Concordia University MECH 6661

Age Hardening, Electrical Resistivity and Thermal analysis (Differential Scanning Calorimetry)

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Agenda

0 Objective

0 Background

- Age Hardening
- Electrical Resistivity
- O Differential Scanning Calorimetry
 - Principles
 - Applications and Examples
 - Preliminary Results

o Conclusions

Objective

The objective of the present project is to introduce the DSC technique as a complementary tool for the study of precipitation behaviour of different aluminum and magnesium alloys through electrical resistivity measurements.

Background – Age Hardening

The enhancement of mechanical properties can be achieved through the process known as precipitation hardening.

Precipitation or age hardening \rightarrow Heat treatment technique which allows an increase of the yield strength of the material by the formation of precipitates. It involves three fundamental steps:

• Solution heat treatment

- o Quenching
- o Aging



There are many methods used to measure and determine different properties in a material (matter transitions, chemical composition, physical and mechanical properties, etc.).

Electrical resistivity has been used to observe the aging phenomena in different systems.

It indicates how strongly a material opposes the flow of electric current. It is independent of the specimen geometry and related to the resistance of the material through which the current passes. The units are ohm-meters ($\Omega \cdot m$).

In terms of the applied voltage V and the current I, the electrical resistivity can be expressed as:

 $\rho = \frac{VA}{Il}$

The total resistivity of a metal is the sum of different parameters: impurities, thermal vibrations and plastic deformation.

Many experiments show that the resistivity rises with the increase of temperature. This behaviour is due to the increase in thermal vibrations and lattice irregularities, which serve as electron-scattering centers. A similar behaviour is observed during the addition of impurities (i.e. aging treatment). In this case the precipitates act as scattering centers and increasing their concentration results in an enhancement of resistivity. Also, the resistivity increases nearly linearly with concentration of the alloying elements in solid solution.

According to Matthiessen's rule, the electrical resistivity has the following form:

$$\rho(T, c_1, \dots, c_n) = \rho(T) + \sum_i c'_i \rho'_i + \sum_k c''_k \rho''_k$$

where

= temperature dependent resistivity of the metal matrix.

 $\sum_{i} c'_{i} \rho'_{i} = \text{resistivity contribution from the alloying elements with a concentration } c_{i} \text{ in solid}$ solution.

 $\sum_{k} c_{k}'' \rho_{k}'' = \text{resistivity contribution from the alloying elements with a concentration } c_{k} \text{ out of solution (precipitates).}$



Due to the resistivity of alloys is influenced by their microstructure, it can be an indicator of the alloy condition during heat treatment.

Measurement of the electrical resistivity requires only a simple procedure, applicable over a range of temperatures (i.e. four point measurement technique).

Electrical resistivity can be calculated theoretically and experimentally, however this may be complicated in commercial alloys where more than one element is precipitating from solution during heat treatment or to predict the behaviour in new systems.

It has been observed that the knowledge of the exact metallurgical state of an alloy is very important when studying its electrical resistivity characteristics. Therefore, it is recommended to work with a complementary technique that could provide more information about the thermodynamic behaviour of the system.

In this case, Differential Scanning Calorimetry technique is used as complement of the electrical resistivity measurements during aging hardening.

DSC is particularly useful for precipitation reactions in light alloys used for structural applications, where successive solid-state reactions of precipitation and dissolution can be analysed at increasing temperatures [5].

This technique measures the energy necessary to establish a nearly zero temperature difference between a substance and an inert reference material, as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate.

It's called "differential scanning" because scans the temperature at a specific rate. It can also perform a test at a constant temperature and in this case it's called "isothermal calorimetry".



Figure 5. Schematic of DSC system [6].







DSC can provide useful information on the starting conditions of the material and hence on the structure modifications on heating. It can give also information on the stability of the phases and on the kinetics of the decomposition processes [8].

Also, it is possible to record Continuous Cooling Precipitation diagrams with the DSC method because it supplies a measure for the amount of precipitates via the amount of released heat in dependence of cooling rate.

Example: Precipitation sequence in Al-5%Cu alloy

Precipitate/transition phase	Heating rate 10 °C/min	Heating rate 20 °C/min
GP zone	58.0 °C	60.5 °C
Θ"	87.6 °C	106.8 °C
Θ'	210 °C	219.4 °C
⊖-Al₂Cu	419.1 °C	421.7 °C





Figure 8. DSC curve obtained at heating and cooling rate 20 °C/min up to 500 °C for casted and homogenized Al-5 % Cu alloy [5].

Example: Determination of cooling rate for AW-6005A alloy

EN AW-6005A solution annealing: 540 °C 20 min



Figure 9. Specific precipitation heat and Vickers-hardness (HV-1) after aging as a function of the cooling rate [9].

Example: Relationship between electrical resistivity and DSC data



Figure 10. ER (a) and DSC (b) of aged AZ80 alloys (heating rate is 5 °C/min) [10].

Results of N9045B8 alloy (magnesium alloy):



Figure 11. DSC of "as quenched" N9045B8 alloy solution treated at 400°C (heating rate is 20 °C/min).

Conclusions

DSC is a useful tool to understand the behaviour of a sample during age hardening.

It can provide helpful information for the improvement of the heat treatment/quenching process.

* An isothermal calorimetry test could be performed to Mg samples in order to observe precipitation.

References

- [1] Callister, W.; Rethwisch, D., Materials science and engineering: an introduction, 2010, pp. 885, Hoboken, NJ; John Wiley & Sons.
- [2] Smith, W.F., Foundations of Materials Science and Engineering, 1993, 2nd Ed, New York, McGraw-Hill, Inc.
- [3] Olafsson, P.; Sandstrom, R. and Karlsson, A., Comparison of experimental, calculated and observed values for electrical and thermal conductivity of aluminium alloys, Journal of Material Science, 1997, Vol. 32, p. 4383-4390.
- [4] Noble, B. and Pike, T.J., Deviations from Matthiessen's rule in some magnesium-based alloys, J. Phys. F: Met. Phys., 1981, Vol. 11, Issue 3, p. 587-595.
- [5] Voncina, M.; Smolej, A.; Medved, J.; Mrvar, P. and Barbic, R., Determination of precipitation sequence in Al-alloys using DSC method, RMZ – Materials and Geoenvironment, 2010, Vol. 57, No. 3, pp. 295–304.
- [6] http://www.benelux-scientific.com/fileadmin/user_files/pdf/workshop_netzsch2009/intro_to_thermal_analysis.pdf
- [7] http://publicpartner.mt.com/content/mailings/UK/Lab/Introduction%20to%20DSC.pdf
- [8] Lussana, D.; Massazza, M. and Riontino, G., A DSC Study of Precipitation Hardening in a WE43 Mg alloy, Journal of Thermal Analysis and Calorimetry, 2008, Vol. 92, Issue 1, p. 223–225.
- [9] Milkereit, B.; Kessler, O. and Schick, C., Determination of Critical Cooling Rate for Hardening Aluminum Alloys using *HyperDSC*, PerkinElmer, Inc., 2010.
- [10] Yakubtsov, I.A., Effects of composition and microstructure on behaviours of electrical resistivity during continuous heating above room temperature in Mg-based alloys, Journal of Alloys and Compounds, 2010, Vol. 492, p. 153–159.