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Quench sensitivity of Al–Mg–Si alloys: A model for linear cooling and strengthening

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ABSTRACT

This work studies the quench-induced precipitation during continuous cooling of five Al–Mg–Si alloys over a wide range of cooling rates of 0.05–2 × 10^4 K/min using Differential Scanning Calorimetry (DSC), X-ray diffraction, optical- (OM), transmission electron- (TEM) and scanning electron microscopy (SEM) plus hardness testing. The DSC data shows that the cooling reactions are dominated by a high temperature reaction (typically 500 °C down to 380 °C) and a lower temperature reaction (380 °C down to 250 °C), and the microstructural analysis shows they are β-Mg_2Si phase formation and B′ phase precipitation, respectively. A new, physically-based model is designed to model the precipitation during the quenching as well as the strength after cooling and after subsequent age hardening. After fitting of parameters, the highly efficient model allows to predict accurately the measured quench sensitivity, the volume fractions of quench induced precipitates, enthalpy changes in the quenched sample and hardness values. Thereby the model can be used to optimise alloy and/or process design by exploiting the full age hardening potential of the alloys choosing the appropriate alloy composition and/or cooling process. Moreover, the model can be implemented in FEM tools to predict the mechanical properties of complex parts after cooling.

1. Introduction

In recent years substantial progress has been reported in modelling of diffusion controlled phase transformations and the modelling of the thermodynamics of commercially important complex alloy systems, including first principles modelling. In this paper we will investigate how this progress can be used to provide a computationally efficient new model for a technically important process: quench sensitivity of heat treatable aluminium alloys. In heat treatable Al-based alloys precipitation hardening is the dominant strengthening mechanism. For most commonly used alloys such as the Al–Mg–Si (6xxx) and the Al–Zn–Mg–(Cu) (7xxx) alloys, age hardening response can be seriously affected by the cooling rate from solution annealing (e.g. [1–9]; and also toughness can be reduced due to reduced cooling rate [10]. To achieve optimal mechanical properties, precipitation during quenching must be fully suppressed, and this is achieved only if the alloy is cooled with the upper critical cooling rate or faster (e.g. [4,8,7]). However, fast cooling can induce residual stresses (e.g. [11–13]), and hence, in order to obtain an optimal balance between strength and residual stresses/distortion, cooling from solution annealing should be done with the upper critical cooling rate or slightly faster. Nevertheless, in some parts with varying wall dimensions it might be difficult to realise the same cooling rate at every location. Nowadays it is relatively easy to calculate the temperature developments in such parts at every location by finite element modelling (e.g. [12]). However, for prediction of the mechanical properties at varying cooling rates, no models that incorporate reliable thermodynamic and kinetic models have hitherto been published, nor have any models been tested using extensive experimental data. The present work addresses these issues in two ways: improved models and model verification through comparison with much increased and more detailed experimental results.

Differential Scanning Calorimetry (DSC) is frequently used for investigation of diffusion controlled precipitation reactions (e.g. [14,3,15,8,16,17]). In recent years significant improvements were obtained in the in situ investigation of the precipitation processes

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during cooling of Al alloys from solution annealing through the development of high sensitivity in-situ DSC techniques [18–21]. These technically and metrological sound DSC methods allow to measure the enthalpy changes over the whole cooling rate range of technical and physical interest: from slow cooling with phase transformation close to equilibrium conditions up to cooling rates near the upper critical cooling rate. In the present work, the enthalpy change during cooling is used as basis for the modelling.

We will in this work derive a model for precipitation during quenching and subsequent ageing of Al–Mg–Si alloys and combine that with a model for precipitation hardening, to provide predictions of strength and hardness for cooling rates that stretch over 6 decades. In the model we will incorporate very recent progress in first principles modelling of the phases in the Al–Mg–Si system results [22] and very recent models for precipitation kinetics [23,24]. The model is tested against an extensive set of experimental data.

2. Experimental

2.1. Investigated alloys

In this work, five different 6xxx alloys covering a wide range of compositions were investigated. The alloys are AA6063, AA6005A, and three alloys within the composition range of AA6082, representing variants with low Mg and Si content ($AA6082_{low}$), typical (medium) Mg and Si content ($AA6082_{typ}$) and high Mg and Si content ($AA6082_{high}$). The chemical compositions of the investigated batches are given in Table 1. All alloys have been cast, homogenised and subsequently extruded. They were received as extruded profiles, from which samples were cut for further heat treatment and investigations. We combine a very large amount of new and existing experimental data (about 600 DSC experiments, about 500 hardness tests, microstructural analysis of more than 100 samples).

