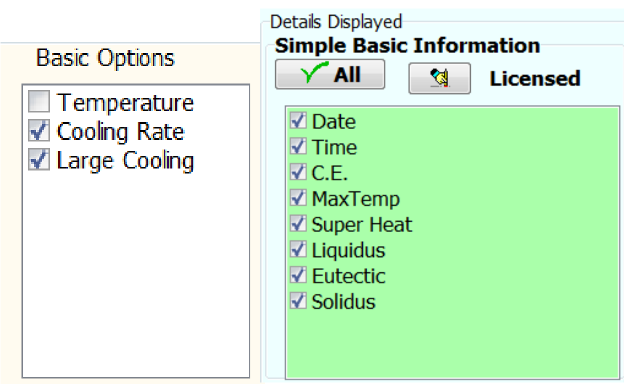
Gray Iron Microanalysis© Information Available

The term Microanalysis© is used in reference to a form of thermal analysis that locates and evaluates both large and small thermal events that pertain to the microstructure and quality of a metal. These include microscopically small events such as carbides, gas, and micro-porosity as well as large events that relate to physical properties such as hardness, chill, strength, machineability, heat treating, fraction solid and macro-shrinkage. These properties can guide the foundry in preparing a more consistent metal in regular production, or in researching the suitability of inoculates, additions and chemistry changes to meet customer needs.

MeltLab has four levels of operation: Simple Basic, Basic, Advanced and Expert for different levels of information. Simple basic provides what most competitors offer and is our least expensive level. Since this is for Gray Iron Microanalysis, a plain cup without tellurium is expected. This allows the iron to solidify as a casting of that chemistry would. To be able to see carbon and silicon calculations, the tellurium cup Chemistry method would be used instead. The following descriptions will contain some new assertions that may be unfamiliar to the reader. We encourage those interested to ask further questions.

Simple Basic Analysis for Gray Iron Microanalysis



The All button selects all information listed in the box. The hand with a pencil eraser unchecks all tests within the box. If this level is licensed, then the box appears green and the word "Licensed" appears. If licensed, then individual tests can be turned on (checked) or turned off (unchecked). This selection only applies to the selected metal type found in the upper right-hand side of the screen. Each Metal type may need configuration, or you may use the Duplicate function to copy the settings from one curve to another.

Simple Basic Option Curves

- Temperature Curve is the basic data gathered by the thermal couple.
- Cooling Rate Curve is the instantaneous change in temperature. For mathematicians, this is the first derivative inverted. This visual occupies the lower 25% of the curve window.
- The Lard Cooling option gives the Cooling Rate curve 50% of the curve window.
- For maximum size of the Cooling rate curve, simply click off the Temperature curve and the Cooling rate will occupy the entire window.

These options suggest that the Cooling Rate curve is very important, and it is. Details of arrests are much clearer in the Rate of Cooling due to magnification. The Temperature curve may span 300 degrees while the rate of cooling spans only 10 degrees – a magnification of 30x.

Gray Iron Microanalysis© Information Available

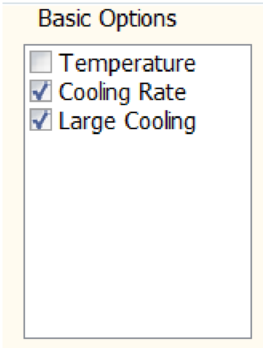
Information and Variables of Simple Basic Microanalysis

