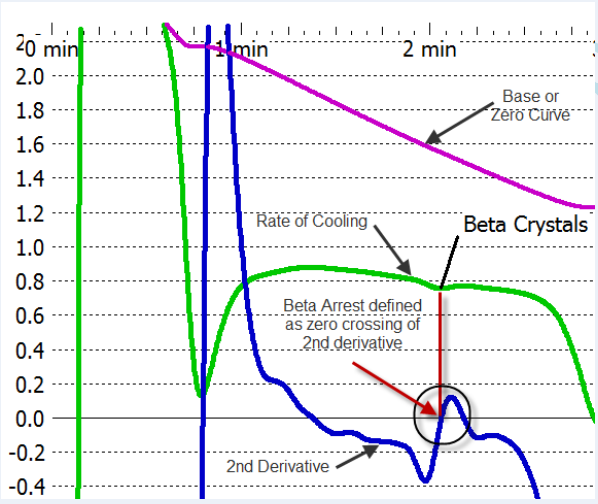
Measuring Microscopic phases using Aluminum Beta Crystals as an example

by David Sparkman July 5nd 2010 all rights reserved

This month's "Hot Topic" is on microscopic events in solidification, using aluminum "Beta" crystals as a benchmark. We will look at the energy signal of an aluminum alloy with 0.5% iron and then compare the background noise to the beta crystal signal and the overall energy, to come up with a confidence level for micro-events.

All production quality castings have problems with contamination and phases to be avoided or minimized. In iron, carbides are generally kept to a minimum except in certain "white" irons. In Aluminum, the beta phase is the crystal phase to be avoided. With enhanced thermal analysis, it is now possible to not only see and detect these phases, but also to measure their energy and estimate their volume.

This "enhanced" form of thermal analysis makes use of several noise suppression techniques and a 16 bit industrial grade analog-to-digital converter. Please don't try this with a 12 bit converter: it doesn't have the resolution. And without shielding, filtering, and the proper smoothing techniques, you won't be able to do what we have done here.



To the left is an example of a beta crystal arrest in an aluminum alloy. The arrest is defined as the positive zero crossing of the second derivative. (The blue arrest line passes through zero in a positive direction.) The arrest typically occurs between liquidus and eutectic in aluminum alloys and has the composition of Al_5FeSi known as "Beta Crystals". These crystals are commonly a result of iron contamination in remelted scrap. The crystals are seen as elongated needles in the microstructure. Their effect is to lower physical properties and have the worst effect on fatigue life.

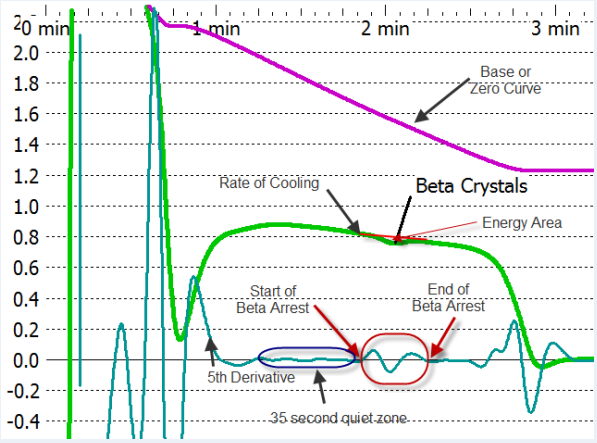
As Lennard Bäckerud describes beta crystals: "*It is well known that high iron content is responsible for inferior mechanical properties of Al-Si alloy. This is mainly due to precipitation of Al_5FeSi. This phase, like most intermetallic particles, is brittle. It's {Its} morphology is plate-like, with extensions up to several mm (appears as needles in micrographs). These two characteristics together decrease the strength and ductibility {ductility} of the cast products.*"[1]

With the detection of the Beta arrest defined as

```
If (AdTemp  <= BetaRngHigh)
and (AdTemp  >= BetaRngLow)
```

and (AdType = Ad2Pos)
then {an arrest has been found}

the next step is to measure the strength of the arrest. First we need to determine the beginning and ending points on the rate of cooling curve, then measure the area that the cooling curve was deflected by the crystalline growth.



Looking at the 5th derivative to the right (cyan curve), it becomes obvious that the derivative has been bouncing around zero up till the point where the first deflection in the green curve occurs. So the start of the beta arrest is simply the last positive zero crossing before the beta arrest. Likewise the end of the beta arrest is the first negative zero crossing after the beta arrest. We can further test the validity of the arrest by taking the standard deviation of the 5th derivative for almost 35 seconds before the arrest and compare it with the height or depth of the maximum/minimum value of the 5th derivative during the arrest. For practical reasons, we just take the 100 readings before the arrest (about 11 seconds) to calculate a normal expected variation of the derivative. In this case the standard deviation for the 5th derivative was 4.450E-05, and the greatest deviation for the 5th derivative during the arrest was 2.274E-4 for the max/minimum variation during the arrest giving a standard deviation during the arrest of 5.1 sigma. Clearly this is a real event and not noise. In some cases, where there is an arrest immediately before the beta phase, it might be better to take the noise measurement after the arrest. The computer can do both and take the lesser of the two.

