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Superplastic behaviour of AZ91 magnesium alloy processed by high-pressure torsion

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Abstract

An investigation has been conducted on the tensile properties of a fine–grained AZ91 magnesium alloy processed at room temperature by high pressure torsion (HPT). Tensile testing was carried out at 423 K, 473 K and 573 K using strain rates from $1 \times 10^{-1}$ s$^{-1}$ to $1 \times 10^{-4}$ s$^{-1}$ for samples processed in HPT for $N = 1, 3, 5$ and 10 turns. After testing was completed, the microstructures were investigated by scanning electron microscopy and energy dispersive spectroscopy. The alloy processed at room temperature in HPT exhibited excellent superplastic behaviour with elongations higher than elongations reported previously for fine–grained AZ91 alloy produced by other severe plastic deformation processes, e.g. HPT, ECAP and EX–ECAP. A maximum elongation of 1308 % was achieved at a testing temperature of 573 K using a strain rate of $1 \times 10^{-4}$ s$^{-1}$, which is the highest value of elongation reported to date in this alloy. Excellent high–strain rate superplasticity (HSRSP) was achieved with maximum elongations of 590 % and 860 % at temperatures of 473 K and 573 K, respectively, using a strain rate of $1 \times 10^{-2}$ s$^{-1}$. The alloy exhibited low–temperature superplasticity (LTSP) with maximum elongations of 660 % and 760 % at a temperature of 423 K and using strain rates of $1 \times 10^{-3}$ s$^{-1}$ and $1 \times 10^{-4}$ s$^{-1}$, respectively. Grain–boundary sliding (GBS) was identified as the deformation mechanism during HSRSP, and the glide–dislocation creep accommodated by GBS dominated during LTSP. Grain–boundary sliding accommodated with diffusion creep was the deformation mechanism at high test temperature and slow strain rates. An enhanced thermal stability of the microstructure consisting of fine equiaxed grains during deformation at elevated temperature was attributed to the extremely fine grains produced in HPT at room temperature, a high volume fraction of nano β–particles, and the formation of β–phase filaments.

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Keywords

AZ91 magnesium alloy, High pressure torsion, Microstructure, Superplasticity
Introduction

Magnesium alloys are widely used in many applications such as transportation, materials–handling, and commercial equipment due to their low density compared to other structural alloys such as steel and aluminium alloys [1]. However, the main limitation in the use of these alloys is their poor workability at room temperature, which makes mechanical processing difficult. The low ductility of magnesium alloys is a result of the hexagonal crystal structure of magnesium [2]. It has been found that severe plastic deformation can improve the strength and ductility of many materials including magnesium alloys [3]. Hence, several attempts have been made to enhance their mechanical properties via these processes, but the majority of these experiments have been conducted at elevated temperature, where dynamic recrystallization and grain growth take place [2,4–6]. Ultrafine–grained magnesium alloys have the potential to be used in automotive and aerospace applications due to their lightweight, a high ratio of strength–to–weight, and consequently, reduced fuel consumption [7,8]. The development of superplastic behaviour of ultrafine–grained magnesium alloys has been attracting significant attention in the last decade. Among magnesium alloys, the AZ91 alloy is widely used in industry. This alloy has a good machinability and castability, high strength–to–density ratio, and good corrosion resistance [7,9]. Fine–grained AZ91 alloy has been produced through several severe plastic deformation (SPD) techniques, such as ECAP [2,4], EX–ECAP [10] and HPT [11]. Superplasticity behaviour in any material requires a fine grain size ($\leq 10 \mu m$), and typically a high temperature ($T > 0.5T_m$, where $T_m$ is the absolute melting point of the material). Fine–grained AZ91 magnesium alloys have shown a wide range of superplasticity depending on strain rate and testing temperature. ECAP processing at 448 K in AZ91 alloy resulted in a superplastic elongation of 661 % at a temperature of 473 K using a strain rate of $6\times10^{-4}$ s$^{-1}$ [2]. Superplasticity behaviour was found in the same alloy processed by EX–ECAP at 473 K with elongation of 800 % at temperature of 423 K using a strain rate of $6\times10^{-4}$ s$^{-1}$ [2]. HPT processing at 423 K of a Mg–9%Al alloy resulted in elongation of 810 % at a temperature of 473 K using a strain rate of $5\times10^{-4}$ s$^{-1}$ [11]. It can be seen then that earlier work on superplasticity in SPD processed AZ91 alloy has been based on processing of the AZ91 alloy at high temperatures, where the limited ductility and workability of the alloy has been improved by the activation of additional slip systems and dynamic recrystallization [12]. So far, no experiment has been reported which investigates the superplastic behaviour of the AZ91 alloy processed by HPT at room temperature. The current investigation focuses on superplastic behaviour and the thermal stability of the AZ91 magnesium alloy that have been processed at room temperature, and tested over different temperatures and strain rates.

