Elevated Temperature Deformation Behavior of Dispersion-Strengthened Al and Al-Li-Mg Alloys

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A model describing the behavior of dispersion-strengthened aluminum alloys, when subjected to elevated temperature plastic deformation, is presented. The aims are twofold:

- **to use the model for extrapolation of laboratory data to predict behavior under service conditions where the strain rate is extremely low ($<10^{-9}$ s^{-1}); and**
- **to design and fabricate materials having specific elevated temperature properties based on microstructural predictions from the model.**

The results of constant strain-rate compression tests covering a range of temperatures from 250 to 550 °C and strain rates of 5×10^{-5} to 10^{-1} s^{-1} are presented in conjunction with microstructural investigations using transmission electron microscopy (TEM) and x-ray diffraction. Materials mechanically alloyed with (a) no dispersoids, (b) 23 nm radius TiO_2 dispersoids, and (c) 10 nm diameter Al_2O_3 dispersoids have been studied. The effect of varying the volume fraction of the TiO_2 dispersoids and adding alloying additions of Mg and Li to the matrix Al have been investigated. In addition, the TiO_2 particles are shown to have reacted to form Al_3Ti. An adaptation to the detachment model of Rösler and Arzt has been proposed to account for the behavior of these types of materials and to enable accurate prediction of deformation behavior at elevated temperatures and low strain rates.

Keywords aluminum alloys, dispersion strengthened, high temperature, mechanical behavior

1. Introduction

The demand for lightweight aluminum-based materials for medium strength elevated temperature aerospace applications has lead to a great deal of research and development being carried out on dispersion-strengthened alloys. Powder metallurgy routes enable the formation of finely dispersed phases that are resistant to coarsening that cannot be produced by conventional ingot metallurgy. Much of the work has centered on rapidly solidified powders where dispersoids of transition metals with low diffusivity in solid aluminum are produced *in situ*, such as Al-Fe-Ce alloys by Alcoa (PA, USA) and Al-Fe-V-Si alloys developed by Allied Signal (NJ, USA). While these materials have good elevated temperature strengths (Al-8Fe-1.4V-1.7Si is reported as having a yield strength of 184 MPa at 315 °C[1]), their use is limited by the hot workability. The strength of the materials deteriorates in each thermomechanical processing step after solidification due to microstructural coarsening at the high temperatures necessary for deformation.

The process of mechanical alloying overcomes the problems of high-temperature stability as dispersoids that are thermally stable and insoluble in aluminum can be added to the materials. It also enables great flexibility in the design of materials with chosen volume fractions, types, and sizes of dispersoids and of alloying additions.

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The results presented are derived from mechanically alloyed aluminum alloys strengthened with ultrafine (10 to 25 nm) thermally stable ceramic dispersoids. These materials have been found to have excellent room-temperature and elevated temperature properties (yield stresses greater than 350 MPa at 20 °C and greater than 200 MPa at 350 °C). This type of fine dispersion-strengthened material has been found to have unusual properties with exceptionally high stress exponents (greater than 20) and activation energies for deformation that are high and strongly dependent on temperature.[2]

While at low temperatures the deformation behavior can be related to the classical Orowan process, the mechanism controlling the elevated temperature strength remains unclear. At elevated temperature, the dislocations are able to overcome the dispersoids by climb, but this mechanism alone is insufficient to explain the high strengths observed experimentally.[3] In order to facilitate alloy design and enable extrapolation of laboratory tests into in-service conditions, it is necessary to have some type of physically based model to describe the behavior of the material. The model must allow the prediction of mechanical properties from physically based input parameters such as volume fraction of dispersoid, dispersoid size and morphology, temperature, stress, and strain rate.

Following transmission electron microscopy (TEM) observations where dispersoids appeared to pin dislocations on the departure side of the dispersoid as dislocations pass over them,[4] Rösler and Arzt have developed the detachment model.[5] The rate controlling mechanism for this model is the detachment of a dislocation from the departure side of an attractive dispersoid. The attractive force results from the reduction in the elastic energy of the dislocation at the incoherent particle interface. This occurs by diffusional relaxation at elevated temperatures,

Table 1 Nominal compositions of materials tested

Alloy Composition (wt. %)	Ceramic Additions (Vol. %)	Ceramic Radius (nm)
Al-1Mg-0.35Li	None	n/a
Al	10%Al_2O_3	13
Al	10%TiO_2	23
Al-1Mg-0.35Li	10%Al_2O_3	13
Al-1Mg-0.35Li	10%TiO_2	23
Al-1Mg-0.35Li	7.5%TiO_2	23

or by a reduction in the dislocation surface energy at the interface, and is represented by a factor *K*. Although the model fits some of the experimental data, it fails to represent the temperature dependence of deformation behavior and is extremely sensitive to the factor *K*, which cannot be measured experimentally.

