

# MICROSTRUCTURE BY THERMAL ANALYSIS

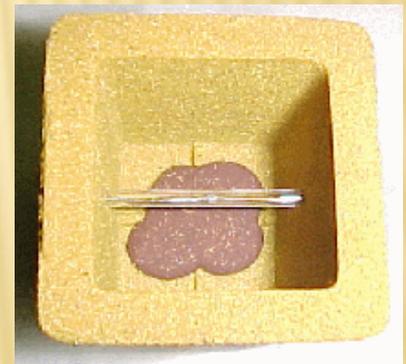
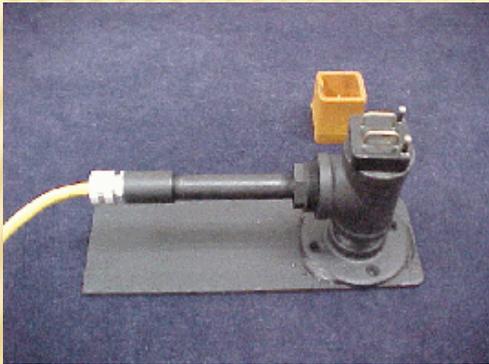
Quick answers for Chemistry, Inoculation and Microstructure

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2013

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# THE PROCESS

Molten metal is poured into a sampling cup containing a thermal couple. May contain tellurium or not.



Takes about 30 seconds of operator time, results ready in 2 to 6 minutes.  
Example cup contains tellurium for chemistry. Microstructure cups are plain and contain no tellurium.