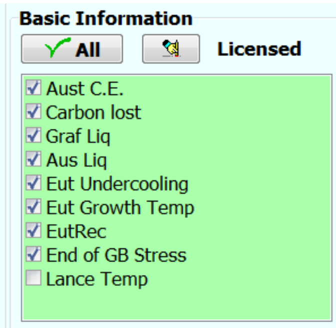
- Date is the day of the sample according to the computer's clock that recorded it.
- Time is the start time of the sample when the computer senses hot metal in the cup.
- C.E. or Carbon Equivalent is determined by the temperature of the liquidus as it relates to the iron-carbon phase diagram. It may not agree with a chemistry-based calculation from a spectrometer, but it is the correct C.E. Chemistry based equations try to imitate Thermal C.E. and are not always correct.
- Maximum Temperature is the highest temperature the thermal couple saw. It is usually 200 to 300 degrees F below the furnace temperature due to heat losses in sampling and the cup cooling the sample. The thermal couple is made of two nickel-based alloys. The lowest one melts at 2540 degrees F. Try to keep the maximum temperature below 2500 and at least 50 degrees above liquidus. With care, even malleable iron, which has a high liquidus, can be sampled.
- Superheat is the Maximum Temperature minus the Liquidus temperature. It is best to have about 50 degrees superheat if that can be managed, but 25 degrees will work if necessary. Higher superheat will reveal oxide arrests as well as giving a good profile of the liquidus. A superheat close to zero may slightly underestimate the liquidus as it only captures the tail end of the arrest. Sampling too cold may even cause the system to miss the liquidus altogether.
- Liquidus is the strongest point of heat generation in the liquidus zone. This is the lowest point of the rate of cooling. The C.E. test is based on the temperature of this strong point. If the arrest is particularly weak, the liquidus detection value may need to be increased. Current research indicates that trace amounts of titanium in the metal can weaken this arrest. This often occurs when the steel content of the charge is increased, or the foundry finds a new supply of steel scrap.
- Eutectic is the larger (longer) arrest and will usually have undercooling and recalescence. These tests are offered in higher levels of MeltLab. For use at this level, the eutectic temperature is the strongest or highest temperature that occurs during recalescence. This has been referred to as the "Growth Temperature" of the Eutectic.
- Solidus or End of Freezing is the strong point in the Solidus zone. This zone is where the grain boundaries turn solid. These grain boundaries are loosely and randomly packed and so the zone is heat absorbing rather than heat producing. Low melting tramp elements are pushed into these areas between the grains in a process called micro-segregation.

While this level provides only the bare essentials in understanding the material, these values are used in the higher levels to provide much more information on inoculation, physical properties, casting gas and shrinkage, and even the relative ability for the castings to be heat-treated.

Gray Iron Microanalysis© Information Available

Basic Analysis for Gray Iron Microanalysis





The next level is called "Basic" and includes many more useful values. At the Basic level all tests in the Simple Basic are included as well. The same rules on licensing apply to each level so if this screen is green, you can use these tests.

To be a full featured system, MeltLab includes tests the never show up in your material. The Graphic Liquidus (Graf Liq) may never show up if you keep your C.E. below a 4.3. But if you do see it, MeltLab will tell you how much graphite expansion you lost back into your risers. Yes, some people still make solid risers and don't know why. MeltLab does and tells you about it.

Basic Option Curves for Gray Iron Microanalysis

These are the same as the Simple Basic options.

- Temperature Curve is the basic data gathered by the thermal couple.
- Cooling Rate Curve is the instantaneous change in temperature. For mathematicians, this is the first derivative inverted. This visual occupies the lower 25% of the curve window.
- The Lard Cooling option gives the Cooling Rate curve 50% of the curve window.
- For maximum size of the Cooling rate curve, simply click off the Temperature curve and the Cooling rate will occupy the entire window.

These options suggest that the Cooling Rate curve is very important, and it is. Details of arrests are much clearer in the Rate of Cooling due to magnification. The Temperature curve may span 300 degrees while the rate of cooling spans only 10 degrees – a magnification of 30x.

Information and Variables for Basic in Gray Iron Microanalysis

- Austenite C.E. is calculated from the normal austenite liquidus.
- Graphite Liquidus is a weaker arrest that only occurs if the C.E. goes too high. Some of your detection limits may need adjusting to pick this out.
- Carbon lost is the difference between the graphitic C.E. and the Austenite C.E. assuming both occurred. An iron showing a graphite C.E. pf 4.50 and an austenite C.E. of 4.20 will make about 0.30 percent graphite in the liquid iron before the gates freeze off. This pushes iron back out the gates and into the risers. When the graphite liquidus finally stops the carbon-content has fallen lower than what might be expected, and the iron is usually several points below eutectic composition. Please note that this is an uncommon situation that generally does not occur. Because we have better control over our chemistry.
- Eutectic undercooling is the lowest temperature before the eutectic arrest. Generally, some undercooling is needed to get the eutectic solidification started. Inoculation tends to raise this temperature.