The results of constant strain rate compression tests for a number of ultrafine dispersion-strengthened aluminum alloys are presented in this article. The effects of temperature, volume fraction, strain rate, dispersoid composition, and alloying additions are demonstrated. The behavior is compared to the detachment model, which is found to be inadequate at representing the material behavior. The detachment model has been adapted by the introduction of a variable *K* value that provides an improved correlation between the predicted and experimental properties.

2. Experimental

2.1 As-Received Materials

The materials studied were provided as consolidated billets by AMC (Farnborough, United Kingdom). The fabrication process involved mechanically alloying Al, Li, and Mg powders with different ceramic powders (Al_2O_3 with a mean diameter of 13 nm and TiO_2 with a mean diameter of 23 nm), followed by hot isostatic pressing into billets. These billets were hot extruded, at Imperial College, in an ENEFCO 5MN hydraulic vertical press, into rods with a diameter of 12 mm. The nominal compositions of these materials are given in Table 1.

2.2 X-Ray Diffraction

A computer-controlled Philips x-ray diffractometer (Philips Electronic Instruments Corp., Mahwah, NJ), with a PW 1050/25-model goniometer, was used with Cu K_a radiation to carry out phase identification of the ceramic powders and the extruded materials. The solid samples were spun during x-ray diffraction to avoid intensity reflections due to preferred orientations resulting from the extrusion process. The samples were scanned from $2\theta = 20$ to 100° in steps of 0.04°. The *d*-spacings corresponding to the resulting peaks were calculated using the Bragg equation.

2.3 Transmission Electron Microscopy

Longitudinal and transverse sections were cut from the extruded and deformed specimens and ground to a thickness of 150 μm. Three millimeter discs were spark eroded and then electropolished in a STRUERS TENUPOL with solution of 10% perchloric acid in ethanol at −50 °C and 15 V. The TEM was performed in a JEOL 120CX microscope (Japan Electron Optics Ltd., Tokyo) at 100 kV.

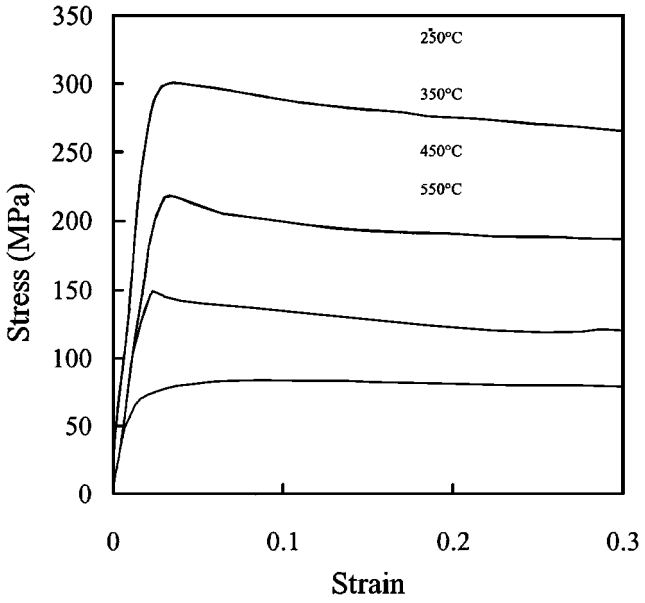

Fig. 1 Stress strain behavior of Al + Al_2O_3 tested at 0.1 s^{-1}

2.4 Mechanical Testing

Cylindrical compression samples measuring 10 mm in height and 8 mm in diameter were machined from the rods parallel to the extrusion direction. Constant strain rate compression tests were performed at 250, 350, 450, and 550 °C and constant strain rates of 0.1, 0.001, and 0.00005 s^{-1} on a hydraulic, 100 kN, computer-controlled Mayes (Leeds, UK) testing machine. An infra red (IR) furnace was used to heat the samples. Polytetraflouroethane (PTFE) tape was used as lubricant at the two lower testing temperatures and almost no barreling occurred in these cases. At the higher temperatures, boron nitride spray was used as lubricant and only very slight barreling of the samples was observed. The load was measured as a function of displacement and converted to provide true stress and strain information. The tests were stopped after a strain of 0.3. Typical results from the tests of the Al + Al_2O_3 material tested at a strain rate of 0.1 s^{-1} are shown in Fig. 1. After testing, the samples were water quenched to retain the dislocation structure.