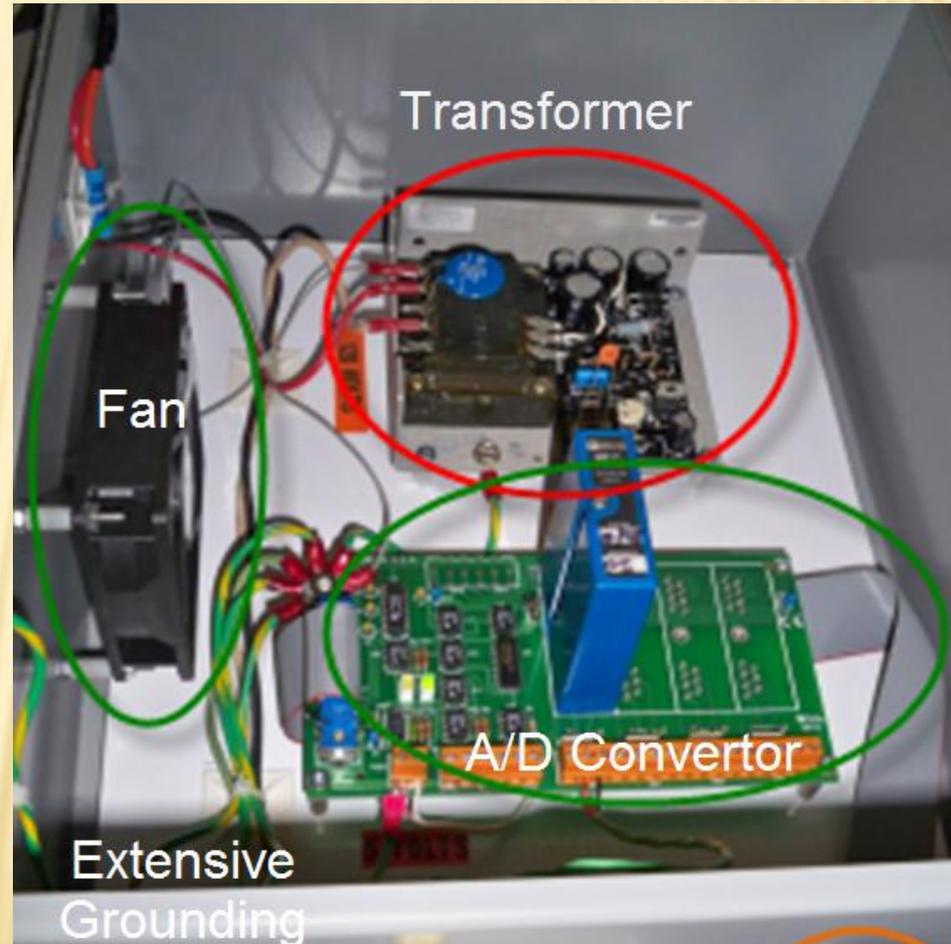
# ELECTRONICS SIMPLISTIC IS BETTER

Transformer

Fan

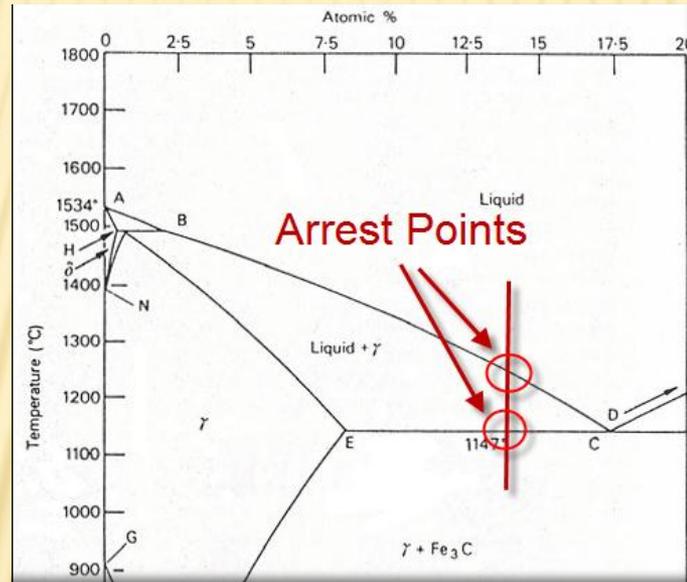
A/D Convertors (1..4)

Extensive Grounding



# THE THEORY

Phase diagrams measure the transition points from liquid to solid, from one structure to another.



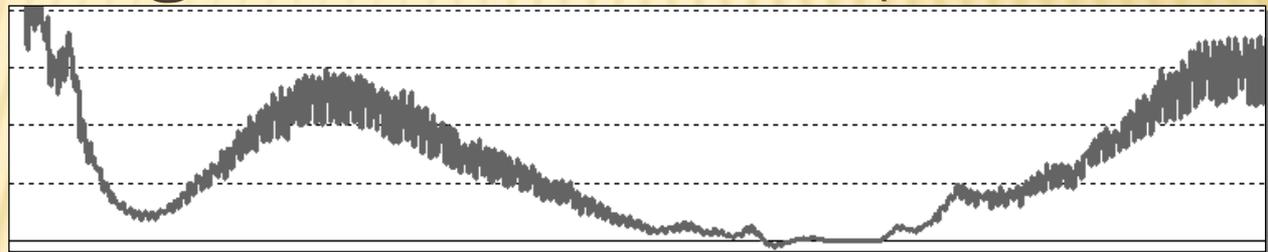
Iron-Carbon phase diagram

# IMPROVED DATA SMOOTHING

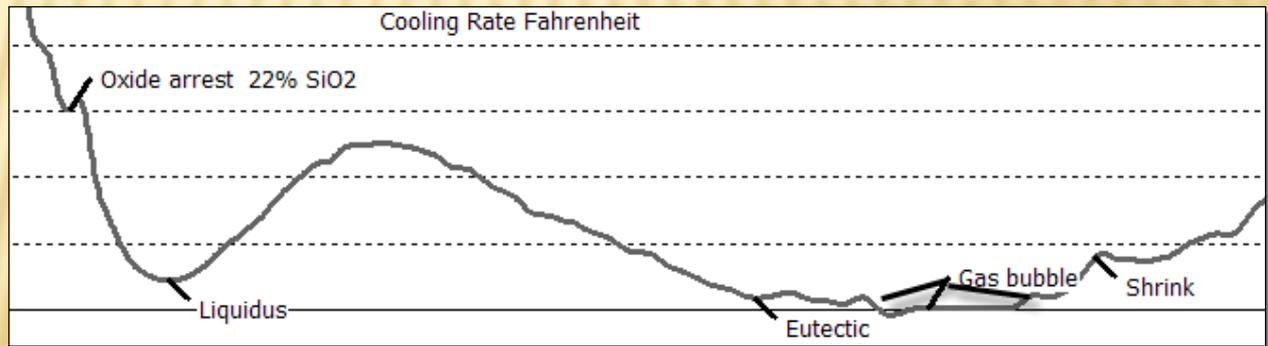
The key to this breakthrough was a new mathematical method of data smoothing.