Gray Iron Microanalysis© Information Available

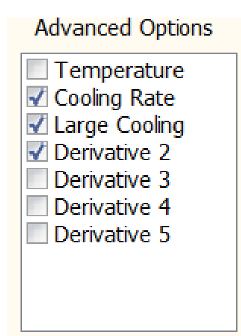
- Eutectic growth temperature is the highest temperature in the eutectic arrest. This is also the liquids temperature. Some people experiment with small cups or accelerated cooling and end up suppressing (pushing down) the liquidus value. Standard cups have been sized large enough to prevent suppressing. There are some ways to tell by looking closely at the curve if this has occurred.
- Eutectic Recalescence is how much reheating has occurred due to the eutectic arrest. Recalescence is the Eutectic Temperature – the Eutectic Undercooling Temperature. Typical readings vary with the casting thickness. Thinner castings generally have more inoculation and thus less recalescence. This also depends on the type of inoculant being used which will depend on the casting shape and the properties needed.
- End of Grain Boundary Stress is when the grain boundaries are fully solidified. The lower this temperature is, the longer any heat-treating will take.
- Lance Temperature is only for systems that have temperature lances included with their MeltLab. It causes the most recent temperature lance reading to be included in the sample report.

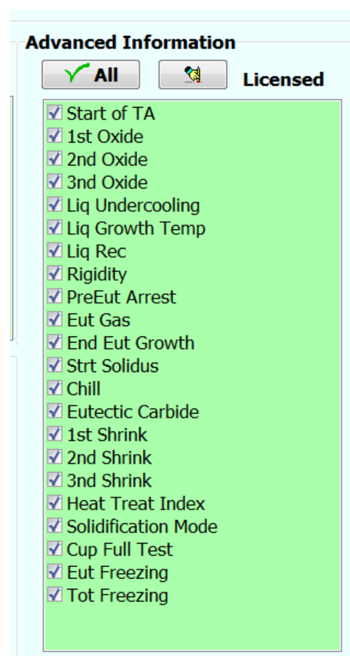
Advanced Analysis for Gray Iron Microanalysis

While Basic and Simple Basic contained tests often addressed in past research, Advanced Information includes most all reported inflection points and calculations and some new ones as well. These points are based on derivatives or changes in the acceleration and deceleration of heat production. These can be seen by turning on higher order derivatives in the Curve configuration module. At this level, derivatives two through 5 are provided as well as the temperature curve and the rate of cooling curve.

Advanced Option Microanalysis Curves

- Temperature Curve is the basic data gathered by the thermal couple.
- Cooling Rate Curve is the instantaneous change in temperature.
- The Lard Cooling option gives the Cooling Rate curve 50% of the curve window.
- Derivative 2 shows inflections in the Rate of Cooling increasing magnification to typically 300x.
- Derivative 3 shows inflections in the 2nd derivative curve further increasing magnification to typically 3000x or more.
- Derivatives 4 and 5 likewise show energy activity at higher and higher magnifications reaching well over 10,000x in sensitivity.





The question is of what use are these higher order derivatives? They give us the data points needed to accurately measure minima and maxima of lesser derivatives. The second derivative gives us the liquidus strong point. The third derivative tells us the steepest slope of an arrest. This is the acceleration or deceleration of the arrest. And the fourth derivative tells us the beginning and the end of an arrest. For a very weak arrest, the necessary derivatives shift down a level. So derivatives are giving us some mathematical certainty in determining location and the beginning and ending of arrests.

