MATC(MN)08

Thermal Properties of Light Metal Alloys by Differential Scanning Calorimetry (DSC)

Summary

Light metal alloys are used in many applications (e.g engine blocks) exploiting their mechanical properties and processability. The design engineer, who uses computer simulation of industrial foundry processes, needs to know the thermal properties of the alloy, principally the specific heat. With this information the cost of processing and the number of rejects can be reduced along with an improvement in quality. Thus there is an increasing need to determine the heat flow properties of low melting alloys over a wide temperature range which can be difficult to measure with very high temperature calorimeters. However, because of the relatively high temperatures involved and the alloying of the test metal (an aluminium alloy) with components of the measuring device (typically a DSC machine) these practical difficulties need to be carefully addressed. Nevertheless the versatility of the DSC and the speed of test render this technique worth adapting to measure these materials. This study describes a system whereby the alloy may be tested and the thermal properties, such as the latent heat of fusion and specific heat may be safely obtained, without alloying with the test apparatus.

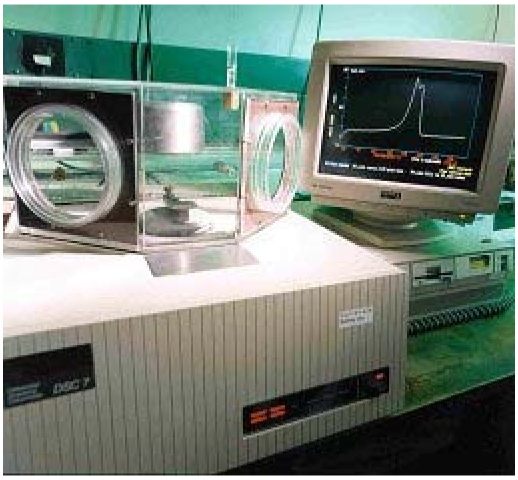
Rover Car Engine Block

C B Hobbs

May 2001

The Differential Scanning Calorimetry Method

The calorimeter (a Perkin Elmer DSC7) measures the heat flow in or out of a sample in a suitable receptacle (normally an aluminium pan system) compared to an identical empty receptacle as a function of temperature (or time). In this study the test sample (an aluminium alloy) requires heating to a high temperature (725°C) and at this temperature it can take part in an alloying reaction. The use of normal aluminium pans, which can react at 600°C, is therefore prohibited. The need for a different arrangement whereby the sample is not in contact with alloying materials is clearly apparent.



DSC with computer for temperature programming and thermal analysis

The purge gas over the sample was changed to argon, from the traditional nitrogen (avoiding possibly nitride formation) which may contain a small amount of oxygen, thus removing the risk of oxidation effects at elevated temperature.

The usual pans are made from aluminium, which melts at 660°C, so were replaced with platinum crucibles. To avoid contact of the alloy (typically aluminium) with the platinum the sample was sandwiched between thin sapphire discs. A platinum lid was then placed on the top to give the reference pan and the sample pan the same emissivity, essential for accurate analysis. This combination is used for all testing during the baseline, temperature calibration, sapphire standard and sample experiments. The DSC temperature calibration is carried out with indium and zinc using the sapphire platinum combination in both the sample and reference pans. Great care has to be exercised to avoid the sample touching the sides of the platinum crucible, as on melting the alloy tends to wick due to surface tension effects.

The response of aluminium alloys to heating depends on the thermal history of the sample. It is thus necessary to heat the sample well into the liquid state and wait until temperature has equilibrated. The analysis can then be carried out at a linear cooling rate of 10°C/minute. The software can then subtract the empty pan experiment from the sample experiment and derive the specific heat of the sample as a function of temperature. The software also allows the operator to draw a line underneath the heat flow curves to calculate the latent heat of fusion. (This baseline fitting is thought to be the largest source of error in the measurement).

Results

The DSC analysis for a low melting aluminium alloy is shown in Figure 1. This is a plot of the change in heat flow with temperature on cooling using the procedure outlined above.

Figure 2 shows the variation in specific heat with temperature for the same alloy as above cooled at 10°C/min.