Cooling Rate of white Iron (tellurium cup)

Before

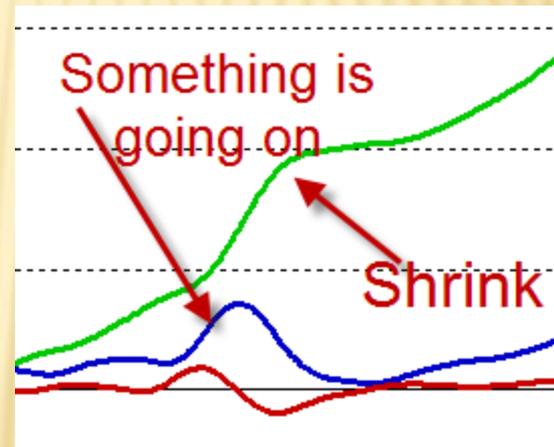


After



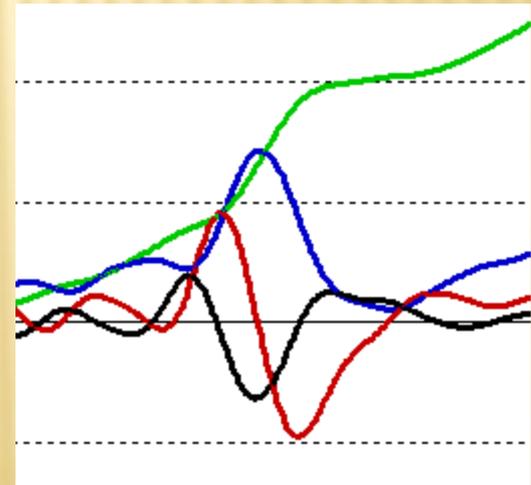
# DERIVATIVES DEFINE THE MICROSTRUCTURE

Here a shrink arrest in the green Rate of cooling curve triggers a fluctuation in the 2<sup>nd</sup> (blue) and 3<sup>rd</sup> (brown) derivatives due to the steep slope of the green curve.



# PUZZLING IT ALL OUT

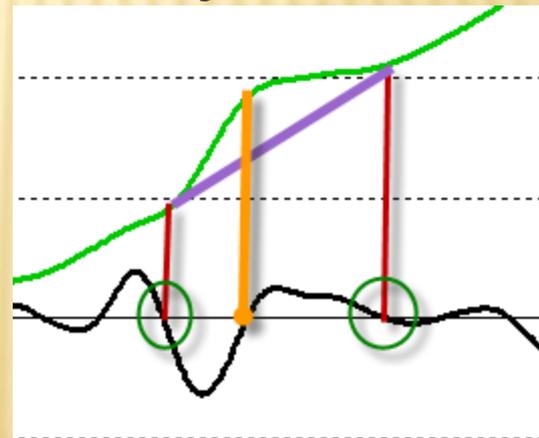
Here the brown 3<sup>rd</sup> passes downward through zero indicating a upward bend in the RoC. The blue 2<sup>nd</sup> is strong enough to warrant investigation. Back tracking we find a downward pass of the 4<sup>th</sup> to mark the beginning and then move forward to the second downward pass to find the end of the arrest.



# THE MAGIC OF THE 4<sup>TH</sup> DERIVATIVE

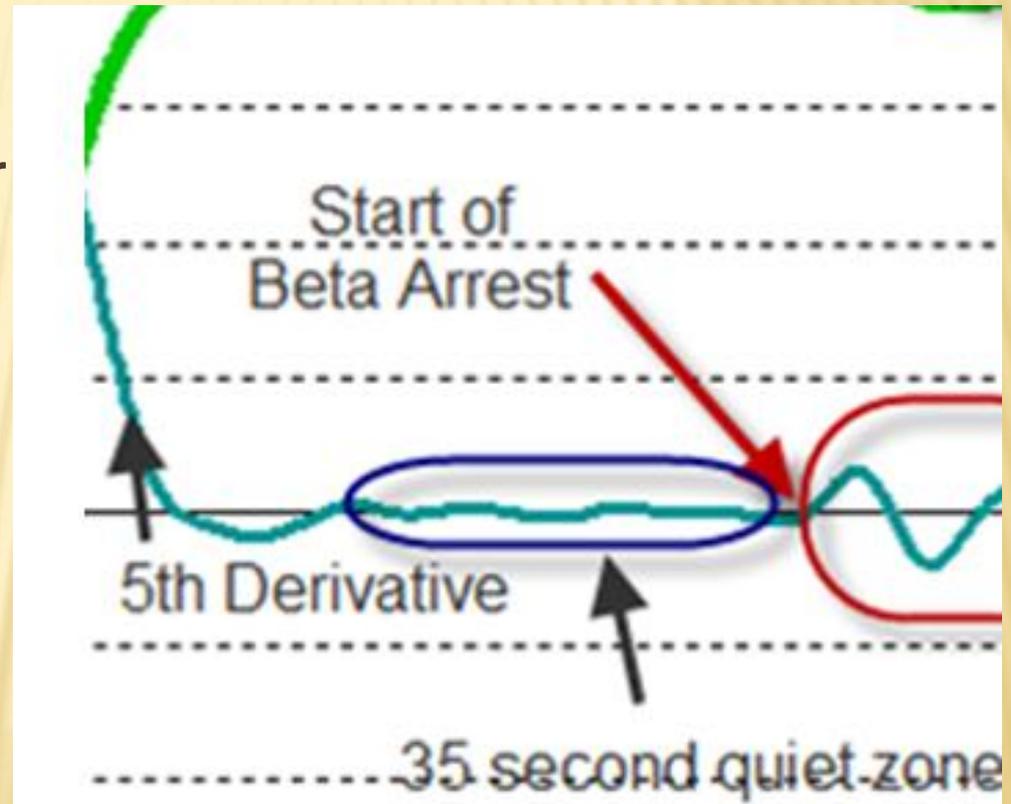
While the 2<sup>nd</sup> and 3<sup>rd</sup> derivatives indicate something is happening, the 4<sup>th</sup> derivative (black) gives the beginning and ending points of the shrink inflection as it passes downward through zero at both the beginning and the ending allowing us to draw a boundary line and integrate the area of the shrinkage.

