Inverse Hall-Petch behaviour in an AZ91 alloy and in an AZ91 – Al2O3 composite consolidated by high-pressure torsion

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Abstract

High-pressure torsion (HPT) is a significant procedure for achieving substantial grain refinement but it may be used also to consolidate metallic particles to form bulk samples or composites where two (or more) different phases are mixed and consolidated. An investigation was initiated to examine the consolidation of particles of the magnesium AZ91 alloy and a composite with an AZ91 matrix combined with 1% alumina powder. The results show it is possible to fully consolidate this alloy after a large number of turns. As a consequence of the severe plastic deformation, the grain structure was significantly refined with average grain sizes of ~116 and ~98 nm in the unreinforced alloy after 20 or 50 HPT turns or ~76 nm in the composite after 50 HPT turns, respectively. This grain refinement is associated with a decrease in hardness and an increase in the strain rate sensitivity due to the onset of a grain boundary diffusion-assisted creep mechanisms at room temperature. The results are consistent with the theoretical prediction of a breakdown in the Hall-Petch relationship at very small grain sizes.

Keywords: composites; Hall-Petch breakdown; high-pressure torsion; magnesium; particle consolidation.

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**1. Introduction**

High-pressure torsion (HPT) is a severe plastic deformation technique that has been widely used to process bulk metallic materials promoting grain refinement and increased strength. In addition to the processing of bulk samples, HPT has been used also to process particles and powders by promoting their consolidation. Early reports examined the consolidation of pure aluminum and copper [[1](#_ENREF_1), [2](#_ENREF_2)], a nanocrystalline alloy [[3](#_ENREF_3), [4](#_ENREF_4)] and amorphous alloys [[4](#_ENREF_4), [5](#_ENREF_5), [6](#_ENREF_6), [7](#_ENREF_7), [8](#_ENREF_8)]. It is also possible to incorporate hard phase particles into the metals producing metal-matrix composites [[1](#_ENREF_1), [2](#_ENREF_2), [9](#_ENREF_9), [10](#_ENREF_10), [11](#_ENREF_11), [12](#_ENREF_12), [13](#_ENREF_13)] which usually display improved strength compared to their metal counterparts.

There is a great interest in increasing the strength of magnesium to produce a material with a high strength-to-density ratio which could be used to produce lighter components. There are recent reports on the consolidation of magnesium by HPT [[14](#_ENREF_14), [15](#_ENREF_15), [16](#_ENREF_16), [17](#_ENREF_17), [18](#_ENREF_18)] and a Mg-10% Al2O3 composite with improved hardness compared to pure magnesium [[19](#_ENREF_19)]. Nevertheless, the hardness of the Mg-Al2O3 composite (~65 Hv) was relatively low compared to conventional magnesium-based alloys. The low strength of pure magnesium matrix composites may be attributed to the existence of an inverse Hall-Petch behavior in this material [[20](#_ENREF_20)] which limits the increase in strength due to grain refinement. Such break in the Hall-Petch relationship has not been reported in a magnesium alloy so far. A recent review of the HPT processing of magnesium and its alloys showed that hardness values in the range of ~120 – 130 Hv are obtained in commercial alloys after HPT processing [[21](#_ENREF_21)]. Thus, higher hardness is expected in a reinforced composite with a magnesium alloy matrix. In fact, a Mg-2% Zn alloy matrix composite, produced by melting, and then processed by HPT [[22](#_ENREF_22)], displayed a higher hardness than a pure magnesium matrix composite. Accordingly, the present research was designed to investigate the potential for producing a magnesium alloy matrix composite reinforced with hard particles using cold-consolidation by HPT and to evaluate the validity of the Hall-Petch relationship in the ultrafine-grained range in a representative magnesium alloy.

**2. Experimental material and procedures**

The materials used in the present experiments were an AZ91 alloy and Al2O3 as coarse and fine particles, respectively. Figure 1 shows scanning electron microscopy (SEM) images of the two starting materials and this shows that the particles of AZ91 were elongated with the major axis having hundreds of microns whereas the individual Al2O3 particles were much finer than 1 micron but with the presence of some agglomeration.