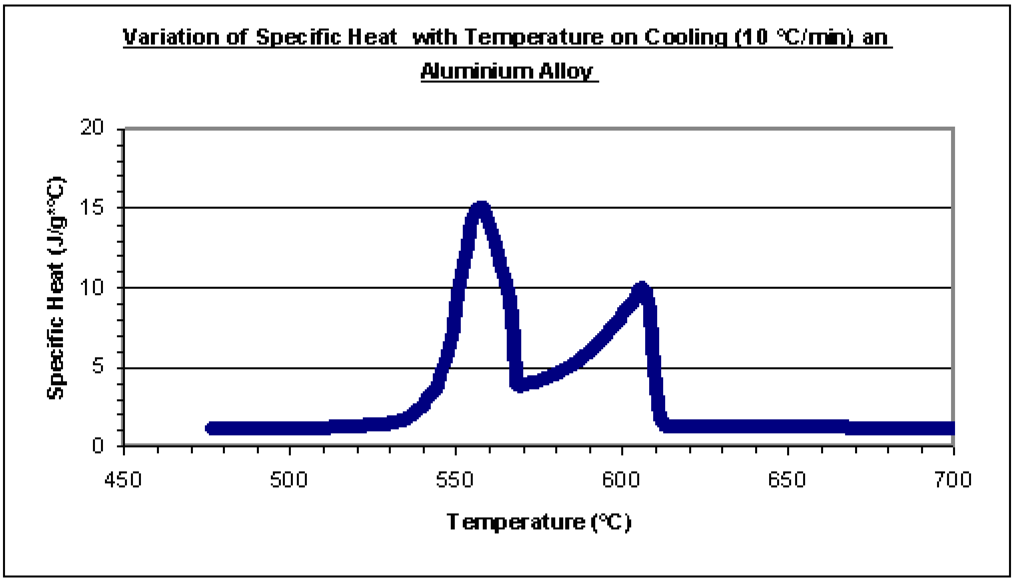


Figure 1: An example of the change in heat flow for a light metal alloy cooled from 700°C

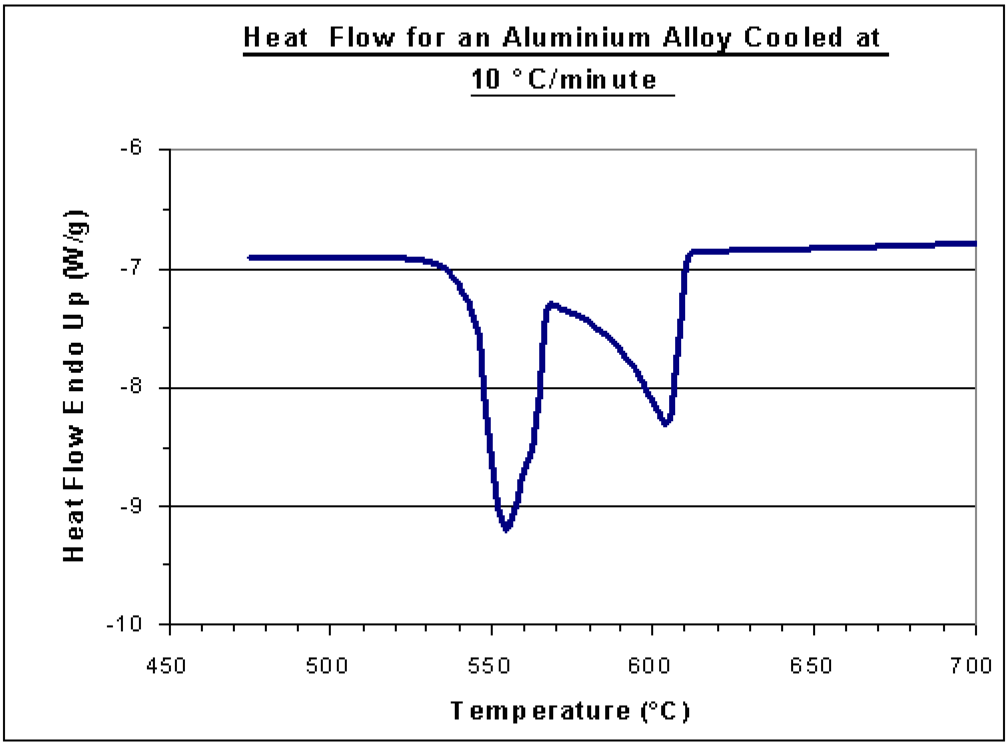


Figure 2: An example of the variation in specific heat for a light metal alloy cooled from 700°C

Conclusions

This DSC set up and procedure widens the scope of the DSC and enables materials such as light metal alloys to be safely tested without affecting the function of the DSC apparatus.

The data will benefit design engineers in both processing and industrial use thus reducing costs to the processor and end user.

The NPL Facilities

The NPL has state of the art thermal analysis instruments of all types including dual cell, pressure, high temperature DSC and the most recent advance: modulated DSC. With this extensive range of facilities accurate measurements can be made on a wide range of materials over an extensive temperature range.

To obtain quantitative and accurate data all the instruments are calibrated over the working range with reference materials, where available (ref 1) and the tests carried out to ISO 9001 procedures and ISO/FDIS 11357 standards (ref 2).

The NPL is unique in offering to industry a complete DSC service over a wide range of materials from -170°C to 1570°C.

DSC Instrument	**Temperature range (°C)**	**Typical Test Materials**	**Typical Purpose**
Power compensated	-170 to (+730)	Polymers, foods, ceramics, metals, oils	T_m, T_g, enthalpy specific heat, crystallisation and reaction kinetics
Temperature Modulated, (heat flux)	-60 to (+300)	Composites thermosets semi-crystalline polymers	T_m, T_g, complex overlapping transitions
Pressure, (heat flux)	ambient to (+725)	Polymers	Heat of reaction, oxidative stability, vapour pressure
Heat flux, (DTSC)	600 to (+1500) 1100 to (+1570	Metals, alloys, ceramics	specific heat, enthalpy, reaction kinetics

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References

1. Reference materials supplied by the Laboratory of the Government Chemist
2. ISO/FDIS 11357 - Part 2 and Part 4 in draft.

The NPL conducts underpinning research on measurement methods relevant to all aspects of industrial materials.

For further information on DSC or to find out more about NPL measurement services:

Visit: http://www.npl.co.uk/npl/measurement

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