3. Results and Discussion

3.1 Phase Distribution

Figure 2 shows part of the x-ray diffraction trace for the TiO_2 containing extrude and the TiO_2 powder added to the mechanical alloying process. It is clear that a reaction has occurred. The major nonaluminum peaks in the extruded material correspond to the Al_3Ti phase. The same behavior is observed in the materials mechanically alloyed with TiO_2 and

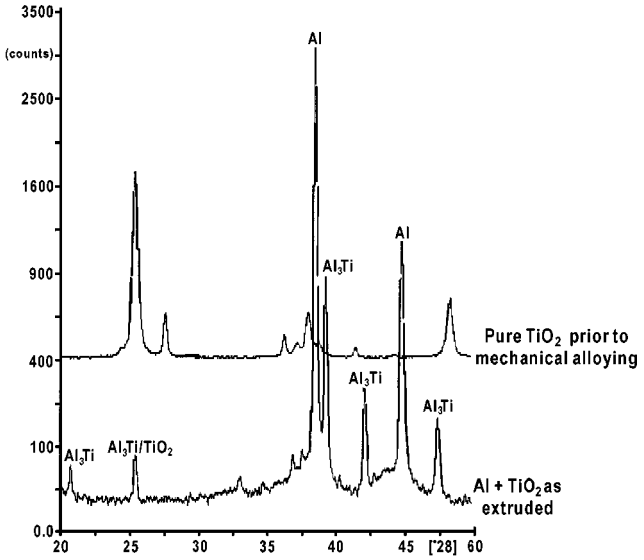

Fig. 2 X-ray diffraction spectra of TiO_2 powder used in the mechanical alloying process and the extruded alloys containing Ti

MgLi additions. The reaction $13Al + 3TiO_2 \rightarrow 2Al_2O_3 + 3TiAl_3$ leads to a large reduction in free energy of the system. The high temperatures used in the consolidation of the powders and the extrusion process result in diffusion rates sufficiently high so as not to impede the kinetics of the reaction.

Rösler *et al.*[6] have observed the substitution of Al_2O_3 by MgO in mechanically alloyed materials of Al and Al_2O_3 containing Mg. This is not discernible in the materials currently being investigated due the proximity of any such peaks to the large Al peaks. Due to the small size of the dispersoids, it has not been possible to directly analyze the dispersoid compositions *via* TEM.

3.2 Microstructure

The microstructures of the materials have been studied using TEM. The materials alloyed with TiO_2 contain dispersoids approximately 25 nm in diameter, and the materials alloyed with Al_2O_3 powder contain dispersoids approximately 15 nm in diameter. Figure 3 is a micrograph of the AlLiMg + Al_2O_3 material in the as-extruded condition. It shows a relatively homogenous distribution of spherical dispersoids with few dislocations. A fine substructure has been developed during extrusion consolidation with a subgrain size of ~300 nm. Figure 4 shows a micrograph of the 7.5% TiO_2 material following deformation at 350 °C and a strain rate of 0.001 s^{-1}. There is a high density of dislocations. This may account for the work softening behavior illustrated in Fig. 1. It is proposed that the dispersoids can act as prolific sources of mobile dislocations from the incoherent dispersoid matrix interface, which result in softening during deformation.

Figure 5 is a micrograph of the same sample as Fig. 4. It contains a dislocation configuration that appears to show pinning of the dislocation on the departure side of a dispersoid. This is in support of the rate controlling mechanism of the detachment model.

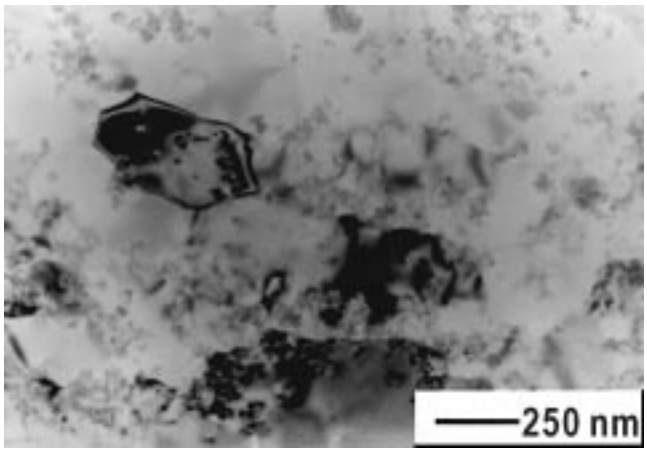

Fig. 3 TEM image of AlLiMg + Al_2O_3 as-extruded showing good distribution of dispersoid particles and fine substructure