Information and Variables of Advanced Microanalysis of Gray Iron

- Start of Thermal Analysis is the point at which the thermal couple has stabilized with the metal. All arrest points excluding Maximum Temperature will occur after this point.
- 1st, 2nd and 3rd Oxide Attests are arrests consisting of Iron Oxide and Silicon Oxide commonly called Mullite. Oxide arrests are from oxygen retained in the metal from rusty scrap. Oxides will react with your inoculant and lessen their effectiveness unless countered by the calcium content of your inoculant. High oxide levels in the iron will result in burn-on.
- Liquidus Undercooling rarely happens but we have included it for people who might make strange irons. It occurs frequently in Aluminum and Steel, but not so often in iron.
- Liquid Growth temperature goes with Liquidus undercooling and the same situation as mentioned in the undercooling.
- Liquidus recalescence is the Liquidus Growth temperature – the Liquidus Undercooling Temperature.
- The Rigidity point is the highest rate of cooling between Liquidus and Eutectic. Here is where the eutectic material first starts to slowly grow. It is also where pre-eutectic carbides grow.
- Pre-eutectic arrests can be several things: early graphite/austenite, carbides or unknows we are still investigating.
- Eutectic Gas are larger bubbles that form from entrained gases.
- End of Eutectic Growth is the point of maximum stress where the energy production falls off.
- Start of Solidus is where the zero or base-line curve intersects the cooling rate curve. It defines the start of the solidus energy adsorption.
- Chill is a calculated chill based on local foundry research into the chill potential of an iron.
- Eutectic Carbides are small carbides that form late into the eutectic. This is different from the massive carbides formed in White Iron.
- Shrink 1st, 2nd and 3rd small macro-shrinks often appear in a series of tiny arrests. These are indicated, Large shrink is more obvious and will also be indicated. The next level goes into this in depth.
- Hat Treat index is the difference between Eutectic and Solidus in degrees C. If the casting is to go through solution heat-treat to make ADI, a maximum of 25 degrees C is recommended by Advanced Process Corporation,
- Solidification mode can be hypoeutectic, eutectic, hypereutectic or hypo and hypereutectic.
- Cup freezing test is the time in seconds for the temperature to fall from the first to the second temperature as set by the user. Generally, we set the start and stop temperatures to bound the eutectic arrest. If ranges are set, then the percent of the midpoint of the range is displayed.
- Eutectic Freezing is the Eutectic temperature – the Solidus temperature.
- Total Freezing is the Liquidus Temperature – the Solidus temperature.

Gray Iron Microanalysis© Information Available

Expert Analysis for Gray Iron Microanalysis

Expert is a large step forward where additional information available for the first time in the world is added to the micro-Thermoanalysis. This level adds fraction solid, and the heat of fusion of the sample by integrating the area between the rate of cooling curve and the baseline curve. Then each segment of solidification is integrated and ratioed against the total heat of fusion. Reporting these values as a percent of the heat of fusion corrects for slight changes in sample size. In addition, the various derivative curves can be displayed, and their data put into an excel format.

Expert Option Curves

- Zero Curve or Baseline Curve is the cooling rate if no crystallization or solidification occurred. The area between this curve and the cooling rate bounds the heat of fusion.
- Color integration adds color to the individual zones for quicker identification.
- % Solid includes the percent fraction solid curve to the temperature curve. This is useful for solidification modeling. A detailed list of the fraction solid is included in the data drop if requested.

Information and Variables of Gray Iron Expert Microanalysis

- Nucleation is the point at which heat energy first begins to slow the cooling of the sample. It can be missed if there is insufficient superheat in the sample. This point anchors the left side of the zero curve and is required for the calculation of the heat of fusion.
- Pre-eutectic activity is the measure of gas in the sample. Internal reduced pressure pulls the gas into bubbles early in solidification before stress-initiated shrinkage is formed so the endothermic arrests before eutectic are classified as gas related. Gas can be seen where turbulence aspirates air into the molten metal.
- Eutectic activity is the measure of micro-shrinkage occurring in the metal. This is sum of the endothermic arrests occurring between the eutectic and the start of solidus.
- Eutectic Acceleration is a feature not yet fully understood. It is a measure of how fast the eutectic recalescence starts and it varies greatly between the metal types of Gray, Ductile and CGI. It is the maximum slope leading into the eutectic undercooling.
- Eutectic deceleration is another feature not yet fully understood. It is a measure of how fast the eutectic reaction ends. It is the average slope of the recalescence or how fast the energy production is falling off.