Experimental procedure

An extruded 10 mm rod of AZ91 magnesium alloy (Mg–9%Al–1%Zn) was used in this study, which was supplied by Magnesium Elektron Co. (Manchester, UK). Thin disks were made of the extruded rod with thicknesses of 1.5 mm and then ground to final thicknesses of 0.85 mm using abrasive paper. The HPT processing was conducted at room temperature using a HPT facility that has been previously discussed in detail elsewhere [13]. This facility consists of upper and lower
anvils and a circular depression of 0.25 mm in depth and 10 mm in diameter, that is located centrally in both anvils. The HPT processing was conducted under a quasi–constrained condition at room temperature and at a speed of 1 rpm using an applied pressure of 3.0 GPa for differing numbers of turns \((N)\): 1, 3, 5, and 10 turns. The peripheries of both anvils were coated with a lubricant containing MoS\(_2\) to avoid any possible damage to these areas during processing. The as–received and the processed alloys were carefully ground, polished and etched in acetic–glycol solution (20 ml acetic acid + 19 ml water + 1 ml nitric acid + 60 ethylene glycol) then the microstructures observed using scanning electron microscopy (SEM, JEOL JSM–6500F, Japan). Small disks of 3 mm diameter were punched from the processed HPT disks and these small disks were ground to a thickness of 150 \(\mu\)m and then thinned using a twin–jet electro–polishing facility (Struers Tenupol–3) with a solution of 15 ml perchloric acid, 15 ml glycerol, and 70 ml ethanol. Subsequently a transmission electron microscope (TEM, JEOL JEM–3010) was used for microstructural observation of the alloy after HPT processing. For tensile tests, the processed disks were carefully ground to thicknesses of 0.6 mm using an abrasive paper to prepare them for cutting into micro–tensile samples as previously reported elsewhere [14]. The method of cutting these micro–tensile samples from the HPT disks is known as the off–centre position method and it is used to avoid the central region in an HPT disk where structural heterogeneity is anticipated after HPT. This allows production of two miniature tensile samples per HPT disk with dimensions of \((1.0\times0.9\times0.6)\ mm^3\) as measured by the optical microscope (OLYMPUS–BX51, Japan). The tensile test was conducted at initial strain rates between \(1\times10^{-1}\ s^{-1}\) to \(1\times10^{-4}\ s^{-1}\) at each testing temperature of 423, 473, and 573 K, and for tensile samples produced from disks processed for \(N = 1, 3, 5\) and 10 turns. For better accuracy, each tensile test evaluation of a particular processing condition was conducted using two tensile samples and hence 96 tensile samples were used in this investigation. Tensile testing was carried out using a Zwick/Roell tension (Z030, Germany) machine operating at a constant rate of crosshead displacement. The load and displacement were gathered using testIIXpert testing software in a computer–acquisition system. Curves of engineering stress versus elongation and elongation–to–fracture versus strain rate were plotted. The flow stress versus strain rate curves were plotted to measure the values of strain–rate sensitivity \((m)\). The microstructures of tensile samples after tests were observed at the gauge section surfaces using scanning electron microscopy. In order to investigate the surface morphology in detail, the as–tested samples were observed without any further metallographic preparation. The average grain size was measured from these SEM images using a linear intercept method and then corrected by a factor of \(1.74\). The variation in grain size versus temperature and time at test temperature was assessed for samples processed in HPT for \(N = 1\) and 10 turns and tested in tension at different strain rates. The chemical composition of the matrix and the secondary phase were analysed using energy dispersive spectroscopy (EDS). To calculate the values of elongations, the final lengths of the tensile samples after the test were measured under the optical microscope. Representative images of the untested and fractured tensile samples were produced using a low–magnification optical microscope (Wild Herrbrugg, Switzerland), and these images show the testing temperature and strain rate used in tensile testing and the resultant percentages of elongations.
Results

The microstructure of the AZ91 magnesium alloy prior to and after HPT processing is shown in Fig. 1 as observed using SEM and TEM. The chemical analysis with weight fractions of alloying elements in the as-received alloy as obtained by EDS is shown in Fig. 2. The microstructure of the as-received alloy shows an average grain size of 30 µm as shown in Fig. 1 (a), where the microstructure consists of two main phases: α–Mg matrix; and lamellar and agglomerate forms of β–phase with the presence of Al₈Mn₅ particles as confirmed by EDS analysis and illustrated by the weight fractions found in Fig. 2. It is obvious from Fig. 1 (a) that the grain boundaries between α–Mg grains are covered with the β–phase. The magnesium matrix appears darker than the β–phase, which appears brighter as shown in Fig. 1 (a). After HPT processing, the microstructure altered noticeably with significant grain refinement and the original decoration of the grain boundaries by β–phase disappeared with increasing number of turns (up to \( N = 10 \) turns) as shown in Fig. 1 (b,c). The β–phase fragmented into fine particles and aligned along the direction of the torsional straining. The microstructure shows a significant level of grain refinement even at the beginning of HPT processing with grain sizes down to nanometre scale as observed using TEM as shown in Fig. 1 (d).