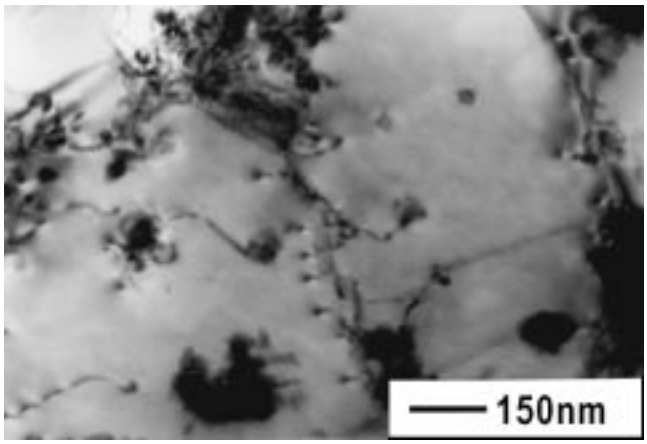

Fig. 4 TEM image of AlLiMg + 7.5% TiO_2 after deformation at 350 °C and 0.001 s^{-1} showing high density of dislocations

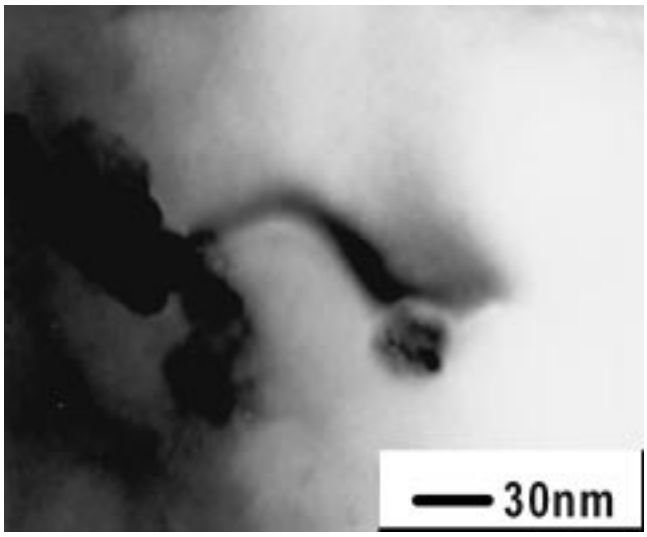

Fig. 5 TEM image of departure side pinning of a dislocation by a dispersoid

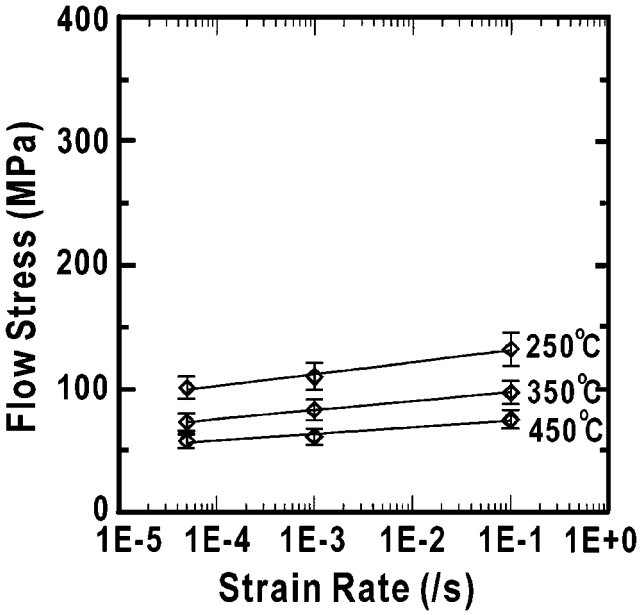

Fig. 6 Mechanical behavior of mechanically alloyed Al-Mg-Li powder containing no ceramic dispersoid

