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Assessment of thermal-treatment conditions for age-hardenable particulate-reinforced aluminium alloys by calorimetric methods[1]

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Abstract

To assess the influence of ceramic reinforcements on the solid-state transformations that proceed during processing, fabrication and application of particulate-reinforced age-hardenable aluminium alloys (PRA), microcalorimetry and differential scanning calorimetry investigations were carried out in combination with microstructural and mechanical investigations. PRA-material studies were conducted on the AlCuMg (2014), AlMgSi (6061, 6082) and AlZnMgCu (7075) alloy systems containing Al_2O_3-and SiC-particulates with volume fractions between 10–25%. In order to cover the whole range of PRA-production and application, the consequences of the presence of ceramic particulates were investigated with respect to high-temperature exposures (homogenisation, extrusion, solution treatment), to cooling rates from solution treatment and to precipitation strengthening by natural and/or artificial ageing. The various effects are highlighted for selected PRA-systems and PRA-conditions, and the overall mechanisms are discussed. Implications for modifications of processing and fabrication parameters in order to optimally exploit the potential of PRA-material are discussed. © 1998 Elsevier Science B.V.

Keywords: Calorimetry; Interface reactions; Precipitation; Particulate-reinforced aluminium alloys

1. Introduction

From the global annual consumption of metal matrix composites (MMCs) of ca. 5000 ton in 1996, by far the largest fraction is held by particulate-reinforced aluminium alloys (PRA). These comprise conventional aluminium alloys of the AlSiMg, AlCuMg and AlZnMgCu systems, containing between 10 and 25 vol% of ceramic particulates (Al_2O_3 or SiC) ranging in size from 5–30 μm. A low-cost liquid processing technique developed by DURALCAN/USA, provides pre-material for casting, extrusion or forging with the price somewhere between 2–3 times higher than monolithic material. P/M techniques have the potential for higher quality (e.g. homogeneous distribution of particulates), but at much higher cost.

The introduction of ceramic particulates into the metallic matrix provides a means to tailor mechanical and physical properties of the resulting PRA-material, mainly by choice of the type, size and volume fraction of particulates. Thermal conductivity and thermal expansion have been successfully adapted for functional applications [1–3] and using liquid infiltration

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techniques, particulate volume fractions of up to 60% are already attainable [4], thereby extending the range of property tailorability.

As to mechanical properties, particulates entail a significant increase of Young's modulus of up to 40% [5], and are able to render wear resistance levels comparable to cast iron [6]. However, yield and tensile strength usually are only slightly enhanced by particulates, the reason for which is still being discussed [7]. Strong indications have been put forward that the major strength contribution by particulates stems from higher dislocation densities that are created during quenching due to the thermal expansion mismatch between the metallic matrix and the ceramic particulates; consequently, it has been observed [8] that strength benefits due to particulates are lost during exposure at intermediate temperatures (e.g. 100 h/200°C) as a result of a thermal annihilation of dislocations.

In this way, for structural applications where high stiffness at low weight is needed in combination with adequate strength properties, the latter have to be provided by conventional precipitation hardening of the metallic matrix. It has also been shown [6], that for optimal wear resistance to be achieved by particulates, the matrix has to be hardened.

Hence, to fully exploit the engineering potential of PRA it is necessary to understand and control the effects of particulates, i.e. the formation of strengthening precipitates and, most important, the interdependence of these two factors. A considerable number of investigations has been made regarding the influence that is exerted by particulates on the nucleation and growth of strengthening GP-zones or metastable phases, and it has even been considered to develop new alloys which are optimised for usage as PRA-matrices. As an overall picture from the literature, it can be summarised that, in age-hardened wrought alloys of the type 2XXX (AlCuMg) and 6XXX (AlMgSi), natural ageing is retarded in the presence of particulates [9,10] whereas artificial ageing is accelerated [11,12]. In the first case, this is put down to the annihilation of excess vacancies on dislocations formed around particulates during quenching, resulting in a reduced diffusion rate, whereas in the second case an increased dislocation density provides a high number of heterogeneous nuclei for the formation of metastable precipitates. In general, the decomposition mechanisms remain the same as in the monolithic alloy, and only minor modifications of the thermal treatment parameters are needed to attain peak-strength values (Fig. 1, T6).