The yellow line is the strong point of the shrinkage.



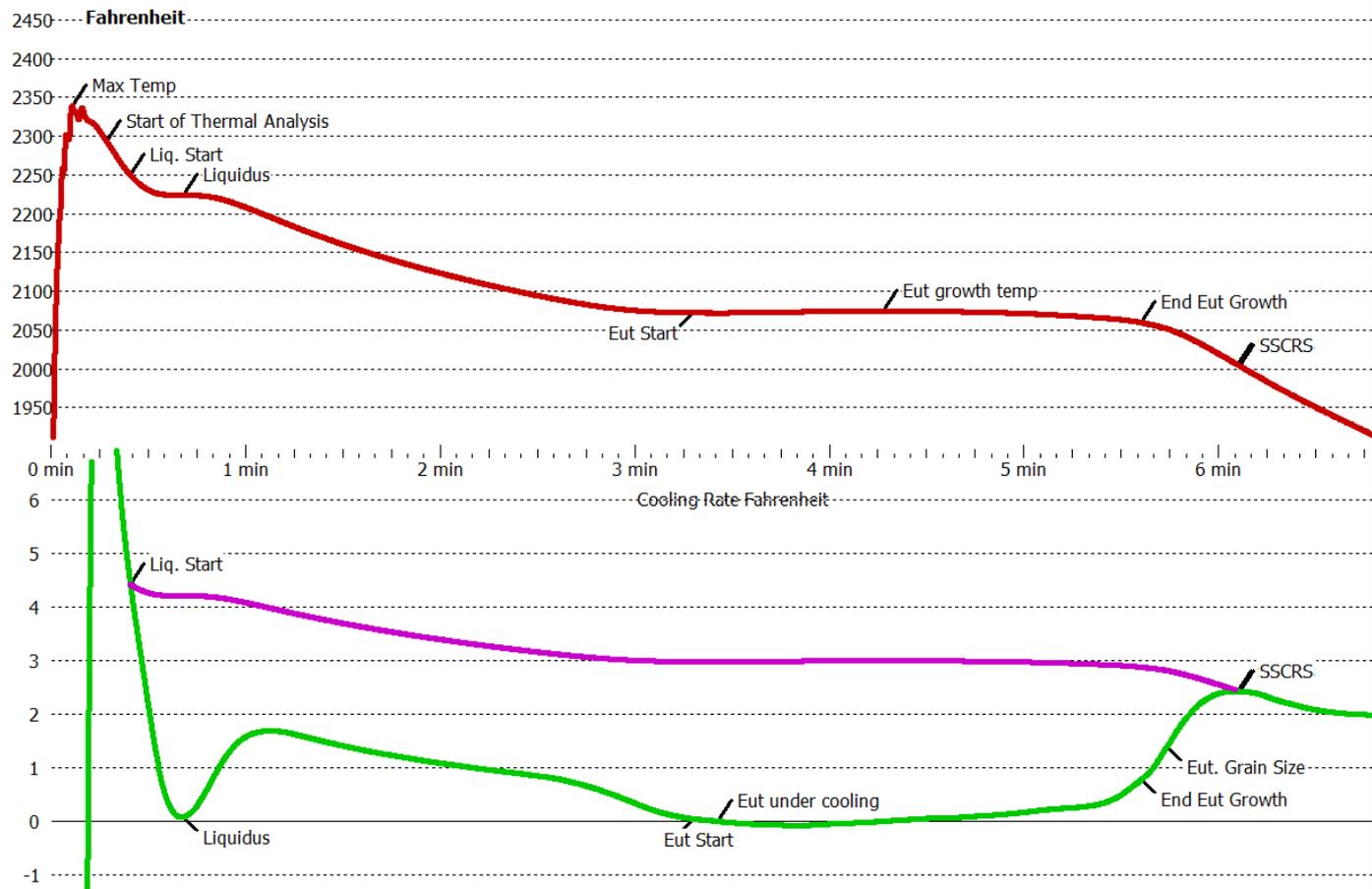
# STANDARD DEVIATION PROVES THE EVENT

Maximum inflection of 5<sup>th</sup> derivative is 5x the Standard Deviation of the derivative for the 10 seconds before the arrest.  
This is not noise!