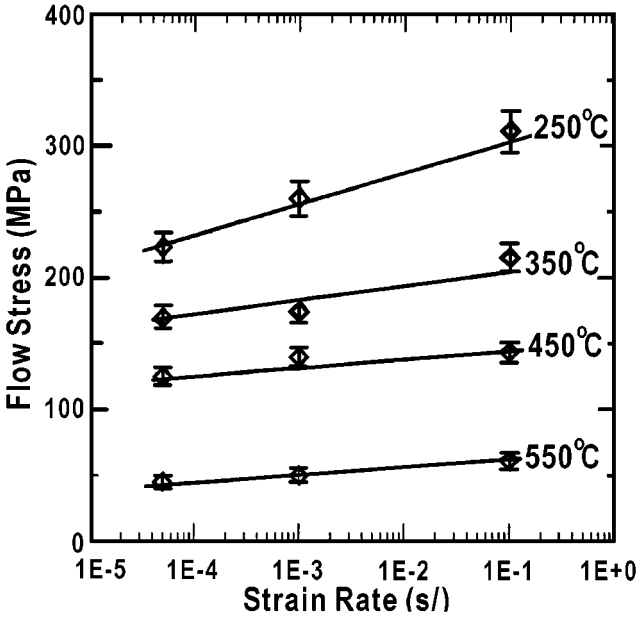

Fig. 7 Mechanical behavior of Al-Mg-Li + 10% Al_2O_3

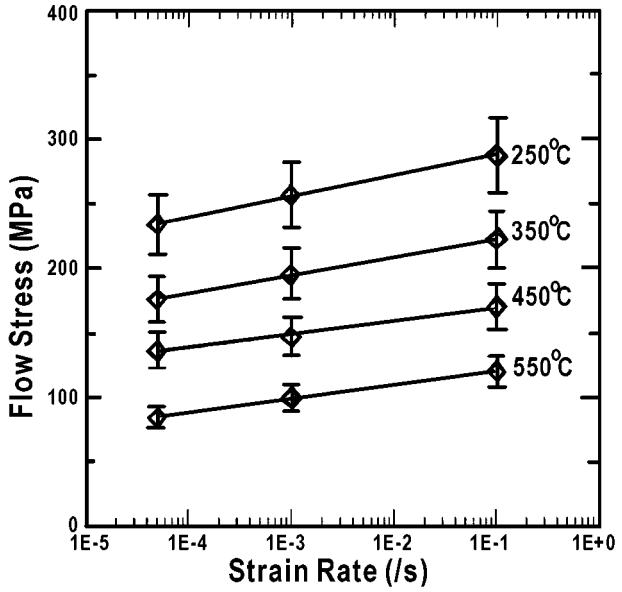

Fig. 8 Mechanical behavior of Al + 10% Al_2O_3

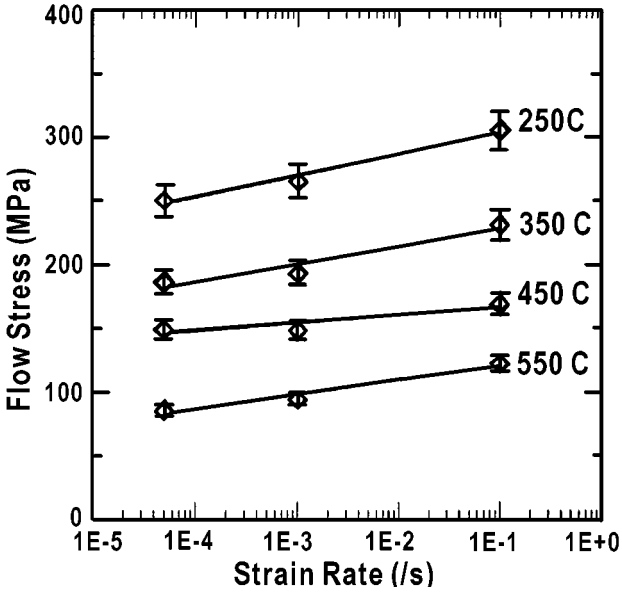

Fig. 9 Mechanical behavior of Al + 10% TiO_2

3.3 Mechanical Behavior

The results of the constant strain rate compression tests are shown in Fig. 6 to 11. All of the dispersion-strengthened materials show excellent elevated temperature properties with flow stresses greater than 150 MPa at 350 °C. The strength at this temperature is more than doubled when compared to the mechanically alloyed, unreinforced material.

The effect of the Mg-Li solute additions is considered in Fig. 12 and 13. From Fig. 12, it can be seen that the addition of Mg-Li to the material alloyed with TiO_2 has no effect. A small decrease is strength has been observed on alloying with Mg-Li additions and Al_2O_3 dispersoids at the highest temperatures and lower strains rates. A weakening effect has previously been observed by alloying Al + Al_2O_3 with Mg[3]. It would appear that this results from reaction between the Al_2O_3 dispersoids and Mg to change either the dispersoid size or the behavior of the dispersoid matrix interface. No such reaction occurs with the Al_3Ti dispersoids; thus, it may be concluded that the solute atoms have no observable effect at elevated temperature.

$$n = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma}\Big|_T \qquad \text{(Eq 1)}$$