In a recent paper [13], it was shown that, the influence of ceramic particles on the precipitation in an age-hardenable matrix can be extremely high if, during a prolonged high-temperature exposure, the matrix is depleted of solute atoms as a consequence of reactions at the matrix/particle interface. Eventually this can result in a total loss of the matrix' hardening potential.

It is the purpose of this paper to demonstrate, by using calorimetric methods, the different ways in which ceramic particulates can change the precipitation behaviour of an age-hardening matrix, covering three important technological steps, i.e. ''homogenisation or solution treatment'', ''quenching from the one-phase region'' and ''precipitation-hardening treatment''.

2. Experimental conditions

A series of different PRA-materials, produced by ingot, spray and powder metallurgy (I/M, Osprey, P/M) and extruded by AMAG/Austria, was investigated. The alloy concentrations of solute atoms, the type and volume fraction of ceramic particulates were as follows:

- *Alloy 2014* (Cu 3.9–5.0%; Mg 0.2–0.8%) with 0, 10 and 20 vol% Al_2O_3 (I/M)
- *Alloy 6061* (Mg 0.35–0.6%; Si 0.30–0.6%) with 0, 10, 15 and 20 vol% Al_2O_3 (I/M)
- *Alloy 6061* (Mg 0.35–0.6%; Si 0.30–0.6%) with 0, 15 and 25 vol% SiC (P/M)
- *Alloy 6082* (Mg 0.5–1.2%; Si 0.7–1.3%) with 0 and 15 vol% SiC (Osprey)
- *Alloy 7075* (Zn 5.1–6.1%; Mg 2.1–2.9%; Cu 1.2–2.0%) with 0 and 15 vol% (I/M)

It has to be pointed out, that the monolithic alloys produced by AMAG had slightly different compositions in comparison to the PRA-materials obtained from other producers (DURALCAN, DWA, Osprey).

To simulate homogenisation and solution treatments, samples were exposed to temperatures >500°C for periods of up to 24 h. Cooling from the

one-phase region was either performed by quenching in water within <0.5 s, or by natural cooling down to room temperature within ca. 5 min. After quenching or cooling the material was aged, either at room temperature (natural ageing) or at intermediate temperatures between 120–180°C (artificial ageing) in oil baths.

A self built Tian–Calvet microcalorimeter in the isothermal mode served to determine the precipitation kinetics for natural and artificial ageing. Differential scanning calorimetry with a Perkin–Elmer DSCII at a heating rate of 40 K/min was used to characterise the type, thermal stability and volume fraction of precipitates, formed before as well as during the DSC scan; similar investigations with a heating rate of ca. 10 K/h were carried out in a SETARAM microcalorimeter in the dynamic mode at 1000°C. Discs (6 mmϕ×1 mm) served as samples for DSC, cylinders (14 mm$\emptyset$×70 mm) were used in the microcalorimeters. Further details for calorimetric measurements are given in [14].

Mechanical properties were assessed by hardness measurements and tensile testing. Microstructural features, including matrix/particulate interfaces were studied by transmission electron microscopy, within a Philips CM 20/STEM.

