DSC investigation on WE43 and Elektron 21 Mg alloys

G. RIONTINO*, D. LUSSANA, M. MASSAZZA
Dipartimento di Chimica I.F.M. and N.I.S. Centre, Università di Torino, Via P.Giuria 9, 10125 Torino, Italy
E-mail: giuseppe.riontino@unito.it

A. ZANADA
Teksid-Aluminum, Getti Speciali Plant, Via Rondó Bernardi 15, 10040 Borgaretto, Italy

Published online: 10 March 2006

The microalloying elements and the control of precipitate-phase nucleation play a very important role in the processing technology of light alloys. Whilst the precipitation process has been reasonably assessed in the case of Al-based alloys, only empirical experience has been accumulated for the Mg alloys, although detailed TEM observations of the latter have been recently reported [1–3]. Conventional thermal treatments, often for long annealing times, have been used to increase the mechanical properties, but the relationship between phase transformations and properties is far from complete. Precipitation hardening mechanisms in these materials are not completely understood and a detailed decomposition sequence is not yet assessed. The probable precipitation process actually used to describe the solid solution depletion following thermal treatments has been summarized by Polmear [4], and in all cases several intermetallic compounds are formed with different degrees of coherency with the Mg matrix.

Calorimetric data in scanning conditions (DSC) have been proved to be a powerful tool in characterizing the structure evolution of supersaturated aluminum alloys, giving also useful hints into design and modifications of thermal treatments [5–7]. We aim to extend such a possibility to the magnesium alloys, for which no systematic data exist in literature. We have initially focused the attention on a rare-earths containing WE43 alloy, a good candidate for many structural applications in aerospace industries. The rare-earths addition gives good casting characteristics to Mg, due to the presence of low melting points which tend to suppress microporosity. Some preliminary data are reported for the Elektron 21 alloy, a new composition recently introduced in foundry industries, which offers a good compromise between castability and technological properties [8].

Rod samples have been supplied by *Teksid-Aluminum* in two thermal states: as-cast (AC) and T6-treated, according to the manufacturer' specifications. The chemical compositions of the investigated alloys are reported in Table I, and the thermal treatments of the as-received materials in Table II.

For both alloys, slices about 1.5 mm thick have been cut, from which small discs 5 mm in diameter have been punched. Some discs have been annealed at 525 °C for 8 hr in air, quenched in water at room temperature (WQ) or in calm air (AQ), and maintained in freezer at about –18 °C prior to successive measurements. A thin oxide layer is mechanically removed by grinding before any measurements.

Calorimetric scans have been made on all the samples with a TA-2010 apparatus in a protective pure argon atmosphere, by using pure aluminum as a reference. In some cases, a zero line has been obtained by scanning at the same rate the empty cell, and it has been subtracted from the original data. Hardness tests have been performed with a Vickers microindenter with a load of 300 N on the same sample used for the DSC measurements. The reported values are the average of five indentations. The results obtained are:

(a) WE43: A preliminary scanning at 10 K/min, without baseline subtraction, gives the results reported in Fig. 1. Large endothermic effects may be evidenced in the range 200–300 °C, and an exothermal signal of precipitation at about 380 °C. Small and extended exothermic effects around 150 °C seem to be present in all the samples. The starting microhardness values, before the calorimetric scan, are reported in the same figure. For the WE43 alloy, the highest value (about 91 VHN) is attained by the sample in the T6 state, as could be expected as this is the usually adopted thermal state for age hardening in the technological practice [4].

The literature reports a slow kinetics for the phase transformation in these kind of alloys [2]. As a thermal drag due to a relatively high scanning rate could overcome some significant thermal effects, we have performed new measurements at 2 K/min. The results are reported in Fig. 2 for the WQ and T6 samples, after the baseline subtraction.

* Author to whom all correspondence should be addressed.

0022-2461
DOI: 10.1007/s10853-006-6375-4

TABLE I Chemical composition (wt.%)

	MM	Zr	Y	Zn	Mg
WE43	3.1	0.6	4.2		bal
El.21	≈4	0.6		≈0.35	″

MM = mischmetal, Ce-free, Nd or Gd-containing.

TABLE II Thermal states of the as-received materials

WE43	525 °C/8 hr + calm air cooling + 510 °C/1 hr + calm air cooling + 250 °C/16 hr (T6)
El.21	525 °C/8 hr + calm air cooling + 200 °C/16 hr (T6)

A reference line is traced in the same figure, to evidence the sign of the calorimetric traces. The reference line is obtained by subtracting the zero line, at empty cell, from the trace for two pure and inert Al samples of approximately the same weight as the examined Mg samples. In this manner, both instrumental effects and heat capacity variations due to mass differences are minimized. The straight line in Fig. 2 is the result of a linear interpolation of the experimental reference line. The small slope (see the sensitivity of the measurements) may be due to small differences between the heat capacities of the two samples.

