Effect of strain rate on the mechanical properties of magnesium alloy AMX602

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Abstract

In the present work, the effect of strain rate on the mechanical properties, particularly the plastic deformation behavior of a magnesium alloy, AMX602 (Mg-6%Al-0.5%Mn-2%Ca; all wt.%), fabricated by powder metallurgy, has been investigated under both quasi-static (strain rate $1 \times 10^{-3} \text{ s}^{-1}$) and dynamic (strain rate $4 \times 10^3 \text{ s}^{-1}$) compressive loading. The alloyed powder was extruded at three different temperatures. The microstructure of the alloy was examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It was found that AMX602 exhibits an impressive mechanical behavior but with a slight anisotropy along different directions in both strength and compressive ductility (or malleability). The strength was found to be nearly independent of the extrusion temperature, particularly, under dynamic loading. Nanoindentation strain rate jump test reveals a strain rate sensitivity of ~ 0.018 to ~ 0.015 , depending on the extrusion temperature. Sub-micrometer-scale particles of the intermetallic compound Al_2Ca were found with sizes ranging from $\sim 100 \text{ nm}$ to $\sim 1.0 \mu\text{m}$. These intermetallic particles are believed to have precipitated out during the extrusion process. They contribute to the formation of the ultrafine equiaxed grains which, in turn, help to improve the strength of the alloy by acting as barriers to dislocation motion. Adiabatic shear bands (ASBs) were observed in the dynamically loaded samples, the propagation of which eventually leads to final fracture of the specimens.

Key words: Magnesium alloy; strengthening mechanisms; adiabatic shear band; strain rate effect.

1. Introduction

As one of the lightest metals that holds great potential for structural applications, Mg-based alloys and composites have attracted great attention in the past few decades (the mass density of Mg is 1.74 gm/cm^3 , somewhat higher than that of water). However, owing to its hexagonal close packed (hcp) structure with a c/a ratio of ~ 1.624 , Mg and its alloys show undesirably low tensile ductility due to the basal slip and sometimes extension twinning dominated plastic deformation mechanism. This fact has greatly hindered the wide application of Mg-alloys as structural materials. In other words, alternative slip systems, other than basal slip, have to be activated during the deformation process in Mg-alloys so as to extend its utility. A theoretical analysis of pure Mg that relates the possibility of concurrently activating various slip systems to texture has been reported [1]. Experimental investigations have revealed that texture plays a significant role in improving or impairing the ductility of Mg-alloys [2-5]. Since the critical resolved shear stresses (CRSS) of the potential slip systems of Mg exhibit a wide temperature dependence [6], non-basal type slips can take place at elevated temperatures, resulting in improved ductility [7-10]. Strength decrease at high temperatures can be avoided by grain refinement that is thermally stable via fine precipitates that work as grain boundary pinning centers [11-18].

The combined effect of grain boundary strengthening, second phase strengthening and activation of non-basal slip mechanisms may lead the way to better Mg-alloys which will have both decent ductility and impressive strength [19-22]. In the past few decades, a number of grain refinement methods have been exploited for Mg-alloys, including severe plastic deformation (SPD) and/or powder metallurgy [12-14, 23-26]. *Ex-situ* addition of micro-/nano-particles to the Mg or Mg-alloy matrix has resulted in grain refinement and strength improvement, but the ductility of such metal matrix composites is generally lower than the matrix due to the significant strain incompatibility between the matrix and particles [27-32]. However, several factors may account for this: the adhesive force and reaction between the particle and the matrix, the mechanical property of the particles and the material processing history, etc. [33-35]. In contrast, *in-situ* composites may have largely overcome the drawbacks of *ex-situ* composites. Meanwhile, several studies report on texture modification by rare earth or special element doping [36-41].

In addition to dislocation mediated slip, deformation twinning has been found to play a significant role in the plasticity of Mg-alloys, depending on grain size, strain rate and

temperature [42-44]. However, the details of twinning processes have been a topic of debate. In contrast to the statements of Barnett et al. and Bhattacharya and Niewczas [17, 45] that twinning occurs after a finite deformation or at the late stage of deformation prior to fracture of Mg-alloys, Koike and Somekawa and Mukai [46, 47] argued that it forms at the early stage of deformation and strongly relates to yield strength. An inverse grain size effect on mechanical twinning was reported by Wu and Zhu [48], who attributed this phenomenon to the increased stress requirement of twinning partials with grain size reduction that it exceeds the stress of the leading partials at a critical grain size. As such, suppression of deformation twinning via grain size reduction in Refs. [17, 46, 49] should relate to the activation volume of plastic deformation. Yet, the variation of activation volumes of plasticity of hcp metals as a function of grain size is still largely an unexplored area compared to fcc and bcc alloys [50].

