extend the measurement range of CEL and to make solidify the iron in the metastable system (white solidified iron) to obtain an easy readable solidus arrest necessary for C and Si determination.

The use of a standard thermal arrest cup with Te as carbide forming element is not possible in this case because of the reaction between magnesium from the nodularizer and the tellurium addition in the cup.

Heraeus Electro-Nite has resolved this problem by the addition of a small, well determined amount of sulphur which reacts with Mg, leaving Te free to promote the required white solidification of the molten iron sample.

However, for the calculation of CEL, C and Si, it must be taken into account that the sulphur content in the test cup decreases TL with 5 °C and TS with 2.5 °C.

4.1. Determination of carbon equivalent

Formula (1) remains valid if the measured TL is increased with 5 °C:

CEL = 14.45 - 0.0089*(TL + 5)

The standard error on CEL determination is +/-0.05 CEL, which is almost the same as found with the test cup with Te only.

4.2. Determination of carbon content

When we consider equation (3) for lamellar cast iron:

%C = - 6.51 - 0.0084*TL + 0.0178*TS

we notice that the ratio between the coefficients of TL and TS is 1/2 and that they have opposite signs. The coefficients are thus inversely proportional to the above mentioned effect of sulphur on TL and TS. This means that equation (3) remains valid with the same accuracy in this case.

4.3. Determination of silicon content

As seen before, it should be taken into account that the sulphur content in the test cup

decreases TS with 2.5 °C. This means that equation (4) must be changed to:

%Si = 78.411 - 4.28087*Si-adj. - 0.06831*(TS + 2.5)

The accuracy on Si calculation is almost not affected by the sulphur content in test cup.

4.4. Inoculation control

Although the use of undercooling in ductile iron is not as evident as in lamellar iron, it has been found that test cups with reduced volume facilitate considerably the examination of different inoculation treatments thanks to the generated high cooling speed increasing the undercooling effect.

5. MEASURING EQUIPMENT

It is obvious that only a high quality measuring system can provide the necessary accuracy to get full advantage of the described applications of thermal analysis.

Fig. 11 shows the basic measuring system, consisting of:

- an appropriate QuiK-Cup[®] disposable test cup (for different types and applications: see further)
- a cup holder with QuiK-Cup contact block
- a type K extension wire
- a cable plug
- a QuiK-Lab-E thermal analysis instrument



Fig. 11 – Basic thermal analysis measuring system

Fig. 12 shows an example of a MeltControl system consisting of local QuiK-Lab-E WinProcess instruments for thermal analysis, a Digitemp-E WinProcess for bath temperature and a computer with installed MeltControl 2000-Win software.



Fig. 12 – MeltControl system for thermal analysis and bath temperature

5.1. QuiK-Cup[®] types and their applications

QuiK-Cup[®] is Heraeus Electro-Nite's brand name for a series of disposable measuring cups for thermal analysis of iron. The cups are made of shell moulding sand and are fitted with a robust and reliable connecting system. Their volume is chosen to meet the best compromise between measuring time and consistency of the measuring results. They are characterized by a quartz protected high grade type K thermocouple for accurate measurements, welded against the quartz tube for obtaining short response times. If white solidification is required, a special tellurium mixture is cemented on the cup's bottom for intensive mixing with the liquid iron.

5.1.1. <u>QC 4010 (cup without Te)</u>

- CEL determination in hypoeutectic iron
- eutectic undercooling measurement



Fig. 13 - QC 4010 and typical cooling trace

5.1.2. <u>QC 4011</u> (cup with Te)

- CEL determination in hypo- and hypereutectic iron
- %C and %Si determination in both hypo- and hypereutectic lamellar iron



Fig. 14 - QC 4011 and typical cooling trace

- 5.1.3. <u>QC 4012</u> (cup with Te and S)
- see QC 4011, but for magnesium treated iron (nodular- and vermicular iron)



Fig. 15 - QC 4012 and typical cooling trace

5.1.4. QC 4000 (cup with reduced volume)

- inoculation control in ductile iron



Fig. 16 - QC 4000 and influence of cup width on undercooling

5.2. QuiK-Cup auxiliary hardware (fig. 17)

5.2.1. QuiK-Cup contact block (ref. LC 31016015)

The heat resistant contact block assures good electrical contact between the cup contacts and the special extension cable leading to the measuring instrument. Two springy large contact bars made on the one hand from a NiCr alloy (positive contact) and on the other hand from a NiAl alloy (negative contact) are positioned in such a way that, even when the test cup is firmly pushed onto the contact block, the hot cup can not touch the electric insulation of the contact block basis preventing that its life time could be shortened. Two embedded centring and guiding pins guarantee correct positioning of the cup on the contact block.



Fig. 17 – QuiK-Cup auxiliary hardware

5.2.2. QuiK-Cup stand (ref. LC 31016001)

The QuiK-Cup stand is a very stable and easy to handle support device which holds the contact block and protects part of the extension wire against heat and possible metal splashes when filling the measuring cup.

5.2.3. Extension wire (ref. LC 31024001)

The type K extension wire conducts the electric signal generated by the cup's thermocouple to the measuring instrument. It is extremely important for the exactness of the measurement that the conductors are made of the same NiCr and NiAl alloys as the thermocouple.