It is useful to divide the thermograms into three temperature ranges. Some considerations, supported by literature data, can be made from the comparison of the two traces. At low temperatures (range I), an exothermic peak is present, more pronounced in WQ than in T6 sample. The persistence of this signal in the T6 sample suggests that a certain degree of oversaturation is still present. From the TEM observations reported in literature [1–3, and references therein] about the precipitation sequence, we can deduct that the signal P1 is due to the

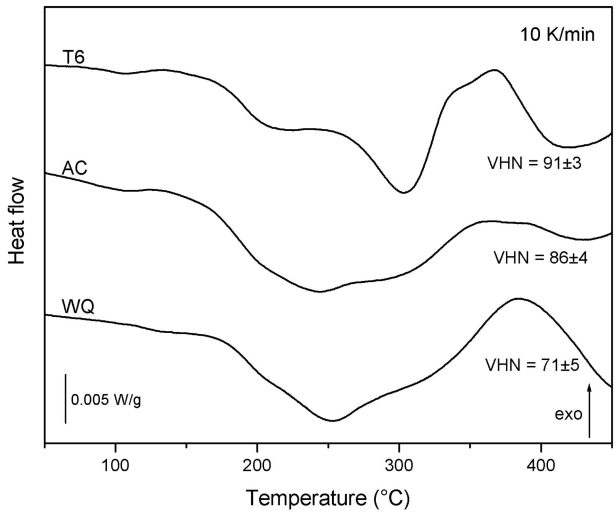

Figure 1 DSC traces of the WE43 alloy in different thermal states (scanning rate: 10 K/min).

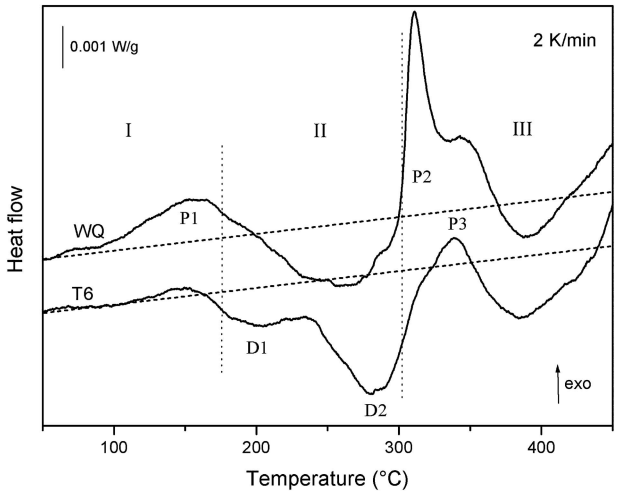

Figure 2 DSC traces of the WE43 alloy in two thermal states (scanning rate: 2 K/min). A reference line is reported (*dotted line*): see text for its derivation.

formation of the metastable phases β'' and β' having different shapes and compositions, not distinguishable by DSC. Both these phases are originated from an ordered phase which forms immediately after quenching, in the very early stages of transformation [1], In the sample T6, the formation of β'' and β' has just partially occurred during the thermal treatment, giving rise to two different dissolution signals D1 and D2 (range II) during the calorimetric scan. The higher amount of β''/β' formed after the T6 treatment justifies the higher hardness value found with respect to the WQ sample (91 against 71 VHN). In the same range of temperatures, the dissolution signal of the same phases during the scanning of the WQ sample is undifferentiated, probably because of a continuous transformation of β'' into β'. Details on the mutual ratio of these phases, on their origin and composition still remain unclear.

In range III of the thermograms, two exothermic signals P2 and P3 are present. We can assume that P2 is due to the formation of the metastable phase β_1, first introduced by Nie and Muddle [3], and confirmed by Apps *et al.* [2]. The P3 signal is due to the transformation of the β_1 phase into the equilibrium phase β [2, 9]. The difference of DSC traces for the WQ sample in Figs 1 and 2 are due to different kinetics of the processes giving rise to the signals. The nearly complete disappearance of P2 in the T6 sample can be explained with the formation of β_1 during the thermal treatment. Alternatively, since the β' phase is considered necessary for the formation of β_1 [3], the wide β'-dissolution signal D2 suggests that there is not enough β' to form β_1. Both hypotheses are possible, and at this stage of analysis the probability of a joined effect is not unlikely.