Recently, adiabatic shear banding (ASB) has been identified as a common mode of plastic instability which leads to final failure of several Mg-alloys, particularly under dynamic loading [51-53]. A general theory based on the competition between work-hardening and thermal-softening of the material concludes that ASB occurs when work-hardening is out-paced by thermal softening [54]. Therefore, normally the propensity to ASB should be minimized for metals with substantial strain hardening capacity. For example, the localized deformation process was captured step-by-step in HY-100 steel by Marchand and Duffy [55], whose results indicated that most of the macro-plasticity at the second stage (non-uniform plasticity) was contributed primarily by localized deformation, which corresponded to a continuous reduction in the strain hardening rate. As such, the occurrence of ASB in Mg-alloys exhibiting extremely strong strain hardening demands further examination. In fact, Rittel et al. [51] argued that the dynamic stored energy of cold work was the governing factor for ASB formation instead of critical stress and/or strain. But this hypothesis needs further elaboration and articulation.

In the present work, the effect of strain rate on an Mg-alloy AMX602 with moderate ductility was investigated under uniaxial compressive quasi-static and dynamic loading conditions, and with the use of nano-indentation strain rate jump experiments. The mechanical properties, particularly, the plastic deformation mechanisms and fracture modes were examined as functions of the strain rate and the microstructure of the alloy. The alloy was extruded at three different temperatures. The microstructure of the as-extruded and post-mortem specimens has also been examined by scanning electron microscopy (SEM) and transmission electron

microscopy (TEM). A preliminary explanation is attempted for the occurrence of ASB in this Mg-alloy based on the 2nd derivative of the stress with respect to strain and stored energy release by the formation of ASB that gives rise to local adiabatic temperature rise exceeding the melting point of Mg.

2. Experimental Details

In this work, AMX602 (Mg-6%Al-0.5%Mn-2%Ca, all in weight percentage) supplied by the Joining and Welding Research Institute, Osaka University, Japan, has been chosen for investigation. Briefly, a spinning water atomization (SWA) technique was used to produce the AMX602 powders with the desired chemical compositions. The powders were then collected, compacted and finally consolidated by extrusion at various temperatures. The details of the alloy production have been provided by Kondoh and co-workers elsewhere [26]. Three different extrusion temperatures, 250°C, 300°C and 350°C were used. The samples were cut from the extruded rods both in the flow (extrusion) and transverse directions, and were tested at room temperature. Quasi-static compression samples had rectangular geometry with square loading faces and a height/width aspect ratio of 2:1, following the ASTM standard for such experiment. High-strain rate uniaxial compression samples have cuboid shape that is also commonly used and well described in the community [56, 57]. More specifically, the dimensions of the specimens for uniaxial quasi-static compressive tests is 2.5mm×2.5mm×5.0mm (5.0 mm is the gauge length), while the geometry for uniaxial dynamic compressive tests is 2.5mm×2.5mm×2.0mm (2.0 mm is the gauge length).

Dynamic (high-strain rate) uniaxial compression was performed using Kolsky bar (or Split-Hopkinson Pressure Bar - SHPB) technique. The working principles, precautions and caveats of this experimental technique can be found elsewhere. Briefly speaking, the specimen is sandwiched between two co-axial elastic bars made of the same material (usually high strength steel). They are the input and output bars. A cylindrical bar projectile launched from a gas-gun impacts one end of the input bar and as such a stress wave is generated which travels through the input bar. When it reaches the interface between the input bar and the specimen, part of the stress wave is reflected back into the input bar, and part of it is transmitted into the specimen. One part of the incident stress wave in the specimen was reflected back and forth until an equilibrium condition is reached. Another part is transmitted into the output bar and is eventually captured by

a momentum trap. Strain gages are attached to the input and output bars which pick up the stress wave signals that are recorded by a multi-channel high-speed oscilloscope. Processing of the signals leads to high strain rate stress-strain data of the specimen. The Kolsky bar has been regarded a milestone for experimental mechanics and has been widely used to characterize the dynamic mechanical behavior of structural materials. In this work, lubricating grease was applied at the specimen/bar interface to prevent or at least mitigate mechanical constraint.

In addition, strain rate jump tests of AMX602 with different extrusion temperatures were performed with a nano-indentation system (Agilent G-200) operated in a continuous stiffness mode (CSM) so as to investigate the strain rate sensitivity and the elastic (Young's) modulus of AMX602. The nano-indentation samples were polished by using diamond paper down to 0.1- μm , and ten indentations were made for each condition to ensure the repeatability of the results.

SEM and TEM were used to evaluate the pre-testing and post-mortem specimens. The purpose was to examine the microstructure of the as-processed and post-mortem specimens, with which the microstructure evolution of the alloy under compressive loading and the underlying deformation and failure mechanisms of the alloy could be identified. The SEM observation was performed on a JEOL JSM 6480 SEM with an energy dispersive spectroscopy (EDS) attachment operated at 10 kV, while the TEM analysis was conducted on a JEOL JEM 2100 operated at 200 kV. The TEM samples were prepared by mechanical grinding a disk to about 100 μm , followed by dimpling and final thinning to electron transparency with a precision ion polishing systems (PIPS, Gatan).

