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A DSC STUDY OF PRECIPITATION HARDENING IN A WE43 Mg ALLOY

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The phase transformations leading to hardening a Mg–Y–Nd–Zr (WE43) alloy submitted to thermal treatments are followed by calorimetric and microhardness measurements. A double-stage thermal treatment is adopted, the first at 210°C for 8 h and the second at 150°C, on samples quenched from a temperature higher than the conventional one. A secondary precipitation of the metastable phase β" in the second stage makes the hardness increase with respect to the primary precipitation.

Keywords: *DSC, Mg alloys, precipitation hardening, secondary precipitation*

Introduction

Magnesium alloys are widely used in modern industry, mainly due to their low density and good mechanical properties that makes them outstanding in strategic sectors like the transport industry. In particular, commercial alloys with elements of the rare-earths (RE) have been intensively investigated since the 1940's as aircraft components: relatively low melting eutectics in the binary compositions offer good casting characteristics and tend to suppress microporosity. An exhaustive review of compositions and properties of commercial Mg-RE alloys is provided by Rokhlin [1], who has recently collected, among the other, many pioneering Russian papers on the argument, and by Polmear [2]. A key process, both from a technical and a scientific point of view, is the precipitation hardening that is obtained by quenching from high temperatures an oversaturated solid solution and by annealing at suitable temperatures. The structure modifications sequence undergoing thermal treatments leads to desirable improvements of mechanical and chemical properties. This is the reason why the precipitation mechanisms including structure and composition variations of the forming phases are widely investigated. Noticeable results in this direction have been obtained by using sophisticated methods of investigation and of direct observation, including positron annihilation spectroscopy and field ion microscopy. In any case, given the composition of a particular alloy, the choice of a beneficial thermal treatment and the reproducibility of its effect on some properties passes through a bulk calorimetric investigation of the material. Differential Scanning Calorimetry (DSC) could provide useful information on the starting conditions of the material and hence on the structure modifications on heating. Of course, the technique is blind with respect to the particular phase originating the energy signals, but concurrent observations and measurements enlighten in many cases the effect of thermal treatments giving also information on the stability of the phases and on the kinetics of the decomposition processes.

In the present paper we report some results on the precipitation sequence and its hardening effect in a Mg–Nd–Y alloy (WE43) which is being used for some aeronautical applications because of its relatively high strength at elevated temperatures. DSC data and microhardness results will be discussed for a double-step thermal treatment following a non-conventional solutioning and quenching treatment.

Experimental

Disc-shaped samples of about 5 mm in diameter and 1 mm thick (for a mass of about 50 mg) were obtained from gravity-cast WE43 alloy containing 4.2Y+2.3Nd+0.6Zr (mass%), corresponding to 1.21Y+0.41Nd+0.17Zr (at%). The samples were solution treated at different temperatures (525, 550 and 575°C), then quenched in water at room temperature.

A DSC TA 2010 instrument and a PerkinElmer DSC7 were used to measure the energy evolution from room temperature to the melting point. Ageing treatments were performed in an air-ventilated furnace. With a Vickers microindenter and a load of 3 N the hardness value after each thermal treatment on the same samples used for the calorimetric scans was obtained as average of at least five indentations.

Results and discussion

The precipitation sequence during different thermal treatments in a WE43 alloy was described in a previous work [3], in which positron annihilation data, TEM

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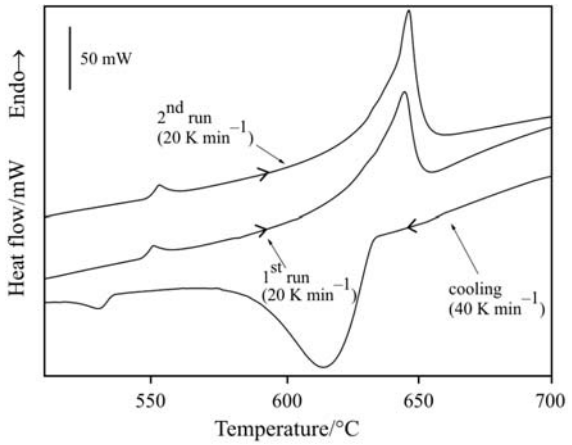

Fig. 1 DSC traces of as-quenched samples up to melting. Heating and cooling rates are labelled