It is expected that the grain refinement for the current alloy continued gradually during HPT processing as indicated indirectly by microhardness measurements that have been done in previously reported work for the same alloy [15] and for AZ31 magnesium alloy also processed by other workers at room temperature [16]. The engineering stress–strain behaviour of fine–grained AZ91 magnesium alloy is shown in Fig. 3, where samples have been processed in HPT for \( N = 10 \) turns and then pulled to fracture at different testing temperatures of 423 K, 473 K, and 573 K using initial strain rates of \( 1 \times 10^{-1} \) s\(^{-1}\) to \( 1 \times 10^{-4} \) s\(^{-1}\). The testing temperatures used in this investigation corresponded to 0.55 \( T_m \) (423 K), 0.61 \( T_m \) (473 K), and 0.74 \( T_m \) (573 K), where the absolute melting point of the AZ91 alloy is 768 K (495 °C) [1]. These curves show the occurrence of superplastic elongation: with increasing number of turns in HPT, at slower tensile rates, as well as at higher testing temperatures, as illustrated in Fig. 3. The strain hardening behaviour of the alloy decreased and elongation–to–fracture increased with decreasing strain rate from \( 1 \times 10^{-1} \) to \( 1 \times 10^{-4} \) s\(^{-1}\) as shown in these curves. The effect of strain rate on elongation during hot deformation can be seen in Figs. (3–4). The samples tested at the slowest strain rate, i.e., \( 1 \times 10^{-4} \) s\(^{-1}\), resulted in the highest elongations for all numbers of turns in HPT processing and at all testing temperatures used in tensile testing as shown in Fig. 5 (a), e.g., for the sample processed in HPT for \( N = 1 \) turn, the maximum elongations were 249 %, 845 % and 1041 %, at testing temperatures of 423 K, 473 K and 573 K, respectively. The maximum elongations for the sample processed for \( N = 10 \) turns were 760 %, 1164 % and 1308 % using testing temperatures of 423 K, 473 K and 573 K, respectively. Significant elongations were achieved at all strain rates with increasing number of turns used in the HPT processing of the AZ91 alloy, where the highest values of the elongation using a strain rate of \( 1 \times 10^{-4} \) s\(^{-1}\) were 760 %, 1164 % and 1308 % at testing temperatures of 423 K, 473 K and 573 K, respectively. These highest elongations were obtained for the alloy processed in HPT for \( N = 10 \) turns as demonstrated in Figs. (3, 4, 5 (a)), where a homogeneous ultrafine microstructure is expected after HPT processing at room temperature as mentioned by several investigators [10,14,16,17]. Increasing the testing temperature resulted in increasing elongation at all numbers of turns in HPT processing, where the maximum elongations were 1041 %, 1190 %, 1234 % and 1308 %.
% found in the alloy processed through $N = 1$, 3, 5 and 10 turns, respectively at a testing temperature of 573 K, as illustrated in Fig. 5 (a) and illustrated in Fig. 3 for samples processed for $N = 10$ turns in HPT. Increasing the testing temperature resulted in the occurrence of significant elongations at even the fast strain rates, i.e., $1 \times 10^{-1}$ and $1 \times 10^{-2}$ s$^{-1}$ at temperatures of 473 K and 573 K, respectively as illustrated in Figs. (3, 5 (a)). This indicates the presence of excellent high–strain rate superplasticity (HSRSP) at these strain rates and temperatures for the current alloy compared to earlier results obtained after processing in EX–ECAP [10], HPT [11], hot extrusion [18] and hot rolling [19]. Tensile testing at a lower temperature, i.e., 423 K, resulted in significant elongations using strain rates of $1 \times 10^{-3}$ and $1 \times 10^{-4}$ s$^{-1}$. These elongations increased with increasing the number of turns in the HPT process as shown in Fig. 5 (a), where the maximum elongations were 660 % and 760 % using strain rates of $1 \times 10^{-3}$ s$^{-1}$ and $1 \times 10^{-4}$ s$^{-1}$, respectively at a temperature of 423 K. These data confirm the occurrence of low–temperature superplasticity (LTSP) for the AZ91 alloy.

The values of strain–rate sensitivity ($m$) were obtained from the slopes of the log–log format plots of the variation in flow stress versus strain rate for samples tested at different testing temperatures as shown in Fig. 5 (b). The values of sensitivity increased with decreasing the strain rate from $1 \times 10^{-1}$ to $1 \times 10^{-4}$ s$^{-1}$ at all testing temperatures, and are associated with an increase in the elongation–to–fracture for the slower strain rates as illustrated in the elongation–strain rate curves in Fig. 5 (a). Increasing the testing temperature resulted in an increase in the values of sensitivity over the strain rate range as illustrated in Fig. 5 (b). For a given number of turns for the sample processed in HPT and then pulled in tension, the flow stress decreased with decreasing strain rate and increasing testing temperature, which is reflected in the values of the sensitivity and in the extent of the elongation–to–fracture. The microstructures of tensile samples after tensile testing to fracture were observed along the gauge lengths by SEM as shown in Fig. 6. These samples were processed in HPT for $N = 1$ and 10 turns and then tested at different testing temperatures and strain rates. The grain sizes in Fig. 6 (a–f) for samples processed for $N = 1$ are larger than for samples processed for $N = 10$ turns as shown in Fig. 6 (g–l) over the range of strain rates and testing temperature. The samples processed for $N = 10$ turns in HPT exhibited significant microstructural stability with fine grains of an average size of $1.5 \mu m \pm 0.2 \mu m$ after testing at 423 K (for 2–1200 minutes) over the strain rate range. After testing at 473 K (for 3–1440 minutes), the subsequent microstructures also showed fine grains with only modest grain growth to an average grain size of $3 \mu m \pm 0.3 \mu m$.