3. Experimental Results

Initially, the microstructure of the as-extruded AMX602 was examined. Additionally, the mechanical properties at different loading rates of the alloy extruded under different conditions were determined, along with the alloy's strain-rate sensitivity using nano-indentation measurements. The characterization was followed by fractography of representative samples in order to identify the fracture mode based on detailed examinations of the post-mortem specimens.

3.1. Microstructure of as-extruded AMX602

Figures 1(a) and 1(b) show a bright field TEM image of AMX602 extruded at 250°C and the corresponding selected area diffraction pattern (SADP). Indexing of the SADP reveals that the

discontinuous rings are from the Mg matrix, with the very first three inner rings corresponding to $\{10\bar{1}0\}$, $\{0002\}$ and $\{10\bar{1}1\}$ planes, respectively. A quantitative chemical analysis by EDS of the particle marked by the dark square indicates that it contains only Al and Ca with an atomic ratio of ~ 2.0 (as shown in Figure 1(c)), besides the matrix element Mg. As such, it is believed that the particle is an Al_2Ca intermetallic precipitate. Figure 2 presents SEM images of AMX602, extruded at different temperatures. The white phases in these SEM images have also been identified as the intermetallic precipitate Al_2Ca via EDS attached to the SEM (not shown here). The white arrows in Figure 1(a) indicate more such particles with varied sizes. This observation is also consistent with the earlier XRD results of Kondoh et al. [26] of the same alloy. Thus, it can be concluded that these white phases are indeed the intermetallic compound, Al_2Ca . These SEM images also illustrate that the size of the intermetallic compound has a wide range, from less than 100 nm to $\sim 1\text{ }\mu\text{m}$; note, the smaller particles are beyond the resolving power of the SEM since TEM reveals smaller particles as displayed in Figure 1 (only about 100 nm or less). Both TEM and SEM suggest that the intermetallic compound particles are uniformly distributed within the Mg-matrix. Such small particles can effectively impede grain growth of the Mg matrix via the Zener pinning effect. Clustering of this compound was also found and the size of the agglomerates were measured to be up to $5\text{-}\mu\text{m}$ as indicated by the arrows in Figure 2. Yet, the number of large agglomerates is relatively low. Larger agglomerates were more likely to form at higher temperatures during hot extrusion. The particle size of the intermetallic was found to increase with extrusion temperature, as evidenced by the SEM images in Figure 2. This observation can at least serve as indirect evidence that these intermetallic particles formed via a nucleation and growth mechanism during hot extrusion. The SEM images of Figure 2 also show that the grain size of the Mg-matrix increases with extrusion temperature, but the increase is not extraordinarily remarkable. This can be interpreted in two ways. Firstly, at higher extrusion temperatures, the size of the intermetallic compound particles becomes larger and their inter-particle spacing decreases. This weakens the Zener pinning effect. Secondly, a higher extrusion temperature lends itself to more rapid diffusion, expediting grain growth. Nevertheless, the matrix grains are no larger than $1\text{ }\mu\text{m}$ even at an extrusion temperature of 350°C .

The TEM and SEM evaluations revealed a submicrometer grain size of the Mg-matrix in AMX602 extruded at different temperatures; however, the grain size increases with extrusion temperature. Within the Mg-matrix, there were intermetallic precipitates of Al_2Ca . Again, the

size of these particles increased with extrusion temperature and was found to have a wide size distribution.

3.2. Mechanical properties of AMX602 at different strain rates

Figure 3 shows representative stress-strain curves of AMX602 for the individual extrusion conditions at various loading rates. It is worth mentioning that the curves from three independent experiments of the various individual extrusion conditions and under different individual loading conditions are highly reproducible. The curves in both the extrusion or flow direction (ED/FD) and transverse direction (TD) exhibit three distinct stages of plastic deformation. This is particularly the case when the specimens are loaded in the ED/FD both under quasi-static and dynamic loading, Figures 3(a1) and 3(a2), respectively. Such a stress-strain curve shape is also called sigmoidal. The first stage of this type of plastic deformation is the nearly flat plastic flow region after yielding; the second stage is characterized by strong strain hardening; and, the last stage of plastic deformation still shows strain hardening, but with a reduced strain hardening-rate, which is eventually followed by failure. The sigmoidal shape is more substantial in the ED/FD stress-strain curves, see Figures 3(a1) and 3(a2), while those in the TD only exhibit marginal sigmoidality, Figures 3(b1) and 3(b2). For example, there is no obvious flat stage in the dynamic curves for TD loading and, under quasi-static loading, Figure 3(b2), the flat part is much less remarkable compared to the ED/FD stress-strain curves. These results suggest that different plastic deformation mechanisms may be responsible for ED/FD loading versus TD loading. Furthermore, different plastic deformation mechanisms are contributing to the plasticity at different loading rates. These observations will be discussed in detail in the next part of this work.