Initial attempts to produce a composite with 10% (in weight) of the reinforcement phase led to significant agglomeration of the alumina phase and generally a poor consolidation of the AZ91 matrix. Therefore, an AZ91-1% Al2O3 (in weight) mix was prepared using a rotating container, which provided mixing of the particles without deformation, and then cold pressing at ~400 MPa into discs of ~1 mm thickness and ~10 mm diameter. These discs were processed through a total *N*, of 50 turns of HPT under a nominal pressure of 6.0 GPa using quasi-constrained anvils [[23](#_ENREF_23)]. A rotation rate of 2 rpm was used. A similar procedure was used to produce discs of AZ91 without a reinforcement phase which were processed to either 20 or 50 turns of HPT, respectively.

The consolidation was evaluated by observing the polished longitudinal sections of the discs using optical microscopy (OM) and SEM. The discs were ground using abrasive papers (#600, #1000 and #4000), polished using diamond pastes (3 µm and 1 µm) and a final polishing step using colloidal silica solution (OPS) provided mirror-like finishes Dynamic hardness tests (using Shimadzu DUH-211 equipment) were used to determine the hardness and the overall creep behaviour at room temperature. A Berkovich indenter was used in these tests and the load, time and penetration depth were tracked. The hardness was determined without any dwell time using a maximum load of 100 mN and a loading rate of 13 mN/s. The creep behaviour was evaluated using a maximum load of 300 mN, loading rate of 70 mN/s and a dwell time of 1000 s. The indenter depth, the time and the instantaneous hardness were used to estimate the instantaneous stress and strain rate. Detailed descriptions of the procedures are given elsewhere [[24](#_ENREF_24), [25](#_ENREF_25), [26](#_ENREF_26)]. Additional Vickers hardness tests were carried out using a load of 50 gf and a dwell time of 10 s. These hardness tests were performed along the longitudinal plane and care was taken to track the distance of each indentation to the disc centre and bottom of the disc. The mean of at least 5 indentations recorded at different positions having the same distance to the disc centre and the standard deviation were calculated to estimate hardness evolution as a function of the imposed strain.

The microstructures of the HPT-processed discs were examined using transmission electron microscopy (TEM). Transparent lamellae were prepared using focus ion beam (FIB) milling and they were extracted at areas ~4 mm from the disc centres. The grain size distributions were determined by measuring the mean diameter of at least 150 individual grains.

**3. Experimental results**

Observations using OM revealed full consolidation of the AZ91-1% Al2O3 composite and the pure AZ91 discs. The boundaries between the initial AZ91 coarse particles, which were visible after low numbers of turns of HPT[[17](#_ENREF_17)], became indistinguishable after 20 turns of HPT processing for the pure AZ91 discs and after 50 turns for the composite. Figure 2 shows SEM images of the longitudinal sections at areas near the mid-radius positions of the AZ91 and the AZ91-1% Al2O3 work-pieces after processing through 50 HPT turns. Both materials display a continuous matrix with β-phase precipitates but the composite also exhibits dispersed alumina particles. Some agglomeration of the alumina particles takes place in isolated areas. Both the β-phase and the alumina particles are reasonably dispersed throughout the metallic matrix and the separations between them are within the range of several microns.

Figure 3 shows representative TEM images of the grain structure of the AZ91 samples processed to 20 and 50 turns and the AZ91-1% Al2O3 composite processed to 50 turns. The grain refinement in the AZ91 alloy does not saturate after 20 turns and there is a slight additional decrease in grain size with further processing to 50 turns. The distribution of grain sizes for these materials is presented in Fig. 4 in terms of the number frequency plotted against the grain diameter. The AZ91 alloy and the composite contain grains within the range of ~20-220 nm but there is a clear trend of decreasing grain size with increasing numbers of rotations in the AZ91 alloy. The majority of grains lies within the range of ~80-140 nm after 20 turns and within the range of ~60-120 nm after 50 turns. The AZ91-1% Al2O3 composite exhibits the finest structure with most of the individual grain diameters within the interval of ~40-100 nm. The average grain sizes were 116 ± 40 nm and 98 ± 35 nm for the AZ91 alloy after 20 and 50 turns of HPT and 76 ± 40 nm for the composite after processing through 50 turns of HPT, respectively.