# WHITE IRON GRAPHITE CONTROL

Stand 1 White Iron Analysis



**2224.0**Liquidus  
**2074.4**Eutectic  
**2005.2**Solidus  
**28.6**G1 secs  
**55.4**G2 secs  
**3.74**C.E.  
**1.9**G2/G1  
**66.0%** 2nd G  
**Hvpo**EutMode  
**115**Super Hea  
**2293**Start TA  
**2071.9**Eut UC  
**2074.4**Eut GT  
**2.4**Eut Rec  
  
**0.78**Activity  
**96**Nodulary  
**4**Nod Count  
**34**Sample Qu  
**97**Ferrite  
**0.051**Eut Cell Cn  
**28**Tramps

# WHITE IRON TESTING

Larger cup

Covered cup with ceramic fiber for longer cooling time

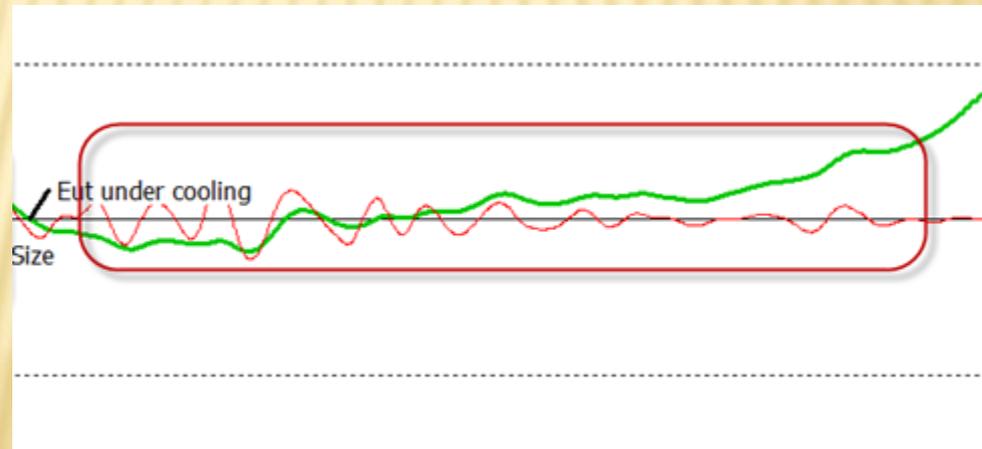
Compared base with inoculated iron by small additions in the cup.

Too much inoculation to be avoided.



# SEEING THE GROWTH OF GRAPHITE IN DUCTILE IRON

During the eutectic of Ductile iron the curve exhibits either a very smooth arrest or a bumpy arrest depending on the nodularity. We believe this is due to the different mode of growth of vermicular graphite from that of spheroidal graphite.



# NODULARITY INDICATORS

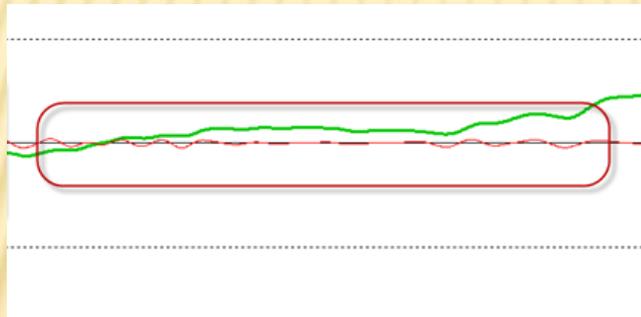
80%



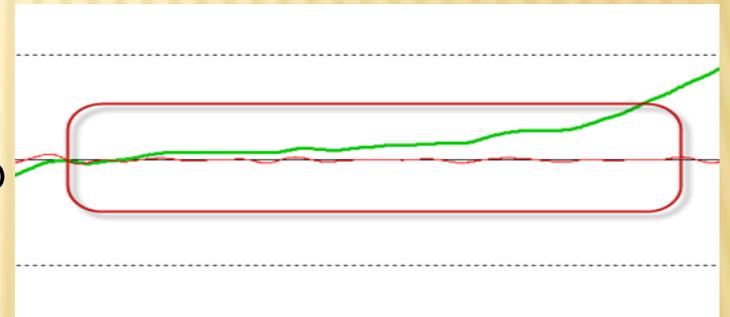
85%



90%



95%



The noise factor (5<sup>th</sup> derivative) of the eutectic arrest in DI over about 2 minutes for 80, 85, 90 and 95% nodularity. Noisier lines indicate lower nodularity.