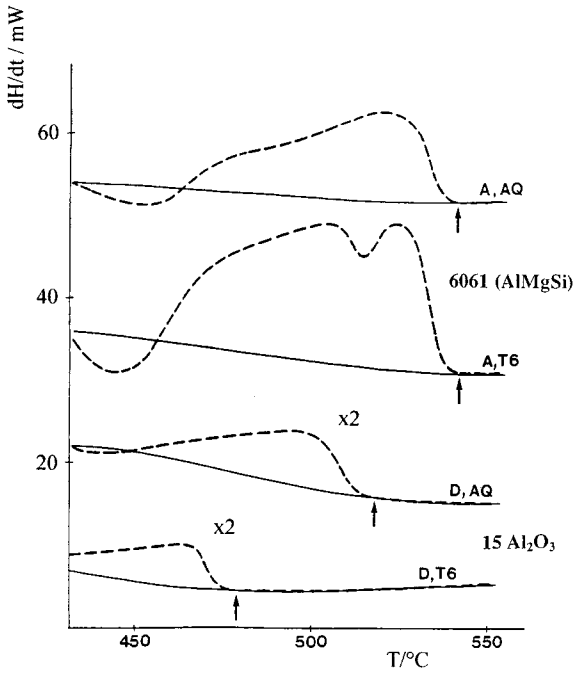

Fig. 1. Dynamic microcalorimetry for alloy 6061 with 0 and 15 vol% Al_2O_3), for the as-quenched and peak-aged condition (AQ, T6).

3. Experimental results

3.1. High-temperature exposure

PRA-samples based on alloy 6061, as well as monolithic samples, that had been held at 525°, 530° and 540°C (being the range of solution treatment temperatures) for exposure times up to 24 h were investigated in regard to their precipitation potential. In Fig. 1, flow-rate curves, dH/dt, for 0 and 15 vol% Al_2O_3 obtained by dynamic microcalorimetry, are given for the temperature range >400°C. Samples were measured in the as-quenched condition (AQ), i.e. solution treatment (1 h/525°C), followed by quenching in water, and in the peak-aged condition (T6), i.e. solution treatment, quenching in water and 16 h/180°C-ageing. In case of the un-reinforced alloy, for both the conditions (AQ and T6) a strong endothermic effect is observed between 46° and 540°C and, although the enthalpies are quite different, the thermal baseline is attained practically at the same temperature of ca. 540°C. For the reinforced material, the endothermic peaks are about half in size and, furthermore, shifted towards lower temperatures.

The kinetics of artificial ageing is shown on flow-rate curves in Fig. 2 obtained by isothermal microcalorimetry at a temperature of 130°C. Each sample of alloy 6061 containing 0 and 15 vol% Al_2O_3 was measured twice; first, after quenching from a 30 min/525°C solution treatment, and then after quenching from 24 h/540°C. For the un-reinforced material, the exothermic heat evolution, $-$dH/dt, is completely unaffected by the duration of a high-temperature treatment, whereas for the PRA-material the heat flow is significantly diminished by a prolonged high-temperature exposure. It can be stated from Fig. 2 that, in general, the flow rate is lower in the reinforced material, with its maximum being shifted towards longer ageing times after solution treatment for 24 h.

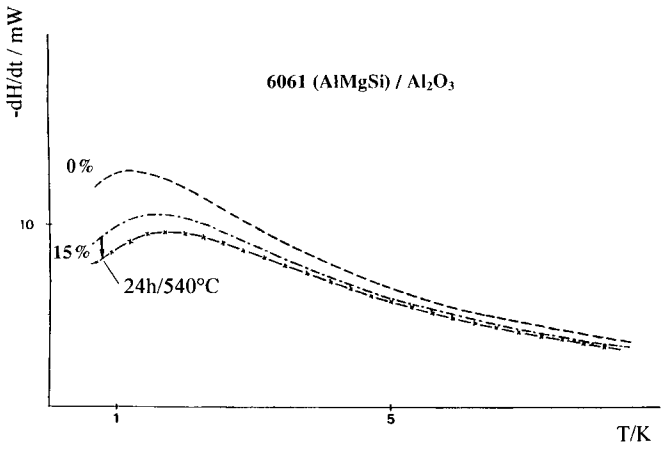

Fig. 2. Isothermal microcalorimetry at 130°C for reinforced and un-reinforced alloy 6061 after 30 min/525°C (———) and 24 h/540°C (→) solution treatment followed by quenching.