Tensile testing at 573 K (for 5–1680 minutes) resulted in maintenance of a fairly fine microstructures with limited grain growth up to about $8 \mu m \pm 0.5 \mu m$ as shown in Fig. 6. The grains remained equiaxed after testing at 423 K, 473 K and 573 K at different strain rates as observed in Fig. 6; where some individual grains have developed into fibrous morphologies at temperatures of 473 K and 573 K at strain rates of $1 \times 10^{-3}$ s$^{-1}$ and $1 \times 10^{-4}$ s$^{-1}$ as shown in Fig. 6 (b,c,e,f), and at a temperature of 573 K using strain rates of $1 \times 10^{-3}$ s$^{-1}$ and $1 \times 10^{-4}$ s$^{-1}$ as shown in Fig. 6 (k,l). The fibrous or filament structures were observed at the slower strain rates and higher temperatures, and they connect the grains, which appear to have separated during the superplastic elongation regimes as observed in Fig. 7. The EDS analysis of these fibrous structures revealed their composition as shown in Fig. 7, and the analysis was conducted at three locations: grain, grain boundary and filament. It was found that the composition of these filaments consists mostly of β–phase ($\text{Mg}_{17}\text{Al}_{12}$) as indicated by the relative estimation of the weights of the constituents. It is noticeable from Figs. (6,8) that the existence of fine equiaxed grains and their relative thermal stability was seen in all samples tested at all temperatures and strain rates despite the differences in the number
of turns in HPT processing. This indicates the level of the considerable grain refinement obtained in HPT processing and its impact on the superplastic elongations obtained at elevated temperatures.