# NODULARITY CALCULATION

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Nodularity =  $m * \text{Ln}(\text{StdDev}) + b$

Where Standard Deviation =

$\text{Sqrt}(\text{Sum}(\text{Sqr}(5^{\text{th}} \text{ Derivative}))/(\text{n}-1) )$

And  $n$  = number of data points

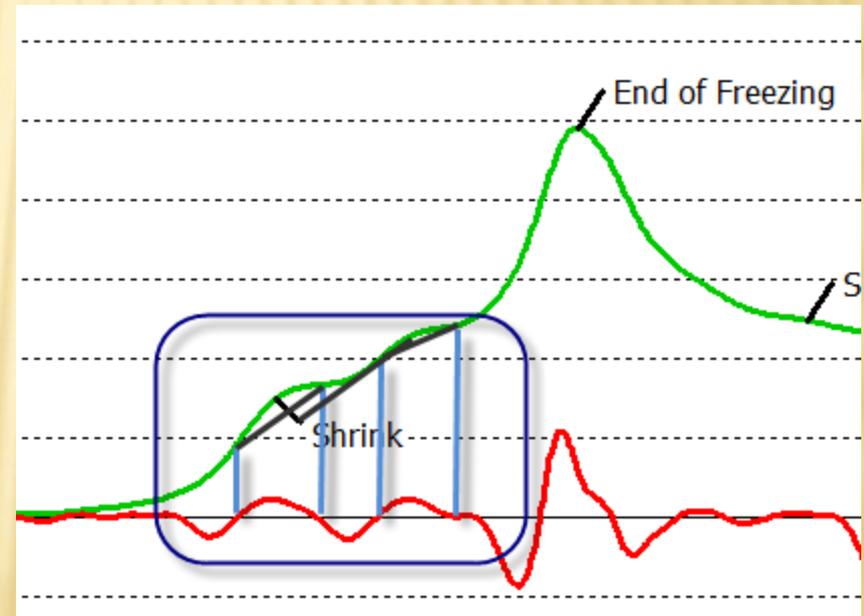
From our experience it seems to work well down to about 75% nodularity. But, it only works with good derivatives...

# ENDOTHERMIC (NEGATIVE) EVENTS

While phases form exothermic (heat producing) events, there are also endothermic (heat absorbing) events caused by gas or shrinkage.

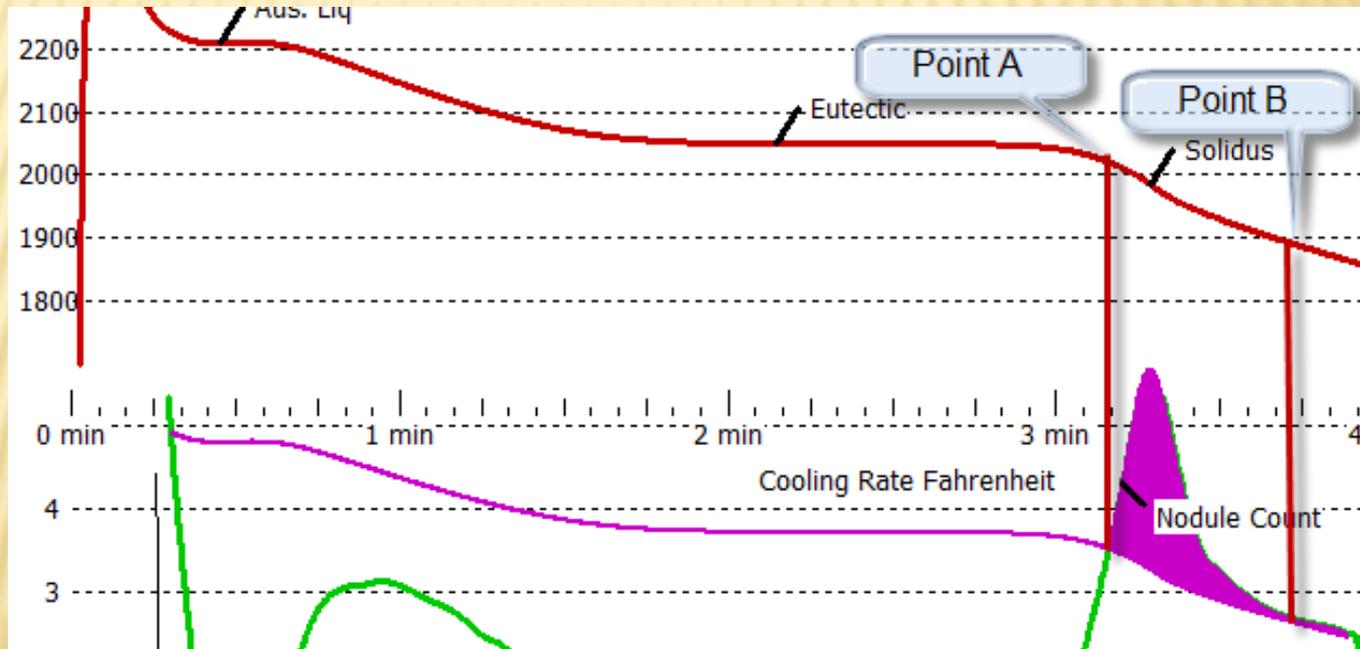
These too can be measured and quantified by higher order derivatives.