Gray Iron Microanalysis© Information Available

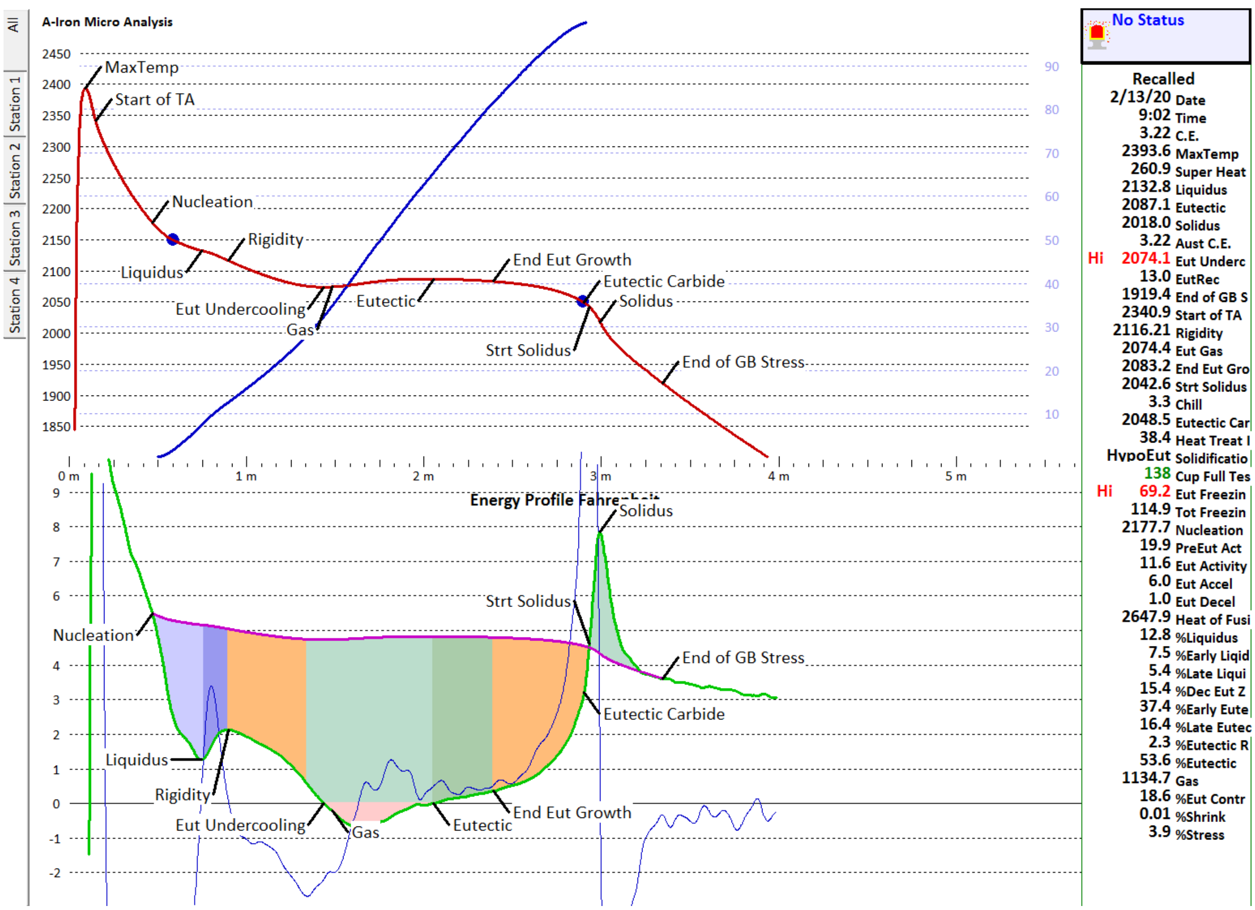
- Heat of Fusion is the total area between the zero curve and the rate of cooling. It is the sum of the distance between the two curves for each data point. Note: all area integrations are based on the background mathematical values and not based on screen pixels.
- % Liquidus is the area between the baseline curve and the rate of cooling from the Start of Nucleation to the Rigidity Point. It includes the Early Liquidus and the Late Liquidus.
- % Early Liquidus is the area between the baseline curve and the rate of cooling from the Start of Nucleation to the Liquidus Point. This is the energy from the initial growth of dendrites.
- % Late Liquidus is the area between the baseline curve and the rate of cooling from the Liquidus Point to the Rigidity Point. This energy covers dendritic thickening and any late secondary branching.
- % Graphite Liquidus is the area of the graphite liquidus if it occurs. Graphite is a very low energy producer and so a Graphite Liquidus is small. No austenite is being formed during this arrest which also means it is very low energy. But it has a strong and undesirable effect on shrinkage.
- % Pre-eutectic carbides are the carbides that form around the rigidity point from lower silicon irons commonly used in pump housings to mitigate wear. They form in thin sections leaving the thinker sections machinable.
- % Deceleration into Eutectic zone is the area between the baseline curve and the rate of cooling from the Rigidity point to the maximum acceleration into the eutectic point. During this zone carbon is concentrating to eutectic levels.
- % Early Eutectic Zone is the area between the baseline curve and the rate of cooling from the maximum acceleration point to the Eutectic Point. This is still a zone where most of the eutectic forms. Depending on the type of iron, and percent dendrites, some micro-shrinkage can form in this zone.
- % Late Eutectic Zone extends from the Eutectic Point to the End of Eutectic Growth Point. Here is where much of the micro-shrinkage occurs.
- % Eutectic Recalescence Zone is the energy released due to the previous undercooling. The less undercooling (better inoculation) the smaller this value will be.
- % Eutectic is the sum of the eutectic deceleration zone, the early eutectic zone and the late eutectic zone.
- % Gas is the energy absorbed in forming gas bubbles in the metal.
- % Eutectic Contraction zone starts with the point of maximum stress in the casting (End of Eutectic Energy Formation) and the start of Solidus. During this zone the hardness of the casting is determined, and macro shrinkage can occur.
- % Shrink (Macro) is the integrated % of energy that was absorbed by the various macro shrinkage arrests. These do not include micro-shrinkage or suck-in defects.
- % Stress is the endothermic energy absorbed by the grain boundaries as they solidify. For a given grade of iron, the larger this number, the less shrinkage will be found in the finished castings. Stress will always be present due to the reduction in volume that occurs from liquid form to solid form. It will be reduced by gas holes, micro-shrinkage, macro-shrinkage or by suck-in. Or it can be stored in the grain boundaries. As the casting cools from Solidus to Eutectoid temperatures, roughly 2,000 F to 1,400 F, additional graphite will form increasing the internal volume of the casting. This increase can tighten the grain boundaries if they are loose, or can cause mold wall movement if there is not enough looseness in the grain boundaries to absorb the expansion.