3.2. Influence of cooling rate

Alloy 6061 samples with volume fractions of SiC and Al_2O_3 particulates from 0 to 25% were either quenched or air cooled from solution treatment and then investigated by DSC analysis. In Fig. 3, flow-rate curves are presented for selected samples after air-cooling. The un-reinforced alloy exhibits a small endothermic effect between 480 and 550 K which is followed by an exothermic one of comparable size; above 600 K a large and a small endothermic peak appear before the baseline is reached at ca. 850 K. For all three PRA-materials, the flow-rate curves in Fig. 3 are quite different from those of the monolithic alloy, as there is only one pronounced endothermic effect with two or three peaks, but no exothermic, and no separate low-temperature endothermic effect, whereas on samples that had been water quenched, significant endo- and exothermic effects were detected <600 K, both for monolithic and PRA-samples.

3.3. Natural and artificial ageing

PRA-material based on alloys 2014, 6061 and 6082 was investigated after solution treatment and quenching in a microcalorimeter stabilised at room temperature ($RT \approx 22°C$), or after RT-ageing (T4 condition) by DSC thermal analysis. In Fig. 4, microcalorimetric flow-rate curves, normalised with respect to the different matrix volume fractions, are presented for alloy

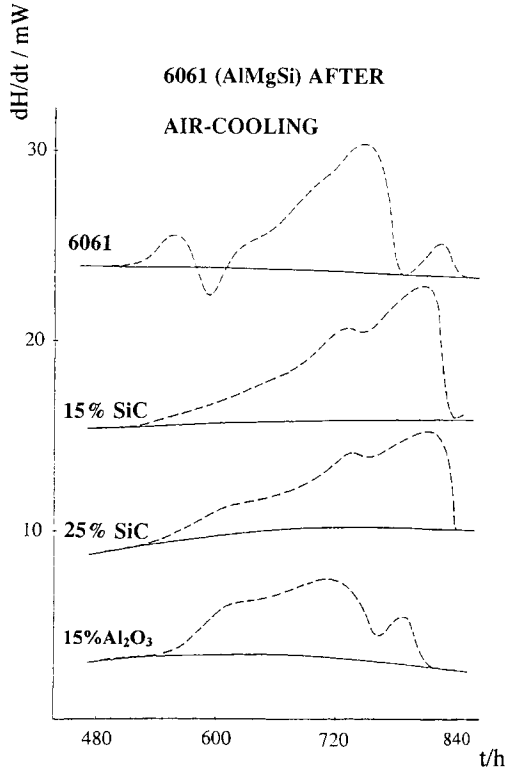

Fig. 3. DSC flow-rate curves for material indicated, after solution treatment and air cooling.

2014 with 0, 10 and 20 vol% Al_2O_3. The maximum of the flow rate, situated at ca. 1 h, is shifted towards

longer ageing times and the total heat released is diminished, as the content of particulates is increased. Complementary DSC investigations indicated that the enthalpy associated with the first endothermic effect is reduced by higher particulate contents.

After quenching in water and solution treatment, PRA-samples based on 6082 were artificially aged according to condition T6 (10 h/165°C). In Fig. 5, DSC flow-rate curves are compared for alloy 6082 with 0 and 15 v% SiC. The first endothermic effect located between 480 and 600 K, although equal in enthalpy content, is shifted in the direction of lower temperatures by about 10 K when particulates are added. From hardness measurements for ageing between 0 and 32 h at 155°C it is found, that after 8 h the hardness of 6082/15 v% SiC is 90% of its peak value, whereas only 84% are attained by the unreinforced alloy.

Artificial ageing including also overageing was studied in 6061 reinforced with SiC and Al_2O_3 for