To facilitate comparison and analysis, Table 1 lists the offset yield strengths and ultimate compressive strengths at different loading rates for AMX602, extruded at different temperatures. The offset yield strength represents the true stress at a strain of 0.2% for quasi-static loading and 5% for dynamic loading, respectively. We choose a 5% offset for dynamic loading because it is well known that the stress data before a 5% offset strain involves significant uncertainty due to the nature of stress-wave loading during Kolsky bar experiments. The strength values given in Table 1 are the average from three independent tests for each individual extrusion and loading conditions. The statistical errors are also provided in the table following the average value. The experimental results suggest that both the offset and ultimate strengths decrease slightly with

increased extrusion temperature. This observation is not surprising taking into consideration of the decreasing Hall-Petch effect since the matrix grains are larger for the alloy extruded at higher temperatures.

The strength difference between ED/FD and TD was measured quantitatively. The result indicates that the strength in the flow direction is $16.9 \pm 3.5\%$ greater than that in the transverse direction under all extrusion conditions independent of loading rates, except for the offset strength at dynamic loading rate. The offset strengths in flow direction are $\sim 4\%$ smaller than those in transverse direction for all extrusion conditions, since the 5%-offset strength was adopted at high loading rate. In addition, the failure strains of AMX602 for the same processing condition are of $\sim 15\%$ and $\sim 20\%$ for quasi-static and dynamic loading, respectively.

Finally, the elastic modulus of AMX602 was measured through instrumented nano-indentation to be close to ~ 48 GPa for all of the samples. This value is very close to the elastic modulus of pure magnesium (~ 45 GPa). The nano-hardness values along with the indentation strain rates of AMX602 that was extruded at different temperatures are plotted in Figure 4. It should be noticed that both the hardness and the indentation strain rates are plotted as logarithmic values, and thus the slope of each curve gives the strain rate sensitivity (SRS) value, m , for a given specimen. The SRS values evaluated in this manner are 0.015, 0.016 and 0.018 for extrusion temperatures of 350, 300 and 250°C, respectively. In other words, the SRS value decreases with extrusion temperature. It is also worth noting that the nano-hardness of AMX602 is consistent with the result of the compression tests in which the strength of AMX602 decreases with the increasing extrusion temperature.

In summary, both quasi-static and dynamic compressive stress-strain curves of AMX602 extruded at different temperatures show distinct sigmoidal stress-strain curve shapes loaded either in the ED/FD or in the TD. But ED/FD direction exhibits substantially sharper sigmoidality in the stress-strain curves. The ED/FD data reveals that the alloy is slightly stronger than in the TD. Both the yield and maximum compressive strengths decrease with extrusion temperature.

3.3. Post-loading SEM examinations of AMX602

The fragments of the failed specimens were collected after each test for further investigation of the failure mode of the alloy. All of the specimens that were tested at a quasi-static loading

rate fractured into two pieces, while most of their dynamic counterparts broke into three main pieces, and some with several smaller pieces. Typical partially recovered specimens from the quasi-static and dynamic loading are shown in Figures 5(a) and 5(b), respectively. It is worth pointing out that all of the quasi-statically loaded samples failed in the same pattern as that shown in Figure 5(a), while all of the dynamically fractured samples had two conjugate primary cracks similar to that shown in Figure 5(b). One exception in the case of dynamic loading is presented in Figure 6(a) which shows that one sample survived the dynamic loading, and remains intact, with only one crack running roughly along the maximum shear stress direction. It will be illustrated later that this specimen provides rich and useful information regarding the deformation and failure mode of AMX602.

Careful examination of the crack-like feature in Figure 6(a) suggests that it appears as an adiabatic shear band (ASB) which did not cause complete separation and failure along its path. The average width of the band is around 6-10 μm , as shown in Figure 6(b). A more in-depth discussion will be provided in the subsequent section that presents evidence for the possible formation of an ASB. The higher magnification SEM images shown in Figure 6(b) ~ 6(d) show close-up details of the areas labeled as b, c and d in Figure 6(a). These images indicate that the ASB becomes narrower as it runs into the interior of the specimen. Figure 6(c) shows that the ASB discontinues at the middle of its apparent propagation, but then continues about 2 μm away from its original endpoint on the plane and direction of propagation. Note, however, the apparent discontinuity may be fictitious as the image is only a two-dimensional cross-section of the shear band. Additionally, microcracks can be observed within the ASB or, more specifically, at the ASB/matrix interface. An increasing change in contrast, associated with the band structure, is also noticeable, presumably from the charging of an electrically insulating phase that is located at the beginning part of the ASB, while a darker contrast appears at the later stage of the ASB. Furthermore, more cracks are observed in the early stage of ASB, Figure 6(b) *versus* Figures 6(c) and 6(d). The cracks are possibly from thermal effects, indicative of a substantial temperature rise within the ASB during dynamic loading.

