Thermal Analysis of Aluminum
Detecting and interpreting solidification

David Sparkman – MeltLab Systems, August 14, 2018

Making a good casting requires that the metal solidifies to the specifications of the end customer. Shape, strength, stiffness or flexibility, and good wear resistance are needed, with no significant voids and hard spots that would cause machining problems. If you could see the atoms solidifying, you could watch these various structures form. Thermal Analysis tracks the heat signatures of these various structures and can see the larger structures form, comparable to a magnification of about 500X with a reliability often better than a microscopic analysis. Further techniques of integration of tiny multiple events can even estimate the overall amount of gas and porosity present in the metal.

This is done by examining a sample of the metal as it freezes in a small container with a thermocouple. Crude systems of the past could not overcome the prevailing electrical and magnetic noise that is typical of manufacturing and were limited to just finding major arrests. However, by filtering and stabilizing the incoming electricity and then by using advanced signal processing software, crystalization features as small as 0.02% can be found with a 95% or better confidence level. This exceeds the ability of standard optical microscopes.

Sampling is important. It has to be adapted to manufacturing conditions of simplicity and reliability. But still, there is some skill involved. In 28 years of development, the sampling stand has evolved into a precision tool. All that is required is that the temperature loss on transfer be minimized and that the sample cup be filled close to the top for consistency.

Results and data interpretation are offered for either go/no-go interpretation by shop floor personnel or as specific values for the engineer/scientist who may be fine tuning the process to maximize properties and minimize costs. The objective values may vary with alloy and even with the product or casting being produced. The system allows for up to 100 different alloys or recipes. You could actually have different objectives based on casting thickness for the same alloy.

The following is a list of the various detections included in the program at the highest 'Expert' level. Some only apply to certain alloys, and some may not be present in all versions of an alloy. The system visually presents the temperature, rate of cooling and the derivatives down through the 5^{th}. Areas of various phases are measured and ratioed against the overall heat of solidification also known as the heat of fusion.

Pre-Liquidus points: These are called maximum temperature, start of thermal analysis, sludge arrests, Chinese script (Alpha Phase) and nucleation point. The maximum temperature point determines if the sample was hot enough to pick up the remaining analysis points. The start of TA is where the temperature change evens out and the thermocouple starts accurately tracking the metal temperature. Some small amount of superheat is required for this. The nucleation point is where crystallization of the main component of the metal begins and anchors the high temperature point of the base line curve. Some crystals happen before this but they are from sludge elements or from alpha crystals.

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Liquidus points: These are undercooling, growth temperature, liquidus point, recalescence, strength, grain size, coherency, and secondary branching. With grain refinement, undercooling of the liquidus may be prevented. Recalescence shows the lack of grain refinement and the degree of large, oversized dendrites forming. The degree of grain refinement is determined by the strength of the energy production of the liquidus arrest. The speed of coming out of the liquidus is determined by the number of cells or dendrites if hypoeutectic. This is measured by the grain refinement number and works with hypo and hyper alloys that use either TiB or CuP for refinement. The coherency point is where in hypo-eutectic alloys the dendrites begin to run together and dendrite growth slows. The metal then takes on a mushy form.

Between Liquidus and Eutectic: Here we find the rigidity point, end of liquidus/start of eutectic, beta crystals, delta crystals, hydrogen gas bubble formation. The Rigidity point is where the eutectic material begins to form and (in the case of hypoeutectic alloys) begins to cement together the dendrites with eutectic material. This is the same as the end of liquidus and start of the eutectic boundary. Before that, beta crystals and even delta crystals will form if enough iron remains in the liquid. It is during the liquidus formation that the casting volume begins to shrink, depressurizing the internal liquid and drawing out the hydrogen gas in the form of tiny bubbles. While the bubbles themselves are too small to detect, they add to the background noise and can be estimated by the amount of this noise as measured by high order derivatives.