2.2. Heat treatment, Differential Scanning Calorimetry and hardness testing

The main focus of this work is the quenching step within the age hardening heat treatment procedure. The basic scheme of the experimental applied heat treatments is shown in Fig. 1. The solution annealing at 540 °C for 20 min was followed by linear cooling with cooling rates varying in a wide range ($0.05–2 \times 10^4$ K/min). Differential Scanning Calorimetry (DSC), covering cooling rates from 0.1 K/min to 375 K/min, are realised by employing three different types of DSC devices: Setaram 121 DSC 0.1–6 K/min; Mettler-Toledo 823 DSC 6–30 K/min; PerkinElmer Pyris 1 DSC 30–375 K/min. Samples were measured vs a thermodynamic inert reference sample of pure aluminium. The baseline measurements were performed measuring pure Al samples in both DSC-microfurnaces. For each measurement one corresponding baseline was measured, and excess specific heat capacity curves reflecting the enthalpy changes due to reactions were determined (Fig. 2A). The specific precipitation enthalpy was evaluated by integrating the excess specific heat capacity curves and precipitation

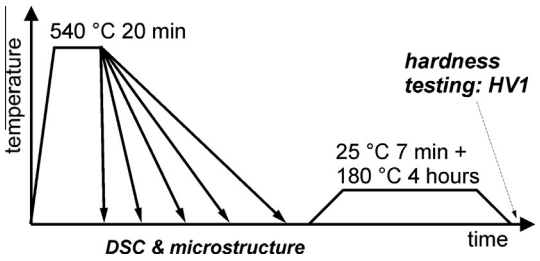

Fig. 1. Temperature–time scheme of experiments.

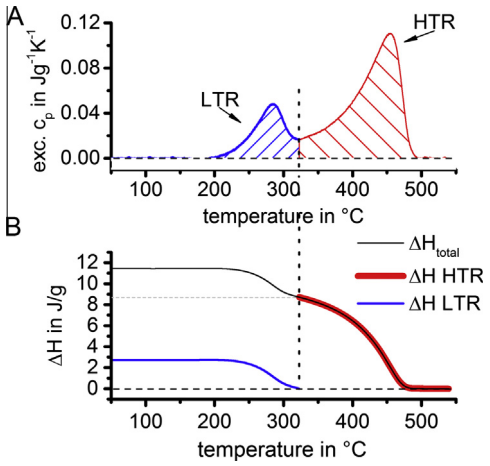

Fig. 2. Schematic evaluation of DSC cooling curves by sections wise integration. (A) DSC curve separated in high- and low-temperature-reactions (HTR/LTR). (B) Related integral curves for corresponding temperature intervals.

enthalpies of partially overlapping precipitation peaks was evaluated using the minimum heat flow as indicated in Fig. 2B (for further details see [18]).

Samples for hardness testing were cooled to room temperature at constant rates of $0.05–2 \times 10^4$ K/min, and, following a brief 7 min ageing at room temperature, they were artificial aged at 180 °C for 4 h.

To achieve very high controlled cooling rates up to 2×10^4 K/min heat treatments of additional samples were performed in a Baehr A/D 805 dilatometer. The complete heat treatments of hardness samples were performed either in DSC or dilatometer, thus ensuring a complete control of the entire temperature–time profile. Vickers hardness HV1 (load 1 kg) was tested with a Shimadzu HMV-2E small-force hardness indenter according to ISO 6507-1 applying an indentation duration of 10 s. At least six indentations per sample were performed. In addition, selected samples for microstructure investigation on $AA6082_{typ}$ were solution annealed and subsequently cooled using procedures approximating industrial practice: quenching in room-temperature-water, cooling in slightly moving air and slow air-cooling (in still air). Cooling rates were measured, and at 370 °C the cooling rates were about 400, 3 and 1 K/min, respectively. These samples were all artificially aged after cooling (i.e. a T6 treatment) and prior to microstructure investigation.

Table 1
Chemical composition of investigated alloys (mass fractions in%) obtained by optical emission spectroscopy (OES) analysis, with experiments performed. cDSC = cooling DSC, hDSC = heating DSC.

Alloys	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Experiments
AA6063	0.5	0.19	0.02	0.03	0.47	0.005	0.03	0.013	TEM, cDSC, SEM, HV, OM, XRD
AA6005A	0.68	0.2	0.01	0.11	0.57	0.04	0.01	0.018	TEM, cDSC, SEM, HV, OM, XRD
$AA6082_{low}$	0.73	0.22	0.05	0.48	0.61	0.003	0.009	0.02	cDSC, SEM, HV, OM, XRD
$AA6082_{typ}$	1.01	0.19	0.03	0.44	0.68	0.04	0.02	0.01	TEM, hDSC, HV
$AA6082_{high}$	1.23	0.2	0.09	0.65	1.05	0.2	0.05	0.03	cDSC, SEM, HV, OM, XRD