Nevertheless, even smaller cracks can be observed from the fracture surface of a specimen that has been fragmented, as indicated by the arrows in Figure 7(a). The appearance of these microcracks will be further discussed in the Discussion. It is also worth mentioning at this point that the fracture surfaces of AMX602 obtained at the same loading rates have very similar

features. The SEM images of Figure 7 show a couple of representative fracture features from 7(a) dynamic and 7(b) quasi-static samples, respectively. The significant difference of these fracture surfaces in Figure 7 indicates distinctly different failure modes of AMX602 at various loading rates. The fractographic view of the dynamically fractured specimen looks like that a melting and quenching process took place in the ASB region, while the quasi-static sample shows a typical, ductile fracture surface characterized by dimples. The dimples were found among all of the quasi-static samples that were examined, while a more smooth fracture surface was found on the dynamic samples. No microcracks associated with the intermetallic compound were found among the post-loading specimens during the SEM observations; this is reasonable as the intermetallic precipitates were of sub-micrometer size. This is also in agreement with the observations of Ayman et al. [18] who attributed the presence of these sub-micrometer intermetallic precipitates to be responsible for the remarkable properties and performance of AMX602 in terms of its strength and ductility.

Under dynamic uniaxial compression, AMX602, regardless of extrusion temperature, exhibited ASBs. Examination of an incomplete ASB indicated its propagation and significant adiabatic temperature rise within a narrow region under dynamic loading. Microcracks were identified within the bands, and on the resultant fracture surface. Under quasi-static loading, the specimens failed along a primary crack roughly along the maximum shear stress plane. Ductile dimples were observed on the fracture surface in the quasi-statically loaded specimens.

4. Discussion

The mechanical properties, especially the plastic behavior of structural materials depends strongly both on the loading conditions, such as temperature, stress state, the imposed strain rate, and on the chemistry and microstructure of the material [58-61]. When the loading conditions are given, the strength and ductility of the material are only dependent on the internal variables of the material such as grain size, dislocation density, second phase particles (their size, volume fraction, shape and distribution), etc. Within this context, a plausible strengthening mechanisms for AMX602, its plastic anisotropy, plastic deformation mode and failure mode will be described.

Grain refinement or the so-called Hall-Petch effect has been regarded as one of the most effective strengthening mechanisms for metals and alloys. However, due to the limited ductility and the low melting temperature of Mg, severe plastic deformation (SPD) techniques when used

in isolation have their limitation in refining the grains of Mg alloys. As such, the smallest grain size achievable via SPD is a few micrometers [3, 62, 63]. This is particularly true for Mg-alloys where popular SPD techniques such as equal channel angular extrusion (ECAE) have to be performed at $\sim 250^{\circ}\text{C}$ (523K , $>0.5T_m$) or above without the use of a confinement stress (more commonly known as back pressure). The processing temperature can be lowered with the application of back pressure, but at the cost of increased complexity of the process. Therefore, other approaches need to be discovered and applied to suppress recrystallization and grain growth during thermo-mechanical processing.

Recent efforts have demonstrated that novel alloying methods that lead to the precipitation of nanoscale particles can effectively pin the Mg grain boundaries, which, in turn, retard dynamic recrystallization and grain growth through Zener pinning and, at the same time, serve to stabilize the fine microstructure at elevated temperatures. This effect is well illustrated by the novel AMX602 alloy described herein that serves as an excellent example. The advantage of this alloy's design is two-fold. First, since at ambient temperature the solubility of Ca in Mg is nearly zero, Ca will form an intermetallic compound phase, Al_2Ca , during the hot extrusion process. The nanometer-sized particles, shown in Figure 1(a), can effectively pin the Mg-matrix grain boundaries, so that grain growth is suppressed during hot extrusion. This explains the small grain size of the Mg-matrix phase ($d < 1.0\ \mu\text{m}$, Figure 2). As a consequence, in part, AMX602 derives its strength from the grain size refinement and associated Zener pinning effect of the intermetallic compound particles. Second, these nanometer-sized intermetallic particles themselves can serve as a medium to give rise to an Orowan strengthening mechanism. An additional effect of the presence of the intermetallic nano-particles is the thermal mismatch between the particles and the Mg-matrix that is usually held responsible for the generation of geometrically necessary dislocations (GND). Thus GNDs can further contribute to the overall strength of the alloy. It should also be pointed out that the final densification of the alloy powder was performed by hot extrusion for less than 5 minutes when the fine Al_2Ca intermetallic compound particles were formed. As discussed in Ref.[64] these fine particles can assist dynamic recrystallization (DRX) by promoting the nucleation process but, at the same time, they can hinder the motion of grain boundaries, both that are newly formed during DRX and those which pre-exist. Combined with the limited time for grain growth during the hot extrusion process, the ultrafine and uniform grain structure may remain in the alloy well after the hot

densification process.