Discussion

In the present investigation, a successful HPT processing of the AZ91 magnesium alloy at room temperature with an ultrafine microstructure was achieved, which was not produced for the same alloy using ECAP processing at a temperature lower than 200 °C, where development of cracking with increasing number of passes was observed [4]. It is believed that the grain refinement in the current work was achieved within the nanometre range due to the imposition of a high plastic strain by HPT. This is indicated by a comparison between: Mg–9wt.%Al alloy [11], AZ31 alloy [15] and the alloy in this study, in terms of: the HPT processing conditions, the achieved refinement, and the obtained microhardness [16]. Increasingly ultrafine–grained AZ91 alloy (albeit with indistinct grain boundaries) developed with increasing number of HPT turns as shown in Fig. 1. The morphology and distribution of the β–phase also altered after HPT processing. The β–phase separated from the grain boundaries and fragmented into fine particles. Increasing the number of turns resulted in a refinement of β–phase down to nano–sized particles. These nano–sized particles aligned in the form of bands in the direction of the torsional straining with a relatively homogeneous distribution within α–Mg grains, and in part this is what makes direct observation of the grain sizes after multiple HPT turns via TEM techniques challenging. The stress–elongation curves in Fig. 3 for the fine–grained AZ91 magnesium alloy reveal significant superplastic elongation and relative thermal stability of the alloy under tensile loading at temperatures up to 573 K for up to 1680 minutes using a strain rate of 1×10⁻⁴ s⁻¹. The achieved elongations varied with microstructure, strain rate and testing temperature as shown in Figs. (3–6). For the alloy processed for \( N = 10 \) turns in HPT and then pulled in tension at a testing temperature of 573 K using a strain rate of 1×10⁻⁴ s⁻¹, the maximum elongation reached 1308 %; which (to the authors’ knowledge) is the highest value of elongation reported to date in this alloy. The elongation results in the current study are significantly higher than data that have been published earlier. For instance, the maximum elongation previously published was 810 % at 473 K using at a strain rate of 5×10⁻⁴ s⁻¹ for the Mg–9%Al alloy processed in HPT for \( N = 5 \) turns at 423 K [11], whereas in the present study, the maximum elongation was 1090 % at a testing temperature of 473 K using a strain rate of 1×10⁻⁴ s⁻¹, for an alloy processed at room temperature in HPT for \( N = 5 \) turns. In addition, the tensile elongations in this investigation are also higher than observed for alloy processed at high temperatures in ECAP as shown earlier [10], where the maximum elongation was 840 % at a testing temperature of 473 K using a strain rate of 3.3×10⁻⁴ s⁻¹ for samples processed with 2 passes in ECAP at a processing temperature of 473 K; whereas our alloy processed by HPT for \( N = 1 \) and 3 turns at room temperature showed maximum elongations of 845 % and 977 % at a testing temperature of 473 K using a strain rate of 1×10⁻⁴ s⁻¹. This difference in the elongation can be attributed to the processing temperature in HPT and ECAP. For magnesium alloys, it is well documented that a much finer microstructure can be produced by SPD processes at room temperature rather than at elevated temperatures [10,14,16,17]. Thus, the observed higher elongations are expected from a finer microstructure during the subsequent hot deformation as the presence of fine grains is one of the prerequisites for achieving superplasticity in polycrystalline materials [20]. The increase in elongation with finer microstructure can be seen in Fig. 5 (a), where the higher elongations towards the superplastic range were achieved with increasing the number of turns up to \( N = 10 \) turns. The microstructural inspections along the gauge
lengths of tensile samples after tension reveal that the grains remained equiaxed with fine sizes until fracture at all temperatures and strain rates. The presence of equiaxed grains was associated with superplastic elongations, which indicated that the main deformation mechanism was grain–boundary sliding (GBS). The retention of an equiaxed microstructure at elevated temperature under tension is necessary for superplastic elongations through grain–boundary sliding [21,22]. In addition, the retention of equiaxed grains after the tensile test as shown in Fig. 6 reveals the migration of grain boundaries during superplastic deformation at elevated temperatures. It was found that the stress concentration at grain boundary discontinuities can be reduced by the migration of grain boundaries during deformation, and thus grain–boundary sliding continues as the main deformation mechanism [23]. The measurements of strain–rate sensitivity \( m \) confirm that grain–boundary sliding is the dominant deformation mechanism, where the \( m \)–values were 0.3–0.5 as illustrated in Fig. 5 (b). It is well known that high values of strain–rate sensitivity indicate a higher resistance to failure by necking and thus high elongations are expected [21,22]. The current superplastic elongations were obtained as strain rate decreased and temperature increased, where \( m \)–values increased to 0.5. The tested samples showed excellent elongations using a strain rate of \( 1\times10^{-2} \text{ s}^{-1} \) at testing temperature of 473 K; and using strain rates of \( 1\times10^{-1} \text{ s}^{-1} \) and \( 1\times10^{-2} \text{ s}^{-1} \) at testing temperature of 573 K as illustrated in Figs. (3, 5 (a)). The current results reveal an excellent high–strain rate superplasticity (HSRSP) for the AZ91 alloy processed by HPT at room temperature compared to those found previously for Mg–9%Al alloy processed by EX–ECAP (360 %, \( 1\times10^{-2} \text{ s}^{-1} \), 498 K) [10], Mg–9%Al alloy processed by HPT (325 %, \( 1\times10^{-2} \text{ s}^{-1} \), 473 K) [11], hot extruded AZ91 alloy (300 %, \( 1\times10^{-2} \text{ s}^{-1} \), 548 K) [18], and hot rolled AZ91 alloy (275 %, \( 1\times10^{-2} \text{ s}^{-1} \), 698 K) [19]. It has been assumed that an improvement in microstructural stability at elevated temperature and additional grain refinement are possible ways to achieve high–strain rate superplasticity in magnesium alloys [24]. In the present work, the AZ91 alloy was processed in HPT at room temperature, which was not the case reported by those using other processing techniques such as equal channel angular pressing [10]. A more extreme grain refinement is expected after processing at room temperature than at elevated temperatures as reported in previous HPT studies [10,14,16,17]. Thus, the room temperature processed alloy with a finer microstructure exhibits better thermal stability and thus superior superplasticity under faster strain rates in hot deformation [25]. It can be seen in Fig. 6 (g–l), for the sample processed for \( N = 10 \) turns in HPT, that high–strain rate superplasticity to elongations of 590 %, 410 % and 860 % using strain rates of \( 1\times10^{-2} \text{ s}^{-1} \) at 473 K, \( 1\times10^{-1} \text{ s}^{-1} \) and \( 1\times10^{-2} \text{ s}^{-1} \) at 573 K, respectively, and the grain growth was insignificant at testing temperatures of 473 K (for 2–3 minutes) and 573 K (for 5–10 minutes). In addition, it is obvious from Fig. 5, that the maximum high–strain rate superplasticity achieved for Mg–9%Al processed in HPT for \( N = 5 \) turns at 423 K [11], was significantly lower than its counterpart for the alloy in the present investigation that was processed in HPT for the same number of turns but at 296 K. This can also be attributed to the effect of processing temperature on the grain refinement of magnesium alloys as discussed previously. The thermal stability of the AZ91 alloy was enhanced by the presence of fine particles of the \( \beta \)–phase (Mg\(_{17}\)Al\(_{12}\)). Prior to HPT processing, the \( \beta \)–phase exists normally along the grain boundaries as lamellar and agglomerate forms in the unprocessed alloy as shown in Fig. 1 (a). After HPT processing, this phase fragmented into fine particles with nanometre sizes due to the high value of the imposed strain by HPT and these nano particles were dispersed in the matrix (\( \alpha \)–Mg solid solution) as shown in Fig. 1 (b,c). It was found that the morphology of the second phase significantly affects the mechanical behaviour of the metallic materials at room temperature and elevated temperatures [26]. The existence of well–dispersed