Figure 5 presents colour-coded displays of the hardness distributions along longitudinal sections of the discs for different experimental conditions. These distributions tend to be reasonably homogeneous with isolated areas of hardness slightly different from their surroundings. A comparison of the two upper distributions shows that the hardness of the AZ91 alloy decreases after 50 turns compared to the 20 turns material. This is unexpected due to the higher imposed strain and the finer grain size after 50 turns. Also unexpectedly, the hardness of the composite is significantly lower than the unreinforced alloy.

In order to ease the observation of any trends in the hardness evolution with increasing torsional straining, the hardness values determined using Vickers hardness and dynamic hardness testing are plotted as a function of the strain as shown in Fig. 6 where the strain was estimated as the effective strain in torsion [[27](#_ENREF_27)]. From these plots, there is a clear trend of decreasing hardness with increasing strain for both the alloy and the composite and this tendency is observed in both (a) the dynamic testing and (b) the Vickers hardness measurements. Furthermore, the hardness of the composite is consistently lower than the unreinforced alloy at all strains.

The flow stress is plotted as a function of the strain rate in Fig. 7 for room temperature indentation creep tests in the AZ91 alloy processed to 20 turns and for the composite processed to 50 turns. The flow stress of the composite is lower compared to the AZ91 alloy which is consistent with the hardness tests despite the finer grain structure in the composite. Both materials exhibit high strain rate sensitivities, *m*, with values over 0.060. An earlier report documented a value of *m* ≈ 0.004 for the room temperature strain rate sensitivity of the AZ91 alloy [[28](#_ENREF_28)] which is over one order of magnitude lower than the values observed in the nanostructured materials in the present investigation.

**4. Discussion**

The results from these tests show that it is feasible to consolidate particles of a magnesium AZ91 alloy into a bulk disc after 20 turns and to incorporate hard ceramic particles to produce a composite after 50 turns of HPT. Earlier attempts to consolidate machining chips of the AZ91 alloy combined with 10% Al2O3 particles failed to produce dense billets after only 5 turns of HPT [[17](#_ENREF_17)]. However, in a later investigation it was shown that pure magnesium chips and alumina particles can be consolidated into a bulk sample after 5 turns of HPT [[19](#_ENREF_19)]. In practice, there are many reports of the consolidation of aluminum and copper powders, with and without reinforcement particles, after 5 turns of HPT [[1](#_ENREF_1), [2](#_ENREF_2), [29](#_ENREF_29), [30](#_ENREF_30)]. This demonstrates, therefore, that the AZ91 alloy is more difficult to consolidate than pure magnesium, pure aluminium and pure copper but nevertheless a full consolidation may be attained by imposing larger numbers of HPT turns.

It is generally accepted that the grain refinement in magnesium alloys saturates after a few turns and this is associated with a saturation in the hardness evolution. However, the present results show that grain refinement continues even after 20 turns of HPT in the AZ91 alloy. Moreover, the grain size in the composite is smaller than in the unreinforced metal processed to the same number of turns. It is known that the presence of hard particles induces the formation of local flow heterogeneities [[31](#_ENREF_31), [32](#_ENREF_32)] and strain gradients [[33](#_ENREF_33)] which these may increase the overall deformation in the matrix. Therefore, the matrix in the AZ91 + 1% Al2O3 composite undergoes a larger plastic deformation than the unreinforced alloy counterpart despite the similar numbers of turns in HPT. As a consequence, the composite exhibits a finer grain structure which agrees with the tendency for increasing grain refinement with increasing strain in the AZ91 alloy.