At temperatures higher than about 370 °C a final dissolution occurs, partially masked by a strong exothermic signal due to oxidation.

From what reported above, a more detailed analysis of the first exothermic manifestations is necessary, since

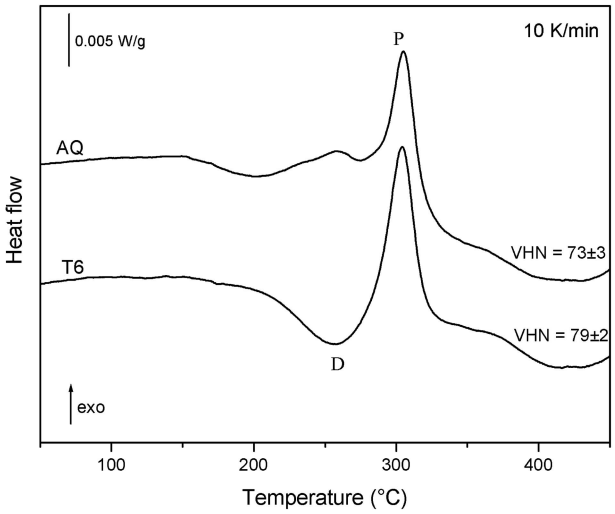

Figure 3 DSC traces of Elekron 21 alloy in the thermal states labeled (scanning rate: 10 K/min).

their evolution seems to affect the successive precipitation at higher temperatures.

(b) Elektron 21 : No previous data are reported in literature for this alloy. A study on a similar composition, a GN72 alloy containing the same rare-earth elements (Gd and Nd) in different proportion, has recently been done by Apps *et al*. [2]. They correlate the increased hardening response during an isothermal treatment at 300 °C to an increased volume fraction of the β_1 phase on annealing. It is interesting to remark that the same precipitation sequence is reported for Gd-containing Mg alloys [10].

Some preliminary comments can be made on the basis of the thermograms reported in Fig. 3 for the two thermal states indicated. The traces suggest that the main exothermic signal at around 300 °C could be due to the formation of the same β_1 phase as in the WE43 alloy, coming from intermediate phases not dissimilar in composition from β''/β'. In effect, the rare earth amount in WE43 is equivalent to that in GN72 and slightly higher than in El.21, but the same solute elements enter in the formation of the β phases (Nd or Gd, and Y). The slightly higher hardness value in the T6 state with respect to the AQ one can be justified by the formation of intermediates during the annealing, intermediates revealed on a successive scanning through their dissolution signal D in Fig. 3.

In this case too, the signals evolution in DSC after suitable thermal treatments as well as direct observations of the undergoing structure should give a hint to the interpretation of structure–properties correlations.

Acknowledgment

This work has been partially supported by a national project MIUR-PRIN no. 2004023079.

References

1. C. ANTION, P. DONNADIEU, F. PERRAD, A. DESCHAMPS, C. TASSIN and A. PISCH, *Acta Mater.* **51** (2003) 5335.
2. P. J. APPS, H. KARIMZADEH, J. F. KING and G. W. LORIMER, *Scripta Mater.* **48** (2003) 1023.
3. J. F. NIE and B. C. MUDDLE, *Acta Mater.* **48** (2000) 1691.
4. I. J. POLMEAR, "Light Alloys: Metallurgy of the Light Metals, 3rd edn." (Arnold, 1995).
5. R. N. LUMLEY, I. J. POLMEAR and A. J. MORTON, *Int. Pat. Classification* **C22F**(1/057) (2002).
6. G. RIONTINO and M. MASSAZZA, *Philos. Mag.* **84**(10) (2004) 967.
7. G. RIONTINO, A. DUPASQUIER, R. FERRAGUT, M. M. IGLESIAS, C. MACCHI, M. MASSAZZA, P. MENGUCCI and A. SOMOZA, in Proceedings of the 9th International Conference on Aluminum Alloys, (Brisbane, 2004) edited by J. F. Nie, A. J. Morton and B. C. Muddle.
8. Magnesium Elektron, sheet no. 455, www.magnesium-elektron.com.
9. J. F. NIE and B. C. MUDDLE, *Scripta Mater.* **40** (1999) 1089.
10. B. SMOLA, I. STULIKOVA, F. VON BUCH and B. L. MORDIKE, *Mater. Sci. Eng.* **A324** (2002) 113.

Received 26 May
and accepted 14 September 2005