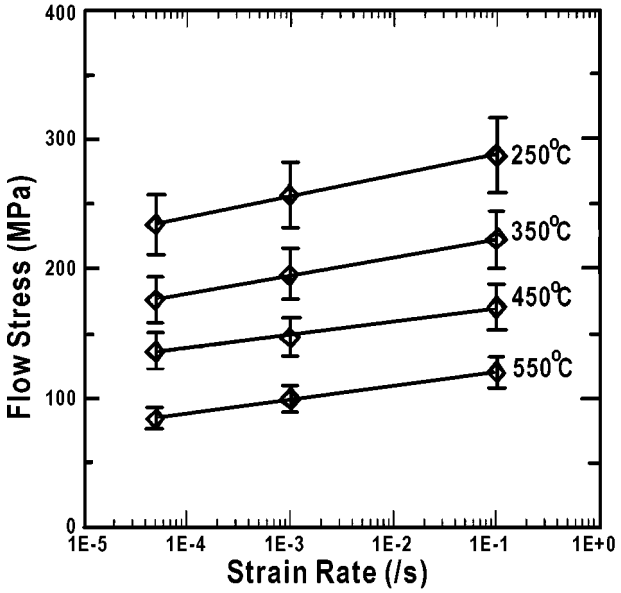

Fig. 10 Mechanical behavior of Al-Mg-Li + 10% TiO_2

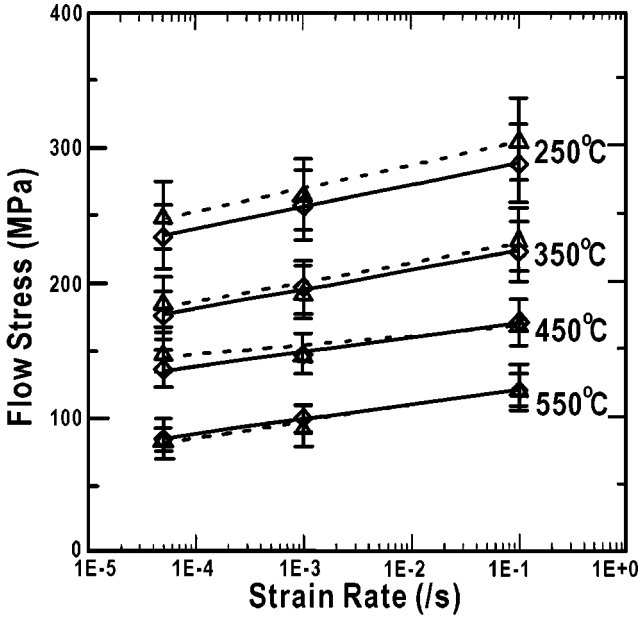

Fig. 12 Overlay of Fig. 9 (dashed lines) and 10 (solid lines)