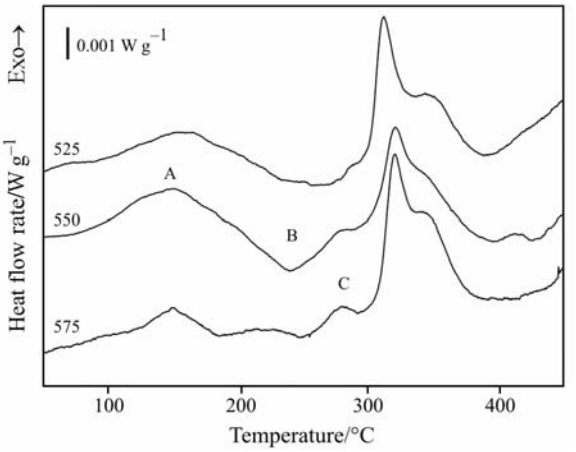

Fig. 2 DSC traces of samples quenched from different temperatures. Scanning rate: 2 K min^{-1}. A zero-line at empty cell has been subtracted from the original traces

observations and microhardness measurements together with DSC results were reported. In that work, a conventional quenching after a solution heat treatment of 8 h at 525°C was adopted. In this work, a preliminary DSC investigation on a small quantity of material was made in order to determine its melting temperature and hence to fix a top limit for the solution treatment. To this aim, a Perkin-Elmer DSC7 was used in order to avoid some possible contamination effects in our cell. The results are reported in Fig. 1.

Two scans have been made, with an intermediate programmed cooling. A large endothermic melting signal is visible, starting around 600°C, together with a small signal at about 550°C. This last can be reasonably ascribed to the dissolution of an equilibrium phase, as confirmed by its counterpart during the intermediate cooling at a higher scanning rate.

On this basis, two alternative temperatures was chosen, higher than the conventional 525°C, to dissolve the maximum quantity of solute atoms into the magnesium matrix. Samples was quenched from 575°C and from 550°C, and preliminary calorimetric scans are reported in Fig. 2.

The phase transformations sequence does not change appreciably for the three quenchings: as discussed in [3], the large exothermic signal A centered at about 150°C is assigned to an undifferentiated precipitation of the phases β" ($D0_{19}$ symmetry, $Mg_3Y_{0.85}Nd_{0.15}$ [4]) and β' (orthorhombic, $Mg_{24}Y_2Nd_3$ [4]), the successive endothermy B centered at about 260°C to their dissolution on scanning, while the two exothermic signals at higher temperatures are due to the formation of an intermediate phase β_1 and to its transformation into the stable β phase (fcc, $Mg_{12}YNd$ [4]). The main difference from the conventional quench at 525°C is the presence of an exothermic signal C at about 275°C in the samples quenched at higher temperatures and the contemporary higher structuration of the first precipitation signals after quenching from 575°C. The signal C can be ascribed to a further formation of β' phase. As reported by Antion *et al*. [4], in fact, the phase β' can form through two different mechanisms, the first consisting in a direct formation from the solid solution, the second by nucleation on the precursor β" phase. The higher amount of solutes available after quenching from higher temperatures enhances the formation of β" phase and then the formation, through the second mechanisms, of β' during the calorimetric scan.

However, the samples solution heat treated at 575°C showed an excessive oxidation when quenched in water, making too difficult to carry out a complete set of analysis. For this reason, only the intermediate temperature of 550°C was chosen, showing the same calorimetric features than after quenching from 575°C (Fig. 2).

Ageing the samples quenched from 550°C at the same annealing temperature (210°C) which has been shown [5] to be the most effective in increasing the hardness of samples quenched from 525°C, we obtain similar results. The calorimetric scans in Fig. 3 reveal the progressive disappearance of the exothermic effects C on ageing and in meantime the persistence of a small exothermic contribution around 150°C for the longest ageing time. The hardness trend is similar (Fig. 4) but the absolute values of the samples quenched from 550°C are higher than those quenched from 525°C. This is due to the higher solute amount available in the Mg matrix for a primary precipitation.