The strain hardening rates, $\Theta = d\sigma / d\varepsilon$, of AMX602 along the two perpendicular testing directions (ED/FD and TD) and at different loading rates (quasi-static and dynamic) were also evaluated. Figure 8 shows typical curves of the hardening rate with respect to strain. The graph indicates that the alloy not only has different strain hardening rates in the two loading directions, but also has a slightly different response to loading rates in the same direction. To be more specific, in the plastic region after $\sim 3.5\%$ strain, the strain hardening rate is slightly higher under dynamic loading along the ED/FD, whereas, it is constantly higher under quasi-static loading along the TD. Since the thermal softening effect is expected under impact loading conditions that progressively offsets the strain hardening effect [8], it is reasonable to expect a progressively smaller strain hardening rate under dynamic loading. In other words, the observed larger strain hardening rate phenomenon at dynamic loading rates versus quasi-static loading rate in the FD suggests that there is an active strain-rate-dependent hardening mechanisms that took place in that direction. It is hypothesized that this strain-rate hardening behavior is caused by the occurrence of extension twinning at high loading rates. There are several reasons for this: (1) deformation twinning, especially, extension twinning, has been frequently reported in Mg alloys with the grain size from micro- to nano-regimes [17, 65, 66], particularly, when the alloy possesses a certain texture and the loading condition favors extension twinning. (2) For pure single crystal Mg, the critical resolved shear stress (CRSS) for extension twinning is only slightly higher than that for basal slip, and activation of extension twinning and its subsequent interaction with dislocations has been held responsible for the sigmoidal stress-strain curve [67] [17]. (3) Extension twinning as well as the basal slip mechanism only show weak temperature and strain rate sensitivity [6, 7, 68, 69]. In turn, this would explain the marginal rate dependence of the strain hardening rate in the ED/FD direction. (4) Other experiments have confirmed the increase of the volume fraction of twins with the increase of either the plastic strain or the loading rate [9, 47, 48]. Furthermore, Li et al. [66] confirmed this speculation by XRD measurement, and meanwhile provided a reason for the difficulty in detecting deformation twinning in post-loading Mg and Mg-alloy samples with grain size in the submicrometer regime. Hence, we attribute the anisotropic appearance of the strain-hardening rate in Figure 8(a) to the initiation of extension twinning; although, it has not been detected in our TEM observations. Further, in this context, there is a competing effect as a function of the strain rate on the strain

hardening rate. On the one hand, extension twinning and basal slip have usually been considered athermal, which explains the only marginal rate dependence of strain hardening rate. On the other hand, a high rate favors the occurrence of deformation twinning in the sense of the Zener-Hollomon factor [43], which, in turn, increases the strength of interaction between deformation twinning and dislocations and exaggerates the sigmoidal feature of the stress-strain curve, and thus the total strain hardening rate of the alloy. These factors taken together may help to give a better, more holistic understanding of the strain hardening behavior of the extruded alloy.

As illustrated by the experimental results in Figure 3, the flow behaviors in ED/FD and TD of AMX602 exhibit similarity and yet with distinct features associated with each loading direction. First, both orientations show the three distinct stages in the plastic regime of the stress-strain curve. But the ED/FD direction has a flatter and wider “easy glide” region (Stage I). As has been pointed out, the sigmoidal stress-strain curves both in ED/FD and TD are to be attributed to the occurrence of extension twinning which tends to rotate the crystals by 86 degrees. During this process, some grains may be rotated to certain orientation where CRSS of basal slip or other slip mechanism can be satisfied. As such, dislocation mechanisms start to contribute to the plasticity. At the same time, the interaction between deformation twinning and dislocations becomes more prominent, and the strain hardening rate becomes larger. This mechanism can be exhausted until the role played by extension twinning is saturated, and the strain hardening rate starts to level off, eventually becoming ineffective [70].

Recently, it has been noticed that Mg as well as Mg alloys tend to fail by ASB which is a mode of plastic instability for certain visco-plastic materials [52] [53, 71]. Conventionally, two approaches have been employed to investigate the problem of ASBs. In the first, an experimental approach identifies which material (intrinsic) and external (extrinsic) factors are identified that promote or retard/suppress ASB [72]. It has generally been agreed that materials with low strain hardening, small strain rate hardening (small SRS values) and high strength exhibit a tendency for adiabatic shear localization under dynamic loading. Artificially introduced concentrated zones of shear stress also tend to promote ASB; such forced scenarios can be realized via careful specimen geometry design, e.g., the hat-shaped specimen, widely used in the community[73]. In the second, the approach is toward the understanding of the physics, mechanics and mathematics of ASB formation by solving governing equations along with various materials-based constitutive equations. In general, this latter approach confirms the former, the experimental

observations. In this context, the occurrence of ASB in the extruded AMX602 Mg-alloys that show strong strain hardening needs further, a more in-depth examination which is as follows.

As has been well known, the generation and storing up of lattice defects, such as dislocations, deformation twinning, grain boundaries and their interactions constitute the fundamental or underlying mechanisms of strain hardening. In contrast, strain softening mechanisms may include DRX, adiabatic temperature rise (especially, under high strain rate loading), development of texture, etc. As such, at least under dynamic loading conditions, the hardening factors are competing against the softening factors. As the hardening factors are progressively exhausted, the effects of the softening factors may still continue, and may eventually outpace hardening and lead to an overall net effect of thermal softening. In turn this favors ASB formation. Our experimental results indicate that for AMX602, at a strain of ~ 0.09 , the strain hardening rate reaches its peak. In other words, this corresponds to the inflexion point on the sigmoidal stress-strain curve. It has been argued that the existence of such an inflexion point may be the critical condition for ASBs in Mg-alloys with a strong strain hardening behavior [52]. In the following section, we propose a basis for the occurrence of ASB in AMX602. First, if we consider that roughly $\sim 90\%$ of the plastic work is converted into heat during high-strain rate loading [74], the adiabatic temperature rise in the localized region could be estimated through the following equation:

$$\beta V_{spec} \int_{\varepsilon_{start}}^{\varepsilon_{end}} \sigma_{ij} d\varepsilon_{ij} = \rho V_{loc} \cdot C_{spec} \cdot \Delta T \quad (1)$$

where

β is the Taylor-Quinney factor, or the ratio of the thermal energy converted from the work done under the action of the applied force (i.e., in this work, β is taken to be 0.9);