More on shrink later.



# MEASURING THE STRESS IN DUCTILE IRON

The endothermic (heat absorbing) arrest at the solidus arrest is an important indicator of shrinkage. The stress energy is the area above the zero curve and is inversely proportional to shrinkage.



# A NEW THEORY ON SHRINKAGE

The graphite formation in ductile iron is delayed with only about 40-50% graphite growth happening before Solidus.

This leaves a volume deficit in the casting that results in stress manifested in the grain boundaries. This stress is later relieved by further graphite growth.

# A NEW THEORY ON SHRINKAGE - 2

Shrinkage or suck-in occurs when this stress is thermally concentrated in one area of the casting, or when a nucleate to shrinkage occurs such as gas or when there has been premature graphite growth in the liquid (graphite liquidus).

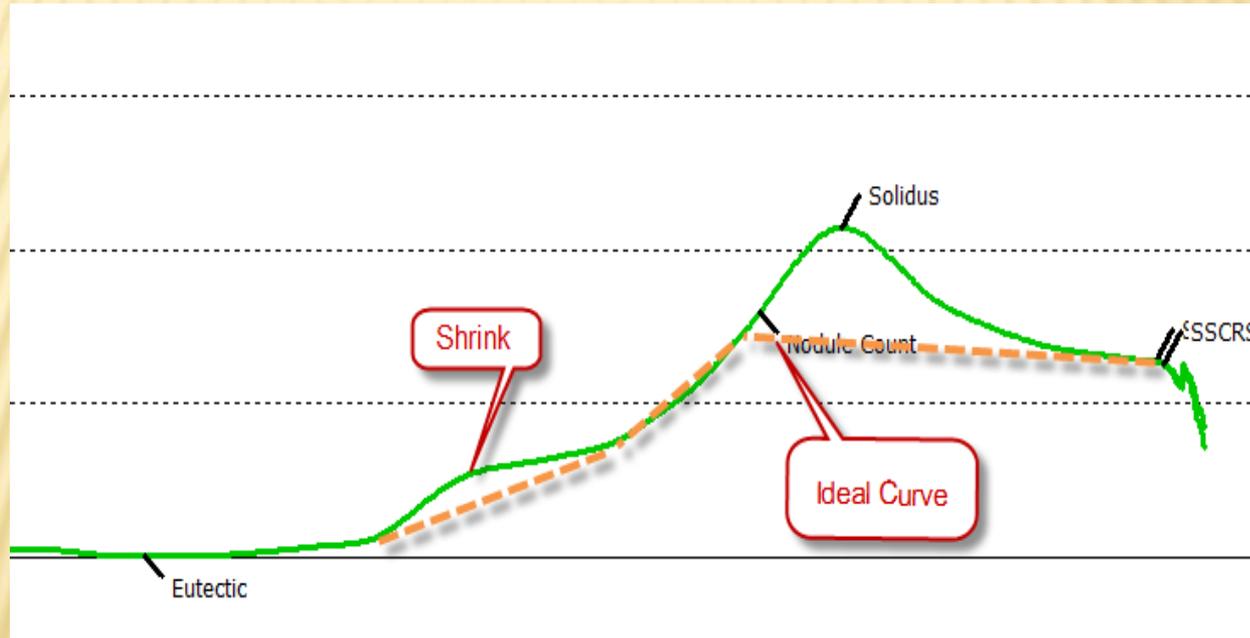
A Graphite liquidus is caused by a C.E. over 4.6 and results in bimodal graphite distributions and solid risers.