Eutectic points: Next we see several more items of interest: undercooling, speed of recalescence, growth temperature, eutectic point, eutectic recalescence, modification, end of eutectic energy production, porosity and shrinkage. Modification increases the undercooling of the eutectic. The speed of recalescence is not well understood yet, but appears to increase as modification effects age. The growth temperature is the same as the eutectic point but is used in the calculation of eutectic recalescence. Modification is based on Geoffrey Sigworth's latest research and measures the energy of modification. The end of eutectic energy production is the divider between porosity (small) and shrinkage (large) energy production. Porosity is determined from background signals in higher order derivatives, whereas shrinkages are large events and are clearly visible on the derivative cures as individual events.

Solidus Point: This is the end of the change from liquid to solid, where the grain boundaries finally freeze off. It is the low temperature anchor point of the baseline curve.

Aluminum Alloy specific inflection points: These include Beta Crystals, Delta Crystals, Copper ($AlCu_2$), Mag-Silicide, Blocky Copper, Nickel. Beta and Delta crystals are iron-bearing, needle-like structures that occur in some alloys when the iron content exceeds a certain level not neutralized by manganese. Manganese ties up iron as the Alpha phase that is blocky and not harmful to fatigue life in contrast to Beta and Delta which are. Blocky copper occurs at a lower temperature than the copper phase and is more difficult to heat treat. Nickel and other metals can produce additional phases for specialty alloys.

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Phase percentages: In most alloys, identifying the presence of a phase does not give enough information. The amount of the phase is needed for quality control. Does the metal require more or less grain refinement? Has the modification aged to the point of needing renewal, or is it over-modified and causing feeding problems? Has too much of the magnesium burned out of the Aluminum? Did the degassing operation function properly? Is the iron level too high? Making high quality castings requires this knowledge for every batch of metal. And if the metal is held too long, it may need retesting.

A common cause of problems is the mixture of new alloy and returns as mixing different alloys can cause problems that need to be detected and measured. But even mixing the same alloy causes different levels of oxides, grain refinement and modification levels, which can produce an uncertain result. Knowing that the modification and grain refinement is in range allows more consistent castings. And the introduction of oxides from returns must be kept under control to prevent too much porosity. Here are the Percent readings measured:

Percent Measurements – Expert Level only: Beta%, Copper%, Mg-Silicide%, Blocky Copper%, Pre-Liquidus%, Post Liquidus% (Dendrite thickening), Eutectic%, Modification%, Shrinkage%. The area of the various arrests and phases are ratioed against the total energy to give a percent energy. These values can then be used to set standards for the different alloys being produced.

Other Measurements – Expert Level only: Gas factor, Porosity factor. These can again be used to control the degassing and the blending of returns with new metal.

Different levels of Analysis

- Basic Analysis: This measures only the major arrests and recalescence. This is similar to the early thermal analysis systems, and is the lowest cost model.
- Advanced Analysis: This includes Basic, but also measures most pertinent arrests as a point or a temperature.
- Expert Analysis: This includes the two above analysis methods, and includes high level calculations of percentages, gas and porosity factors.

System hardware: The system consists of a PC type computer (customer provided), software, an industrial hardened analog to digital converter box, and an amazing sampling stand. Cables connect all three. The converter box electronics are warrantied for 10 years against manufacturing defects. Specify the AC voltage to be used (120, 220, or 240). The system is network-compatible and generates CSV result files to a local or network location.

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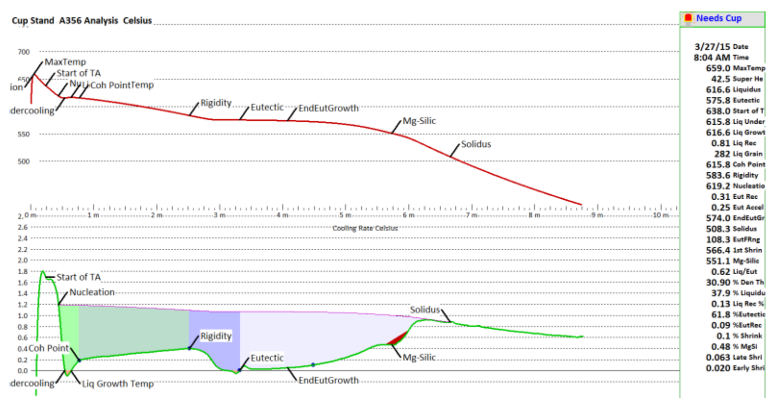

Stand with sample cup, Converter box, sample A356 curve with all results