V_{spec} and V_{loc} are the volumes of the specimen and the region of localized shearing;

ε_{start} and ε_{end} are the strains at the onset and failure due to localized shearing;

σ_{ij} and ε_{ij} are the stress and strain tensors (here only the uniaxial components are considered and Poisson's effect is neglected); and

ρ , C_{spec} and ΔT are the mass density, heat capacity and temperature rise.

As exemplified in Figure 6(b), in this work, the width of the shear band of the dynamic specimen is $\sim 6.0 \mu\text{m}$. Since the onset of non-uniform deformation should be larger than the

width of the shear band, we assume that the width of the non-uniformly deformed region is ~ 10 times the width of the ASB. Once the ASB occurs, the stored energy within the entire specimen is released within this narrow band; this is the physical meaning of Equation (2). Figure 9 is a schematic illustration of a pair of conjugated ASB for the calculation of the volume of localized deformation. The calculation is for a fully developed ASB which runs through the specimen. The post-loading dimension of the specimen is used which is measured from the SEM image: height is 1.6mm and the square loading faces are 2.65 mm wide (20% strain). The volume is for the two conjugate ASB as shown in the schematic illustration. Then the volume of localized plastic deformation, V_{loc} , corresponding to a rectangular slab, can be calculated as:

$$V_{loc} = 2(1.33^2 + 1.6^2)^{1/2} \cdot 2.65 \cdot 0.066 = 0.73 \text{ mm}^3$$

In addition, taking $V_{spec} = 12.5 \text{ mm}^3$, $\rho = 1.74 \text{ g/cm}^3$, $C_{spec} = 1.02 \text{ J/g}\cdot\text{K}$ and $\beta = 0.9$, one can integrate from a strain of 0.09 to the end of the deformation, see Figure 8. As such, one obtains an adiabatic temperature rise of $\sim 1000 \text{ K}$. Considering that the strain localization can be more intense within the center of the shear band, the temperature rise close to the fracture surface could be underestimated. On the other hand, the melting temperature of Mg is only around 923 K. Although the calculation yields only an approximate value, there is no doubt that a very high temperature is reached in the shear bands as indicated by the morphology in Figures 6 (b, c, and d). The smooth features of the fracture surface in Figure 7(a) can also be interpreted as evidence for local melting.

Unlike those conditions operating under dynamic strain rates, at low (quasi-static) loading rates, the heat generated would have enough time to diffuse into the whole specimen; therefore, the effects of the temperature rise in the specimen was not as noticeable. That is why there was no evidence of local melting but only dimples (ductile failure) on the fracture surface under quasi-static loading.

5. Summary and Concluding Remarks

The mechanical properties, particularly, the plastic deformation and failure mode, of an Mg alloy AMX602 were investigated under uniaxial quasi-static and dynamic compressive loading. With the assistance of SEM and TEM, the microstructure and fracture surfaces of the alloy were examined and the underlying deformation and fracture mechanisms were discussed. Several

conclusions have been drawn based on the experimental results and discussion.

1. The specific alloy design and the material processing via melting, fast quenching, compaction and hot extrusion is effective in obtaining ultrafine equiaxed grains for this alloy. The consolidation by extrusion at high temperature assists the formation of an intermetallic compound (i.e., Al_2Ca) with size ranging from ~ 100 nm to ~ 1 μm . The intermetallic strengthens the alloy two-fold: ultrafine grain size retention via Zener pinning centers as well as lend themselves to Orowan strengthening.
2. Both dynamic and quasi-static compression curves of the extruded alloy show sigmoidal stress-strain curves when loaded either in the ED/FD (extrusion/flow) or in the TD (transverse) directions, pointing to the operation of extension twinning in the specimens and subsequent interaction between extension twinning and dislocations. For a given extrusion condition, the strain hardening rate reaches its peak at about the same strain level, irrespective of the loading rate.
3. ASBs were observed in AMX602 at dynamic loading rates. The temperature rise in the shearing zone could exceed the melting point of Mg (923 K), which results in local melting of the ASB region. Based on observations and a discussion of the resultant fracture surface, it may be concluded that adiabatic shear failure is responsible for the final failure under dynamic loading, while only ductile fracture occurs under quasi-static loading.