Gray Iron Microanalysis© Information Available

Summary

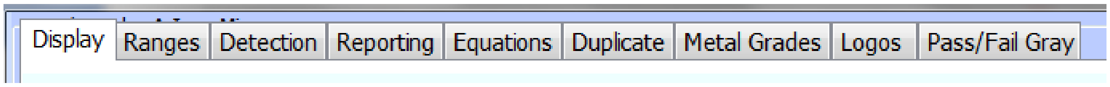
MeltLab is a tool to better control your metal and to look for causes for common and uncommon problems with your metal. In my early career as a plant metallurgist, there were several quality meetings where scrap was blamed on sand or metal and no one really knew where the problem lay. Today using this new tool, there are now answers to those quality problems. Not a lot has been published using this in-depth tool, but we are hopeful that researchers and quality people themselves will use this technology to make a better product. A good background in physical metallurgy will help with understanding what is important here. For those interested in diving deeper into some of these topics we will be glad to share our knowledge and work with you. Our next area of investigation will be in solid state transformations from the solidus point down to the eutectoid point where pearlite forms.

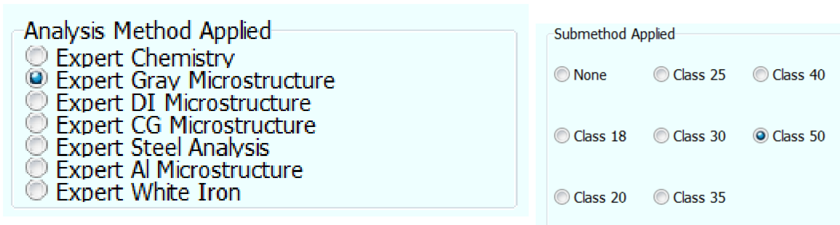
Full Screen with blue Fraction Solid curve, red Temperature curve, green Rate of Cooling and magenta baseline curve.



Gray Iron Microanalysis© Information Available

Curve options





Conclusion

MeltLab is a new reinvention of the cooling curve peering into the nucleation and growth of metals. It is more complete and more accurate than anything else. It has great potential in measuring the influence of inoculants, tramp elements, and impurities on the physical properties of strength, yield, elongation and hardness. And it measures the gas and shrinkage defects in castings. And it is simple to use. Once configured, the computer is only used to change metal grades. The rest is automatic.

David Sparkman
February 16, 2020