particles of the β–phase inhibited significant grain growth during superplastic deformation at elevated temperature and then enhanced the extent of superplasticity. The β–phase has a melting point of about 733 K (460 °C) which is relatively lower than 768 K (495 °C) for the AZ91 alloy [1]; thus, the β–phase along the grain boundaries may glide relatively earlier than the grains during hot deformation. For samples processed in HPT for a low number of turns (N = 1), it was noticed that the distribution and volume fraction of the fine particles of the β–phase are relatively lower as shown in Fig. 1 (b) than in samples processed for a high number of turns (N = 10) as shown in Fig. 1 (c). Therefore, during tensile testing at elevated temperature, with a low fraction volume of the fine particles of β–phase, where the β–phase is located mainly near and/or on the grain boundaries, it acts as pinning phase and the sliding of grains is probably accommodated with limited sliding of the fine particles of β–phase. Increasing the distribution and volume fraction of the fine particles of the β–phase leads to a more significant sliding of the β–phase particles, which can be expected in samples tested at a temperature of 573 K, which represents 0.78 $T_m$ of the β–phase. It has been proposed that the β–phase acts as a lubricant for matrix sliding during tension [27]. Therefore, the highest elongations were obtained at all strain rates at a temperature of 573 K for samples processed in HPT for N = 10 as illustrated in Figs. (5 (a), 6). The effect of volume fraction of β–phase on the superplasticity has been reported earlier for Mg–15%Al–1%Zn alloy [27], Mg–33%Al alloy [28] where elongations have been improved considerably with increasing the amount of aluminium (or the volume fraction of the β–phase). As the strain rate decreased to $1 \times 10^{-4}$ s$^{-1}$ and testing temperature increased to 473 K and 573 K, the microstructure showed filaments and surface cavities as shown in Figs. (6 (c,f,i,l), 7). The formation of filaments has been reported for AZ91 alloy in the temperature range of (623–698) K [19], Mg–15%Al–1%Zn alloy in the temperature range of (548–598) K [27] and in AZ61 alloy in the temperature range of (573–673) K [29]. The filaments appear to have reconnect the disconnected grains and grain boundaries and relinked the cavities at the final stage of superplastic deformation. These fibres were formed and aligned in the direction of tension, and their lengths increased with decreasing strain rate and increasing temperature. Thus, the superplastic elongations were enhanced and maintained by the continuous fibrous structures at the lowest strain rate and elevated temperatures [19,27,29]. The micro–superplasticity of filaments in superplastic materials has been proposed to explain the superplastic elongations [27,30] as shown in Figs. (6 (c,f,i,l), 7). EDS analysis was conducted on matrix, grain boundary and filament as shown in Fig. 7, for a sample processed in HPT for N = 10 turns and then tested at a temperature of 573 K using a strain rate of $1 \times 10^{-4}$ s$^{-1}$. It can be seen that the alloy has oxidized since the testing was conducted in the air, as the chemical analysis revealed the presence of oxygen on the grain and grain boundary. The filaments were oxidized also but showed the presence of a higher aluminium level, 26.13 %, than in the structures of the grains and grain boundaries (9.97 % and 7.87 %, respectively). Therefore, it can be concluded that the filaments were composed mainly of β–phase as shown in previous work [27]. The variation in the average grain size as observed using SEM is illustrated in Fig. 8 for tensile samples with increasing testing temperature using different strain rates. It can be seen that the processed alloy with fine particles of β–phase retained its grain size below 10 µm over the range of subsequent testing temperatures, strain rates and times. The alloy exhibited only modest grain growth at lower temperature and/or high strain rates (lower times), and a limited grain growth at higher temperature and slow strain rates (higher times at temperature). Moreover, it was found that presence of a high volume fraction of these fine particles retarded cavity formation at elevated temperatures and slow strain rates due to softening of these particles at temperatures over 573 K [31]. Low–temperature superplasticity
(LTSP) was also noticed in the AZ91 alloy during tensile testing at a low temperature of 423 K using strain rates of $1 \times 10^{-3}$ s$^{-1}$ and $1 \times 10^{-4}$ s$^{-1}$ as illustrated in Fig. 5 (a). The lower testing temperature is equivalent to 0.55 $T_m$, where $T_m$ for the AZ91 alloy is 768 K (495 °C) as illustrated in the phase diagram of the alloy [32]. The current performance in the low–temperature superplasticity regime is better than previous data obtained for the AZ91 magnesium alloy [2,11]. It has been found that finer grain sizes are preferable for achieving low–temperature superplasticity as well as for achieving high–strain rate superplasticity [2]. It can be seen that the grain sizes were retained to within 1 µm and 4 µm for samples tested at 423 K and 473 K, respectively, using strain rates of $1 \times 10^{-3}$ s$^{-1}$ and $1 \times 10^{-4}$ s$^{-1}$ as observed in Fig. 6 and illustrated in Fig. 8. The current results for low–temperature superplasticity reveal the potential for superplastic forming of magnesium alloys at lower possible temperatures to overcome their poor workability at room temperature and excessive oxidation at elevated temperatures [33]. A small difference was observed in the maximum values of the low–temperature elongations, for samples processed by EX–ECAP (800 %, $1 \times 10^{-4}$ s$^{-1}$, 423 K) [10], and its counterpart obtained in the current study (760 %, $1 \times 10^{-4}$ s$^{-1}$, 423 K). This can be attributed to the difference in dimensions of tensile samples for ECAP and HPT. The ECAP tensile sample was cut from a cylindrical billet with a gauge length of 5 mm and gauge cross–section area of (3×2) mm$^2$ [10], whereas in this study, the HPT tensile sample was cut from a circular disk with a gauge length of 1.0 mm and gauge cross–section area of (0.9×0.6) mm$^2$. Therefore, the small difference in the calculated elongations can be attributed to the difference between the relatively large–scale and micro–scale tensile samples produced in ECAP and HPT, respectively [34,35]. Moreover, the direction of cutting for tensile samples from ECAP billets and HPT disks has a further impact. The tensile samples after EX–ECAP were cut parallel to the longitudinal axes after the extrusion step and after the ECAP [10], whereas the disk–shaped samples were cut firstly from an extruded rod perpendicular to the extrusion direction; then after HPT processing, the tensile samples were cut parallel to the shear–plane direction [14]. Therefore, occurrence of a strong texture is anticipated due to the extrusion and subsequent ECAP through the route $B_{c}$, and alignment of the basal planes parallel to the extrusion direction [36], which leads to easy slip in tension at a testing temperature of 423 K and the occurrence of low–temperature superplasticity [37]. In contrast, the monotonic HPT mode was used in the processing of the AZ91 alloy in this investigation, which leads to a more random texture with equiaxed grains at a high number of turns [38]. Therefore, at temperatures of 423 K, a relatively lower elongation is expected in the alloy processed by HPT compared to its counterpart processed by EX–ECAP. The average strain sensitivity of 0.3 and the equiaxed grains were found for all samples tested at a testing temperature of 423 K using a strain rate of $1 \times 10^{-4}$ s$^{-1}$, which suggests that grain boundary sliding is the deformation mechanism at low temperature [2]. Lower elongations were found at a low temperature of 423 K using high strain rates of $1 \times 10^{-1}$ s$^{-1}$ and $1 \times 10^{-2}$ s$^{-1}$, and these elongations were associated with low values of strain–rate sensitivity with an average of 0.25. Thus, glide–dislocation creep is assumed as the deformation mechanism accommodated with grain–boundary sliding as the grains retained their equiaxed shapes and did not elongate as in the case of dislocation creep only [19,39]. The activation energy was calculated at a fixed strain rate using the following equation [40,41]: $Q = nR(\partial (\ln \sigma) / \partial (1/T))$, where $Q$ is the activation energy, $n$ is stress exponent ($n = 1/m$), R is the gas constant, and $(\partial (\ln \sigma) / \partial (1/T))$ is the slope of plot in Fig. 9. For the AZ91 alloy, the activation energy was obtained for a sample processed in HPT for $N = 10$ turns then tensile tested using a strain rate of $1 \times 10^{-4}$ s$^{-1}$ over the temperature range (423–573) K as shown in
Fig. 9. It was found that the activation energy equals 80.34 KJ/mol that is close to the activation energy of grain boundary diffusion of pure magnesium (92 KJ/mol). Therefore, grain–boundary sliding is the dominant deformation mechanism, which is consistent with the observed microstructures as shown in Fig. 6. The grain–boundary sliding mechanism was accommodated with diffusional flow at temperatures of 473 K and 573 K using a strain rate of $1\times10^{-4}$ s$^{-1}$ as shown in Fig. 6 (c,f,i,l). It can be seen that the shapes of grains under these conditions were changed from equiaxed to elongated and oriented towards the tension axis and thus the highest level of superplasticity produced [42]. The relative difference in the obtained activation energy and its counterpart for pure magnesium is attributed to the presence of $\beta$–phase in the AZ91 alloy [27], where this phase has an activation energy for grain boundary diffusion of 65 KJ/mol, which reduces the overall activation energy for the present alloy [43,44]. The steady–state strain rate for the superplastic flow at high temperatures is expressed by [45]:

$$\varepsilon = \left( \frac{ADGb}{kT} \right) \left( \frac{b}{d} \right)^p \left( \frac{\sigma}{G} \right)^n,$$

where $D$ is the appropriate diffusion coefficient $D = D_o \exp(-Q/RT)$, $D_o$ is the pre–exponential complex constant, $A$ is a dimensionless constant, $G$ is the dynamic shear modulus, $b$ is the Burgers vector, $d$ is the grain size, $\sigma$ is the applied stress, $p$ and $n$ are the exponents of the inverse grain size and normalized stress, respectively. Using the following values of $p=2$, $n=2$, $A=10$, $D = D_o$ [22], $D_o = 7.8\times10^{-3}$ m$^2$s$^{-1}$, $G = 1.92\times10^4 - 8.6\times T$ (MPa), and $b = 3.2\times10^{-10}$ m [45] in the former equation for the grain–boundary sliding mechanism results in Fig. 10, which represents the temperature and grain size compensated strain rate versus the normalized stress for the alloy processed for $N = 10$ turns and tested in tension at different temperatures and strain rates. The solid line represents the predicted strain rate for superplasticity with a slope of $n=2$ ($m=0.5$). Good agreement was obtained between the observed experimental data for the current alloy and similar work in a series of magnesium alloys [46,47] and the constitutive equation of superplastic flow based on the assumption that grain–boundary sliding is the dominant deformation mechanism over these testing temperatures and strain rates. It is important to note that the values of grain sizes were collected from the SEM micrographs of the gauge lengths of the tensile test as indicated in Fig. 6 and these values were used in the constitutive equation.