6. Acknowledgments

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Figure Captions:

Figure 1. (a) Bright field image of AMX602 extruded at 250°C. The image shows a fine microstructure with small precipitates. (b) The corresponding selected area diffraction pattern of the sample showing nearly continuous ring pattern. (c) EDS results of the selected square-shaped area in (a). Note the strong elemental Ca signal in the EDS spectrum.

Figure 2. SEM micrographs at two magnifications of the AMX602 Mg alloy, extruded at various temperatures. (a₁ and a₂): 250°C; (b₁ and b₂): 300°C; and (c₁ and c₂): 350°C. The white contrasted areas, indicated by arrows in the images, correspond to the clustering of the fine Al-Ca intermetallic precipitates.

Figure 3. Compressive true stress-strain curves of the AMX602 Mg alloy extruded at various temperatures (extrusion temperatures are given in the insets). Results in the ED/FD (extrusion/flow direction) are given in (a₁ and a₂) and those in the TD (transverse direction) in (b₁ and b₂), under both quasi-static (QS) and dynamic (DY) loading conditions. Note that the extrusion temperature has only a marginal effect on the dynamic mechanical properties of the alloy (compare a₁ and b₁). Under quasi-static loading, the lower extrusion temperature leads to a higher flow stress at the initial stage of plastic flow. This difference is smeared out in the strong strain hardening regime. Under both quasi-static and dynamic loading, all specimens exhibit easy flow at the onset, followed by strong strain hardening which levels off at relatively large strains. In light of this observation, we may divide the plastic flow of this alloy into three regimes: Stage I is the easy flow; Stage II is strong strain hardening and Stage III is declining strain hardening.

Figure 4. The nano-hardness *versus* strain rate of the AMX602 Mg alloy extruded at different temperatures (tested in the flow direction). Both strain rates and hardness are plotted on a logarithmic scale and, therefore, the slope of each straight line represents the strain rate sensitivity (SRS) of the specimen. A clear trend can be seen showing that a low extrusion temperature results in a slightly higher strain rate sensitivity.

Figure 5. Representative post-loading specimens of the AMX602 Mg alloy recovered from quasi-static (a) and dynamic testing (b). The alloy specimens shown here were extruded at (a) 250°C and (b) 350°C, respectively, and the specimens are from the transverse direction. In (a), the specimen failed along a primary crack at *ca.* 45° with respect to the loading direction (roughly along the horizontal in the image). That is, the specimen failed into two pieces along the plane of maximum shear stress. This deformation and failure mode is reminiscent of the failure of bulk metallic glasses (BMGs). However, the difference between AMX602 and BMGs is that in the latter, there is nearly no global plasticity present before failure. In (b), the specimen failed into several pieces. The two primary cracks have a conjugated pattern, indicative of adiabatic shear localization and concomitant final failure along the shear band.

Figure 6. (a) Low-magnification image of an incomplete adiabatic shear band (ASB) of an AMX602 specimen (alloy extruded at 250°C with the sample along the transverse direction) under dynamic testing. The loading axis is roughly along the vertical. Images of (b), (c) and (d) are high-magnification micrographs of the boxed regions in (a), showing the details of the band. Strong evidence of an adiabatic temperature rise within the band can be observed from (b) to (d).

Melting has apparently taken place within the shear band. These images also show the evolution and development of the band.

Figure 7. Representative SEM micrographs of the fracture surface of AMX602 (extruded at 250°C and the specimens are taken from the flow direction) after (a) dynamic and (b) quasi-static loading. Under dynamic loading, the high temperature in the shear band leads to melting and evaporation of the alloy material. The microcracks might be formed due to thermal shock. Under quasi-static loading, dimples are profuse, even though failure is along a primary shear band, indicating gross plastic deformation accompanying the failure process.

Figure 8. The strain hardening rate, θ versus ϵ , of AMX602 (extruded at 300°C) at both quasi-static and dynamic loading rates. (a) along the ED/FD (flow direction); (b) along TD (transverse direction). Note that the strain hardening rate reaches its peak value at nearly the same strain value under both quasi-static and dynamic loading. Also note that the strain hardening rate is significantly higher in the ED/FD than that in the TD. The peak strain hardening rate in the ED/FD occurs later, at a larger strain value, relative to the TD.

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Table 1. Yield and maximum strengths of the AMX602 Mg alloy (extruded at different temperatures) under quasi-static (QS) and dynamic (DY) compressive loading

Extrusion Temperature [°C]	Flow Direction (ED/FD)				Transverse Direction (TD)			
	Yield Strength ($\sigma_{0.2\%}$ or $\sigma_{5\%}$) [MPa]		Maximum [MPa]		Yield Strength ($\sigma_{0.2\%}$ or $\sigma_{5\%}$) [MPa]		Maximum [MPa]	
	QS	DY	QS	DY	QS	DY	QS	DY
350	283±18	348±2	510±20	585±5	242±7	361±20	441±7	489±12
300	291±29	354±2	504±41	588±7	265±1	368±13	445±18	484±2
250	318±10	355±0	517±22	604±5	275±0	362±3	453±21	513±21