5.2.4. Cable plug (ref. RM 24167003)

The male cable plug connects the extension cable to the built-in female connector of the measuring instrument. The conductor pins are made of the same NiCr and NiAl alloys as the cup's thermocouple. Both shape and dimensions of the pins are different to exclude wrong connection.

5.3. Measuring instruments

Heraeus Electro-Nite offers two types of digital thermal analysers for automatic interpretation of the cooling trace, calculation and display of the final results.

5.3.1. QuiK-Lab-E

The QuiK-Lab-E measuring instrument automatically recognizes grey or white solidification. Within the instrument's measuring range from 400 to 1370 °C, its trace evaluation method accurately determines TL, TS, ΔT and ΔTM . Based on these measuring results and the earlier given formulas (1 to 4), CEL, Sc, %C and %Si are calculated.

Each individual measured or calculated value is selected by means of a rotary switch and displayed on a bright and large multi-function display.

An additional calculation program for Rm, HB, Z/H, K and MEG of unalloyed lamellar iron, is available as an option.

The measuring status is indicated by three coloured signal lights (ready, measurement and complete).

QuiK-Lab-E exists in different versions: freestanding, for wall-mounting (with max. two additional displays) and for panel-mounting (with max. three additional analysis displays). A QuiK-Lab-E wall-mounting instrument with integrated Digitemp-E bath temperature measuring instrument is also available.



Fig. 18 – QuiK-Lab-E thermal analyser

5.3.2. MeltControl system

The MeltControl system consists of local measuring devices such as QuiK-Lab-E WinProcess (thermal analyzer), Digitemp-E WinProcess (bath temperature measuring instrument) and a PC with installed MeltControl 2000-Win software.

Although it can operate as a local independent measuring station for thermal analysis, the QuiK-Lab-E WinProcess instrument is mainly intended to form an integral part of the MeltControl system.

QuiK-Lab-E WinProcess instruments have the same evaluation and calculation programs as the QuiK-Lab-E instruments. In addition, they are designed to run in an internal network environment.

The MeltControl software allows storing and processing of all incoming values. Cooling traces are clearly visualized together with the measured and calculated values. Customized calculation formulas may be programmed. Up to three cooling traces can be displayed for analysis comparison.

Optional program modules for charge calculation, weighing data, active oxygen measurement, spectrographic analysis, melting reports, casting reports, SPC, etc. can be installed.



Fig. 19 – Examples of MeltControl 2000 Win displays

6. MEASURING PROCEDURE

Make sure that the cup stand is levelled.

Clean the contact block contacts if necessary, and make sure that contact block and measuring instrument are correctly connected and work properly.

Plug the appropriate type of QuiK-Cup[®] onto the contact block so that good electrical contact is achieved.

Take a sample of molten iron and pour it, after removal of the slag, progressively into the cup until the cup is filled completely. Avoid pouring straight on the thermocouple. By preference, pouring is done by means of a ceramic fibre spoon to keep heat loss to a minimum and provide clean metal to the cup.

To get a usable cooling trace, it is necessary that the peak temperature reached in the cup remains below 1370 °C and is at least 20 °C higher than the liquidus temperature.

As soon as the cooling trace is recorded and the calculated values are displayed, remove the used cup by means of a pair of tongs to prevent overheating of the contact block.

7. MAINTENANCE

To maintain reliable, consistent and high-precision results, it is necessary to keep auxiliary hardware and measuring instrument in good working condition by following actions.

Wipe off possible resin deposits from the contact block contacts by means of a cleaning cloth or a brass brush.

Replace the contact block if contacts or insulation is damaged.

Do the same with the extension cable if the outer sheathing is burnt or damaged. Pay attention that the proper type K cable is used.

Control regularly the insulation (with the QuiK-Lab-E disconnected) of contact block, extension cable and connections as well as the calibration of the measuring instrument. Specially designed for this purpose, use preferably the Checkmate Positherm/QuiK-Cup portable instrument with adapter fitting on the QuiK-Cup contact block (fig. 20). This test instrument is battery operated and can simulate three bath temperatures and three thermal arrest temperatures. Insulation check is made between all conductors and between each conductor and earth.



Fig. 20 – Checkmate Positherm/QuiK-Cup with QC adapter

8. PRACTICAL TIPS AND SAFETY MEASURES

Store QuiK-Cup[®] test cups in a dry place and take them only out of their plastic packing if they are going to be used soon.

Put some dry sand on and around the QuiK-Cup stand base. Spilled iron can be removed much easier in such case.

If metal pouring spoons are used, warm them up before sampling to minimize heat loss. Keep in mind that graphite based coatings may be a source of spread on the measurements. Better is the use of ceramic fibre pouring spoons.

Keep used cups containing tellurium separate from other scrap; Te is a strong carbide forming element and may affect a grey iron melt if disposed in it.

When cups with Te are filled, fumes containing Te may escape. It is advised to foresee a draught hood above the QuiK-Cup set up.

Wear fire-proof protective clothes, safety shoes, safety glasses or a face shield and a proper helmet.

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