**Conclusions**

1. The HPT processing at room temperature for the AZ91 magnesium alloy resulted in excellent superplastic elongations that were higher than their counterparts obtained in earlier work through HPT, ECAP and EX–ECAP.

2. High–strain rate superplasticity (HSRSP) was obtained with excellent elongations of 590 % at a testing temperature of 473 K using a strain rate of $1\times10^{-2}$ s$^{-1}$, 410 % and 860 % at a testing temperature of 573 K using strain rates of $1\times10^{-1}$ s$^{-1}$ and $1\times10^{-2}$ s$^{-1}$, respectively.
3. Significant low–temperature superplasticity (LTSP) was achieved with maximum elongations of 660 % and 760 % at a testing temperature of 423 K and using strain rates of $1 \times 10^{-3} \text{ s}^{-1}$ and $1 \times 10^{-4} \text{ s}^{-1}$, respectively.

4. The samples processed by HPT at room temperature revealed fine equiaxed grains with significant thermal stability at all testing temperatures and strain rates.

5. Equiaxed microstructures and high values of strain–rate sensitivity indicate that grain–boundary sliding was the main deformation mechanism during the high–strain rate superplasticity regime. Glide–dislocation creep accommodated with grain–boundary sliding is suggested as the deformation mechanisms operating during the low–temperature superplasticity regime. At high temperature and slow strain rate the grain–boundary sliding accommodated with a diffusion creep mechanism.

6. Considerable thermal stability of the processed alloy was attributed to the ultrafine grains produced by HPT at room temperature and to the high volume fraction of fine, nano–sized β–phase particles.

7. The fibrous structures were mainly composed of β–phase and they enhanced the superplasticity at high temperatures and low strain rates through resisting the cavitation and relinking the disconnected grains.

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References


Fig. 1: SEM observations of microstructure for (a) the as–received AZ91 alloy, (b) the processed alloy for $N = 1$ turn, (c) the processed alloy for $N = 10$ turns and (d) TEM observation of the processed alloy for $N = 1/2$ turn. The corresponding numbers (1,2,3) in the micrograph (a) represent the lamellar, agglomerate forms of the $\beta$–phase ($\text{Mg}_{17}\text{Al}_{12}$) and $\text{Al}_8\text{Mn}_5$, respectively.

Fig. 2: The chemical analysis with weight fractions of the as–received alloy showing: (a) $\alpha$–Mg matrix, (b) $\beta$–phase ($\text{Mg}_{17}\text{Al}_{12}$), (c) $\text{Al}_8\text{Mn}_5$ compound.
Fig. 3: The engineering stress–strain behaviour at a testing temperature of 573 K using different strain rates for AZ91 samples processed in HPT for $N = 10$ turns.

Fig. 4: Appearance of samples processed by HPT for $N = 10$ turns after tension to fracture at a testing temperature of 573 K at different strain rates. The upper sample represents the untested case.
Fig. 5: (a) Elongation–to–fracture versus strain rate at different testing temperatures for alloy processed by HPT through $N = 1, 3, 5$ and 10 turns. (b) log–log plot of the variation of flow stress as a function of strain rate shows the strain–rate sensitivity values ($m$) at different testing temperatures for the samples processed in HPT for $N = 1, 3, 5$ and 10 turns.

![Comparison of elongation and strain rate at different testing temperatures](image-url)
Fig. 6: Magnified views of the microstructures of the AZ91 alloy as observed by SEM on the gauge lengths after the tensile test. The samples above were processed by HPT for \( N = 1 \) turn (a – f) and \( N = 10 \) turns (g – l) before tensile testing. The tested samples showed fibrous morphologies as shown in (c,f,i,l).

Fig. 7: EDS analyses with the weight fractions of the elements at: (a) grain, (b) grain boundary and (c) filament, for the sample processed in HPT for \( N = 10 \) turns and tested in tension at a testing temperature of 573 K using a strain rate of \( 1 \times 10^{-4} \) s\(^{-1}\).
Fig. 8: The variation in the grain size after tensile testing at different testing temperatures (423 K, 473 K, 573 K) and different strain rates and testing times (dotted lines) for the AZ91 alloy processed in HPT for \( N = 1 \) (dashed lines) and \( N = 10 \) turns (solid lines).
Fig. 9: The variation in the flow stress with the reciprocal of the temperatures from 423 K to 573 K to determine the value of the activation energy (Q) for the tensile samples tested using strain rates of $1\times10^{-3}$ s$^{-1}$ and $1\times10^{-4}$ s$^{-1}$. The straight line represents the linear least squares fit for the obtained data and its slope then refers to the value of the activation energy.
Fig. 10: The temperature and grain size compensated strain rate versus normalised stress for the AZ91 alloy processed in HPT compared with a series of magnesium alloys. The slope of the straight line has a value of the stress exponent of 2, and represents the predicted superplastic strain rate.