The final step is to draw a line between the start and stop, and figure out the area of the inflection. Given two points, we solve for a straight line equation y=mx +b. Then for each data point between the start and stop we calculate the difference between the straight line and the measured rate of cooling. This value is then divided by the area between the base line or zero curve and the rate of cooling curve or

Beta Arrest Energy / Total Energy

The result is then reported as a percent of the total energy.

The obvious omission in this mathematics is the specific heat of the alloy verses the specific heat of the beta phase. The problem is that these specific heats are unknown at the present time. Our solution is to provide a ratio factor where the customer can compare the MeltLab percentage of energy to what is found under the microscope and enter in a correction factor for specific heat. Since these ratios should remain consistent for any alloy class, we can move the science of thermal analysis forward.

The following configuration is necessary to “teach” the MeltLab to find beta crystals:

1. The temperature range for beta crystals in this alloy (depends on silicon level).
2. The minimum degree of certainty required to call it a real arrest.
3. The specific correction factor (defaults to 1.0 for no correction).

4. The minimum amount of beta phase to be concerned about: i.e. don't report beta phase if calculated amount is less than xxx.

And of course you may pick the following items to display concerning beta phase arrests:

1. Temperature
2. Energy of beta phase
3. Energy ratio of beta phase to total energy (uncorrected volume estimate).
4. Estimated beta phase volume (corrected).

A common question is: what is the threshold for phase detection. The truthful answer is that we don't know. We once found an arrest as low as 0.03% of energy, but with the latest versions of MeltLab, we may be able to go lower than that.

Date	Time	Temp	% Energy Area	Noise	Sigma
5/4/2010	11:05 AM	331.6	0.144	2.250E-05	11.4
5/3/2010	11:52 AM	331.5	0.143	4.450E-05	5.1
4/30/2010	2:01 PM	333.5	0.270	4.640E-05	14.1
4/28/2010	11:15 AM	333.9	0.105	4.510E-05	9.9
4/23/2010	11:12 AM	330.9	0.148	4.990E-05	7.8
4/22/2010	12:04 PM	329.5	0.182	9.310E-05	5.2
4/20/2010	5:12 PM	331.8	0.297	5.180E-05	5.1
4/16/2010	10:05 AM	331.2	0.245	4.260E-05	15.8
4/13/2010	12:20 PM	328.4	0.229	6.970E-05	10.2
4/12/2010	10:48 AM	332.0	0.252	3.250E-05	4.5
4/8/2010	10:47 AM	338.9	0.031	6.610E-05	3.9
4/7/2010	2:58 PM	328.0	0.100	5.190E-05	9.1
4/6/2010	12:35 PM	339.2	0.410	3.770E-05	5.5
4/5/2010	9:13 AM	326.3	0.123	2.240E-05	6.4
4/2/2010	12:48 PM	324.8	0.305	2.363E-04	4.1

Table 1 Results of multiple tests

The two tests on the 11th of May indicate some of the repeatability. I wouldn't worry about the differences in Sigma. Five-sigma is already overkill. But the temperatures and the energy area repeated nicely. Please note that the customer providing this data uses their aluminum for a unique process that is not affected by the presence of beta crystals. Their chemistry, though high in silicon, has a typical iron content of 0.5%, similar to A319-1. Taking the average energy percent area verses the average iron content; we can derive a correction factor of 2.5. Remember that the beta phase crystal is only partly iron, so the correction factor shows that the crystals produce about 2 ½ times the volume that would just be accounted for by just the iron. It is then possible to reverse calculate the iron content of the metal. Some variation could be expected due to the manganese content tying up some of the iron. Copper and magnesium have similar effects but the technique can still be useful when those elements are stable.

Next month, we will look at another aluminum solution: measuring copper and magnesium phases in 319 and 356 alloys. If you have any questions or would like to direct us to discuss a certain topic, please drop me a message at david@meltlab.com.

Meanwhile, we have a summer sale going on through the end of August on all MeltLabs. Click here for more information www.meltlab.com. We are less expensive than the competition, *and* you get all the "bells and whistles".

[1] L. Bäckerud "Solidification Characteristics of Aluminum Alloys volume 2, Foundry Alloys", pg 72 by AFS/Skanaluminum 1990.