The widely known Hall-Petch relationship states that the material strength increases with grain refinement. Nevertheless, the current experiments, as documented in Figs 5 and 6, clearly show the opposite trend. Specifically, the hardness of the AZ91 alloy decreased after 50 turns compared to the sample processed to 20 turns and the former displayed a finer grain size than the latter. Also, the composite, although having the finest grain structure, displays the lowest hardness. This clearly demonstrates a breakdown in the conventional Hall-Petch relationship. It is worth noting that the incorporation of hard ceramic particles was expected to increase the strength of the material. However, the distance between the particles is many times larger than the grain size in the processed composite. Therefore the grain boundaries are the major barrier to slip propagation and the Al2O3 particles are not expected to significantly affect the hardness.

Such a breakdown at room temperature has been reported in pure magnesium for much larger grain sizes and attributed to the onset of grain boundary sliding [[20](#_ENREF_20), [24](#_ENREF_24)]. For example, it was found that the activation energy for grain boundary diffusion was significantly lower than expected and this gave an enhanced grain boundary diffusivity. Furthermore, a decreasing hardness with decreasing grain size is expected in nanocrystalline materials due to the occurrence of diffusional creep [[34](#_ENREF_34)] but this effect has not been observed in a magnesium alloy processed by HPT. A recent review showed that Hall-Petch holds for magnesium alloys and a reverse Hall-Petch was not considered due to the difficulties in obtaining magnesium with very fine grains [[35](#_ENREF_35)].

In order to evaluate the occurrence of a Hall-Petch breakdown due to grain boundary sliding and/or diffusional creep, the flow stress, *σ*, of the AZ91 alloy was collected from numerous reports [[36](#_ENREF_36), [37](#_ENREF_37), [38](#_ENREF_38), [39](#_ENREF_39), [40](#_ENREF_40), [41](#_ENREF_41), [42](#_ENREF_42), [43](#_ENREF_43), [44](#_ENREF_44)] and then plotted as a function of the grain size, *d*, in Fig. 8 for a broad range of grain sizes. With the objective of comparing the Vickers hardness values, *Hv*, and the flow stress, σ, determined by tensile tests, the relationship of *σ = Hv/3* was used to convert hardness to stress [[45](#_ENREF_45)]. Although this relationship may tend to overestimate the flow stress calculated from hardness data for large grains, this discrepancy decreases with decreasing grain size and there tends to be an excellent agreement for grain sizes <1 µm [[40](#_ENREF_40)]. As shown in Fig. 8, there is a general trend of decreasing flow stress with increasing grain size for large grain sizes and this is in agreement with the Hall-Petch relationship. Accordingly, a trend line for this relationship was plotted in Fig. 8 using the following expression:

(eq. 1)

Thus, the Hall-Petch relationship is no longer followed for grain sizes <1 µm where the slope changes in Fig. 8. It is worth noting that a decrease in the Hall-Petch slope for grain sizes < 2 µm was reported for an AZ31 alloy [[46](#_ENREF_46)]. Moreover, a deviation from the Hall-Petch trend for a sample with the finest grain size (0.33 µm) was reported in an AZ91 alloy processed by rapid solidification [[44](#_ENREF_44)]. The data in Fig. 8 shows the flow stress does not vary significantly for grain sizes in the range from ~0.1 to 1.0 µm and instead there is an inverse trend of decreasing flow stress with decreasing grain size for grain sizes that are smaller than ~100 nm.

An inverse Hall-Petch behaviour has been reported in different materials and different mechanisms were proposed to explain this effect [[47](#_ENREF_47), [48](#_ENREF_48), [49](#_ENREF_49)]. Two widely known creep mechanisms which predict a decrease in stress with decreasing grain size are Coble creep and grain boundary sliding. The theoretical predictions for these mechanisms are also included at the smaller grain sizes in Fig. 8 for comparison purpose. These predictions were based on creep mechanisms developed for Coble diffusion creep where vacancy flow occurs along the grain boundaries [[50](#_ENREF_50)] and grain boundary sliding [[51](#_ENREF_51)] and the relevant relationships are given as follows:

(eq. 2)

(eq. 3)

where *k* is Boltzmann’s constant, *T* is the absolute temperature, *δ* is the grain boundary width, *Dgb­* is the grain boundary diffusion coefficient, *b* is the Burgers vector modulus and *G* is the shear modulus, and and are the strain rates for the Coble and grain boundary sliding mechanisms, respectively. A reasonable and relevant strain rate of 10-4 s-1 was taken for both mechanisms and for this strain-rate the Hall-Petch relationship is expected to be valid for grain sizes of ~200 nm and larger. The flow stresses for the creep mechanisms are lower for nanostructured alloys and this directly explains the inverse trends observed in the present experiments.