The persistence of the signal A allows to consider that even after a long ageing at 210°C a certain amount of solute remains available for a secondary precipitation at lower temperatures. To verify this hypothesis, an annealing at 210°C for 8 h has been performed on quenched samples. In this way, a hardness increase of about 80% of the total amount obtainable by annealing at this temperature is reached. A successive

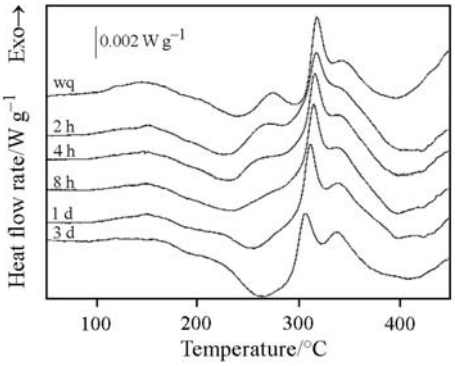

Fig. 3 DSC traces of samples quenched from 550°C and annealed at 210°C for the labelled times. Scanning rate: 2 K min^{-1}

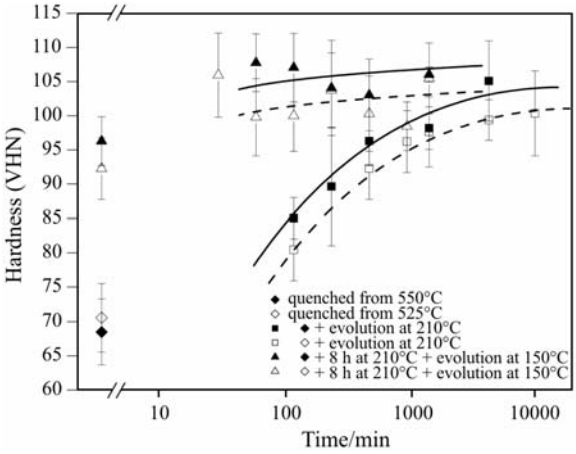

Fig. 4 Hardness trend for samples annealed at 210°C after quenching, and for samples submitted to the double-stage treatment (8 h at 210+150°C). The dotted lines are referring to the samples quenched from 525°C [5]

annealing at 150°C for increasing times gives the results reported in Fig. 4 for the hardness measurements and in Fig. 5 for the DSC traces.

The calorimetric traces evolution shows two main features: 1) a progressive reduction of the first extended exothermic signal and 2) a contemporaneous small increase of the dissolution signal B. It is worth noting the faintness of the signals, a general characteristics of the Mg-based alloys with respect, for example, to the Al-based alloys ([6, 7]). Nevertheless, the hardness measurements at 150°C after a primary annealing at 210°C for 8 h enlighten a significant increase just after the lowest annealing times. This increase is of the same order of magnitude of that obtainable by annealing at the higher temperature of 210°C for the longest times. In Fig. 5, the hardness trend for the same double-stage treatment in samples quenched from 525°C is reported for comparison. In both cases, a secondary precipitation of the β" phase is claimed to occur.

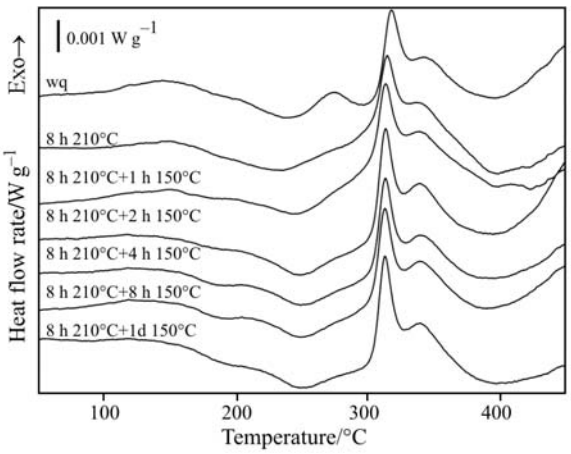

Fig. 5 DSC traces of samples quenched from 550°C, annealed 8 h at 210°C (first step) and subsequently annealed at 150°C (second step) for the labelled times. Scanning rate: 2 K min^{-1}

Conclusions

The higher content of solute atoms after quenching from 550°C with respect to a conventional quenching from 525°C allows:

- a more evident evolution of the calorimetric traces;
- a more relevant formation of the metastable phases responsible for the hardness increase.

In particular, a double-stage thermal treatment gives the maximum hardness increase, through a secondary precipitation, with a significant time and energy saving.

Acknowledgements

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