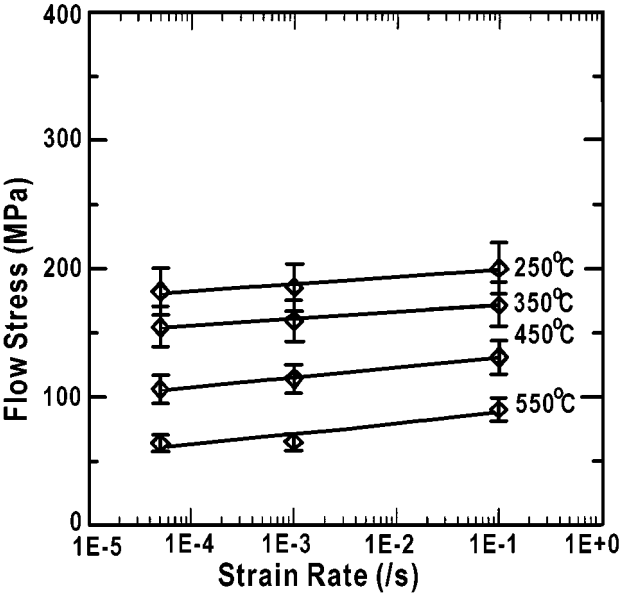

Fig. 11 Mechanical behavior of Al-Mg-Li + 7.5% TiO_2

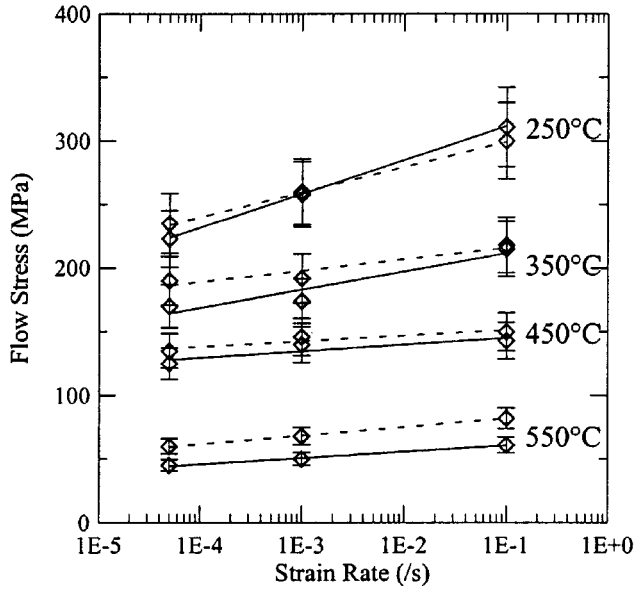

Fig. 13 Overlay of Fig. 7 (solid lines) and 8 (dashed lines)

As observed in similar materials,[7] n is found to be extremely high and to vary considerably with temperature, making traditional deformation equations inappropriate for modeling the behavior. The n values for the materials Al + 10% Al_2O_3, Al-Mg-Li + 10% Al_2O_3, and Al-Mg-Li + 10% TiO_2 are plotted against temperature in Fig. 14.

The behavior of the Al-Mg-Li + Al_2O_3 material has been compared to the detachment model using a K value of 0.8, as used previously for Al_2O_3 dispersoids by Rösler *et al.*[6] A dislocation density of 10^{13} has been used and other variables in the model have been taken to be the same as for Al. The results are plotted in Fig. 15. While the sensitivity of the strain rate to the stress is adequately predicted, the temperature dependence of the flow stress cannot be reproduced.

On further consideration, it would seem reasonable that K might vary with temperature due to variations of dislocation energy with temperature. The value of K relates the energy of a dislocation at a dispersoid to the energy of a dislocation in the matrix. $E_{\text{Dispersoid}} = KE_{\text{Matrix}}$. As the temperature of the material increases, more defects exist in the matrix so the strain energy of the dislocation in the matrix is reduced, increasing the K value. The strain energy at an incoherent interface would be affected less by an increase in temperature. At higher temperatures, there will also be more mobile dislocations. Increasing

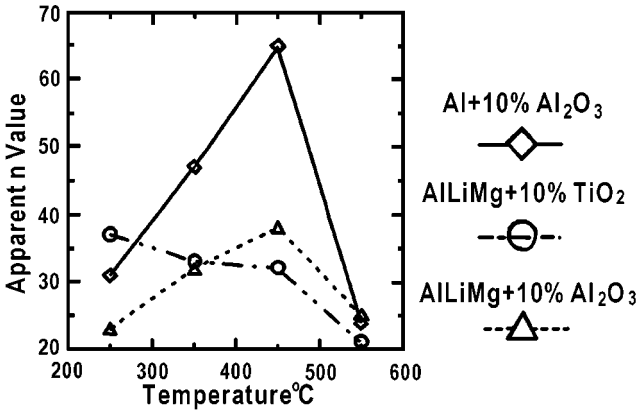

Fig. 14 Variation of stress exponent with temperature for two of the materials tested

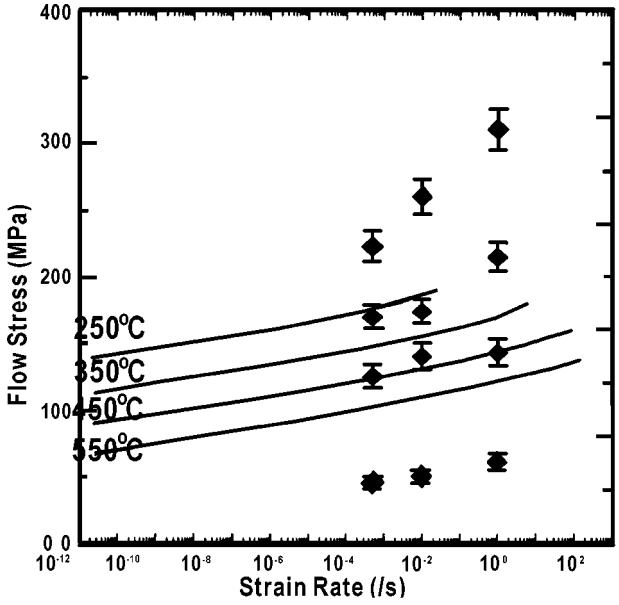

Fig. 15 Detachment model predictions (lines) and the experimental results for the Al-Mg-Li + 10% Al_2O_3 material