Also, although coarse-grained magnesium displays a low strain-rate sensitivity at room temperature, the creep mechanisms are associated with relatively high values of *m*. The present experiments show there is a significant increase in the strain rate sensitivity which also corroborates the contribution of creep to room temperature plastic deformation for the nanostructured alloy.

A similar trend was reported in pure magnesium processed by ball milling and consolidated through hot extrusion [[52](#_ENREF_52)]. Thus, compression tests showed that the Hall-Petch relationship was valid for grains larger than ~1 µm but there was a reduction in slope for grains within the range of ~100 nm – 1 µm and then a negative slope for grain sizes below ~100 nm. An increase in the strain rate sensitivity was reported up to ~0.08 with decreasing grain size.

The continuous decrease in hardness with increasing deformation in HPT is associated with the continuous decrease in grain size which activates grain boundary diffusion assisted creep mechanisms such as grain boundary sliding and Coble creep. A similar reduction in hardness after very high straining in HPT was reported in a Mg-3.4% Zn alloy with a grain size of ~140 nm [[53](#_ENREF_53)]. This suggests that the breakdown in the Hall-Petch relationship at grain sizes in the range of ~100 nm is also valid for other magnesium alloys. By contrast, a higher strength was reported in a Mg-2% Zn alloy / 14% SiC composite processed by HPT despite a very small grain size of ~64 nm compared to the unreinforced alloy with a grain size of ~105 nm [[22](#_ENREF_22)]. Although this appears to contradict the present results, the composite displayed a homogeneous distribution of nano-precipitates which may hinder grain boundary sliding and thereby extend the range for conventional Hall-Petch behaviour. The composite in the current investigation contained fewer hard particles and the separation between them was very high. This suggests they were not effective in restraining any grain boundary sliding. Thus, it appears that the major effect of the hard particles in the present experiments is to increase the amount of deformation imposed to the matrix due to local flow heterogeneities.

**5. Summary and conclusions**

1. Bulk discs of AZ91 and AZ91 + 1% Al2O3 were successfully produced by consolidation of particles through high-pressure torsion. The number of turns required for consolidation was higher than in pure magnesium and other metallic materials.

2. The grain size decreases continuously with increasing deformation and a minimum grain size of ~76 nm was achieved in the composite after 50 turns of HPT.

3. The hardness decreases with increasing strain and the strain rate sensitivity increases. It is shown that these effects are associated with the onset at room temperature of a grain boundary diffusion-assisted creep mechanisms such as Coble diffusion creep or grain boundary sliding.

**6. Acknowledgements**

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Figures:



Figure 1 – Particles of AZ91 and Al2O3 used to produce the composites.



Figure 2 – SEM images of the mid-radius area of the AZ91 alloy and the AZ91 + 1% Al2O3 composite processed to 50 turns of HPT.



Figure 3 – Representative TEM images of the grain structure of the AZ91 alloy processed to 20 and 50 turns and the AZ91 + 1% Al2O3 composite processed to 50 turns of HPT.



Figure 4 – Grain size distribution of the AZ91 alloy and the AZ91 + 1% Al2O3 composite after HPT consolidation and processing.



Figure 5 – Hardness distribution along the longitudinal section of the AZ91 alloy and the AZ91 + 1% Al2O3 composite.

(a)

(b) 

Figure 6 – (a) Dynamic hardness and (b) Vickers hardness plotted as a function of the effective strain for the AZ91 alloy and the AZ91 + 1% Al2O3 composite.



Figure 7 – Flow stress as a function of the strain-rate determined by room temperature creep testing of the AZ91 alloy and the AZ91 + 1% Al2O3 composite.



Figure 8 – Flow stress plotted as a function of the grain size [36-44].