# THERMAL ANALYSIS AND SHRINKAGE

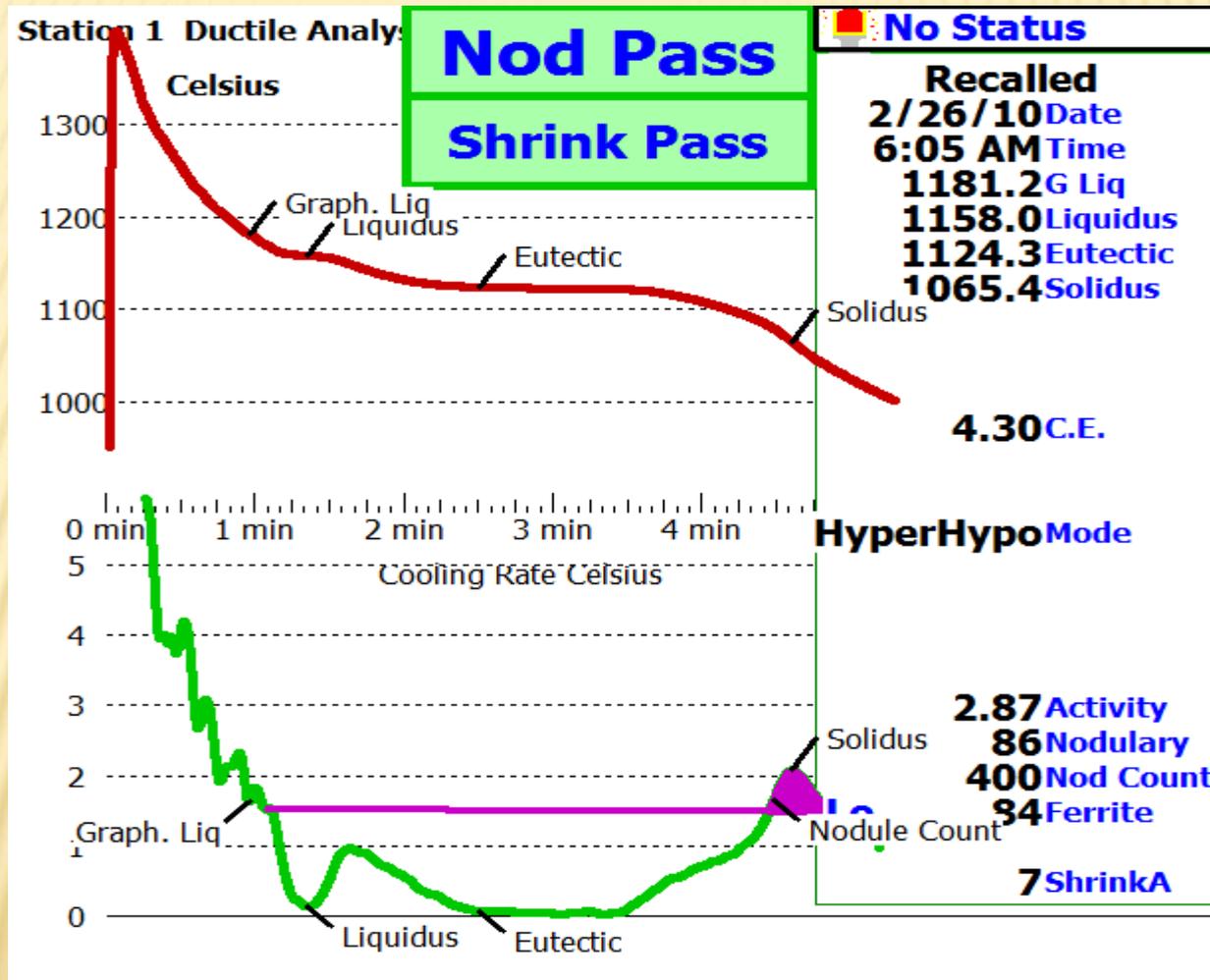
Thermal Analysis can detect graphite liquidus arrests and measure the degree of stress in the solidus arrest giving a predictor of shrinkage problems.

# SHRINKAGE REDUCES THE STRESS

Here an actual shrinkage arrest has reduced the stress at solidus.



# IRON MICROSTRUCTURE FOR DUCTILE



# DUCTILE IRON MICROSTRUCTURE CONTROL

Gating – Gates should feed until solidification starts, then should close off to capture graphite expansion within casting.

Chemistry – determines if graphite grows in liquid (hyper-eutectic), if dendrites block gates (hypo-eutectic) or if eutectic freezing mode (thin walls with possible suck-in).

# DUCTILE IRON MICROSTRUCTURE CONTROL 2

Inoculation – early or late graphite, nodule size, excess magnesium, pearlite, carbides. Depending on section size, the degree of inoculation and kind of inoculation needs to be varied.

Interaction of all three – results in either stress or shrinkage.

# TUESDAY AM WHITE IRON ANALYSIS