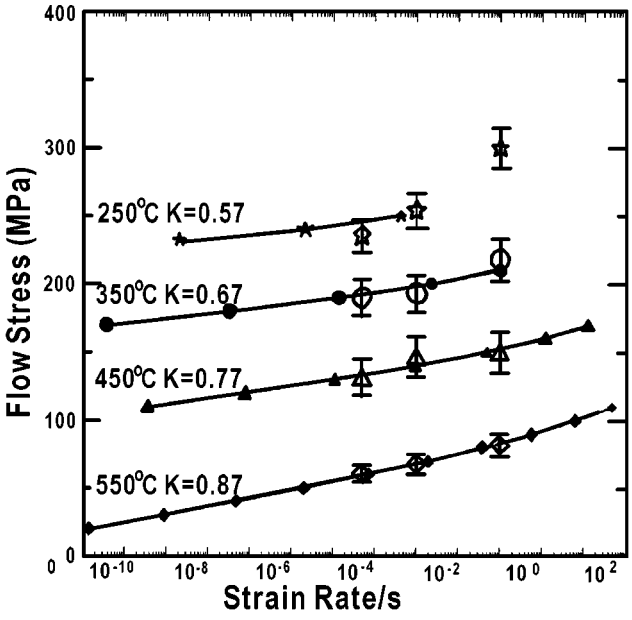

Fig. 16 Modified detachment model (lines) with variable *K* values and experimental results for the Al-Mg-Li + 10% Al_2O_3 material

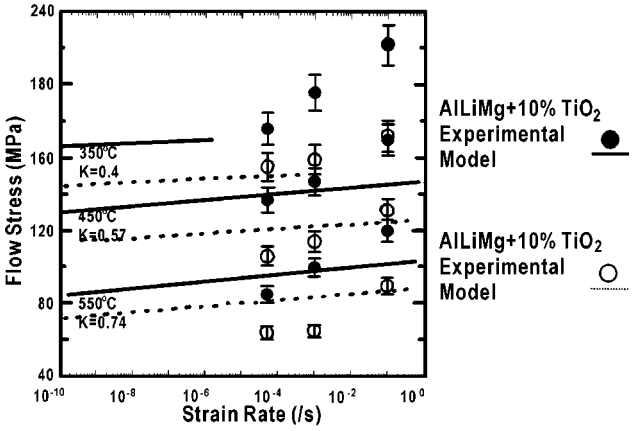

Fig. 17 Effect of volume fraction in TiO_2 containing material and the adapted detachment model

the mobile dislocation density with temperature in the model would also lead to an increased temperature dependence of the deformation stress. In order to fit the experimental data by changing dislocation density alone, it must increase by a factor of more than 10^{20}, which would be unrealistic.

It is proposed that the ratio of the energy of the dislocation at the dispersoid to the energy at the matrix decreases with temperature. By varying *K* linearly with temperature, a much improved fit with the experimental data is obtained. The fit of the experimental data to the modified detachment model with a variable *K* value, for the same material, is shown in Fig. 16. The fit is less satisfactory for the lowest temperature of 250 °C. At this temperature, the stress developed at the strain rates used is greater than the detachment stress, so this will no longer be the rate controlling mechanism.

The variation of *K* with temperature is different for the different dispersoids. The values for *K* have been determined for the Al-Mg-Li + 10% TiO_2 material by fitting the detachment model to the data. These *K* values have been substituted in the model for a volume fraction of 7.5% of 23 nm radius dispersoids and compared to the test results for the 7.5% TiO_2 containing material. Figure 17 shows the result. The solid lines are those used to determine *K* values for the 10% TiO_2 containing material. The dashed lines are the predicted results using these *K* values for the 7.5% TiO_2 containing material. The results for temperatures of 350 to 550 °C are shown, where the correlation to the experimental data (solid circles and hollow circles, respectively) is best.

While the fit of the model with the experimental data is still far from perfect, it is much improved on the original version of the detachment model. The strain rate dependence and the temperature dependence can be represented well using the

model, and the prediction of the effect of volume fraction is reasonably good.

5. Conclusions

The elevated temperature deformation behavior of the fine dispersion-strengthened materials tested appears to be extremely promising for elevated temperature applications.

Addition of solute atoms to these types of alloys seems to have little effect at the temperatures investigated, except when there is a reaction between the solute atoms and the dispersed phases.

Different dispersoid phases cause different deformation behavior. This may be due to the dispersoid size, or composition, or both.

The detachment model fails to predict the correct temperature dependence of these materials. However, by varying the *K* value in the detachment model linearly with temperature, a much improved fit with experimental data is obtained. While certain drawbacks of the detachment model still exist, for example, the fact that the *K* value cannot be measured or predicted directly, the new version of the model would seem to be the best currently available as a predictive tool for deformation behavior in these types of fine dispersoid-reinforced materials.

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