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Emerging hot topics and research questions in wrought magnesium alloy development

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Abstract

Scientific understanding of the behaviour of wrought magnesium alloys is quite mature, with literally thousands of papers published on the topic, along with several reviews. Most of this research is relatively recent, being published after the year 2000. With such a large body of work available to the reader, it could easily be missed that the field of magnesium metallurgy is poised for significant advances. Access to synchrotron and neutron scattering has revealed new knowledge about deformation and fracture behaviour, and the complex interaction between solutes, dislocations, interfaces, and disconnections is only just becoming clear. Super-computing makes it now possible to use atomic-scale simulation techniques to make material calculations such as stacking fault energy predictions, and the simulation of atom-by-atom movement at boundaries and twins in 3-dimensions is now feasible. Advanced microscopy techniques such as atom probe tomography are allowing us to examine the complex chemical nature of grain boundaries on the nano-scale. These are just some of the areas ripe with potential for new discovery. With so much information available to the reader, it can be difficult to identify those areas of study in which the greatest knowledge gaps exist or those which are most ripe for exploration. For this reason, several authors have teamed up to write this short discussion piece which highlights research areas which the authors agree have the most potential for impact in the field.

1. Texture development

Deformation of magnesium typically leads to the alignment of basal planes parallel to the principal direction of material flow. The formation of a strong basal-type texture during rolling or extrusion, which is associated with a preferential activation of basal slip or extension twinning, results in a limited capacity for strain accommodation during subsequent forming of the semi-finished products and therefore restricted ductility and formability. Furthermore, the geometrical directionality of twinning activation leads to distinct asymmetric yielding and work hardening behaviour during tensile and compressive deformation.

Alloying additions of rare earth elements (RE) like Ce, Nd, Y, or Gd as well as other elements with large atomic size, like Ca can contribute to quantitative changes in the texture, whereas common alloying elements such as Al, Zn or Mn do not have a significant impact in that regard (neither when added solely nor in combination). Alloying combinations of RE or Ca and Zn into Mg give rise to synergetic effects leading to important qualitative changes of the texture components, as well as a reduction in texture intensity. While a weak texture with basal pole spread in the sheet rolling direction (RD) is mostly observed in Mg - RE binary alloys, recrystallisation annealing of ternary magnesium alloy sheets of Mg-RE-Zn and Mg-Zn-Ca alloys generate a texture with an additional basal pole split in the sheet transverse direction (TD) [1,2]. This type of sheet texture stands out among others for its “quadrupole” characteristic, which is usually more desirable for sheet metal forming processes than a texture with distribution of basal poles along one direction.

Deformation texture changes such as those described above have been associated with a variety of mechanisms. This could particularly involve increased activation of non-basal prismatic <a> and/or pyramidal <c+a> slip [3-6], different twin types [7,8] and an increased propensity for shear banding [9]. In addition, the processing conditions, e.g. temperature and strain rate [10], also influence the activation of these mechanisms [11-15], and the extent of their relative contribution to the texture development. Several works have concentrated on implementing the effects of strengthening mechanisms on the activation of different deformation modes in polycrystal plasticity models to improve the quality of deformation texture predictions [16,17]. However, since the behaviour of individual deformation mechanisms is strongly influenced by alloying additions (through solid solution or precipitation effects), grain size, and temperature, more theoretical [18,19] and experimental work in this field remains essential to further refine the predictive capability of such models, particularly for complex loading conditions.

Experimental [20] and numerical investigations of the deformation behaviour and related texture development in magnesium alloys is further complicated by the occurrence of static and/or dynamic recrystallisation (DRX), and its interaction with active slip and twinning modes. Although in some cases – for example during extrusion – DRX was seen to weaken the basal texture component (e.g. Mg-Zn-RE or Mg-Zn-Ca alloys [2]), there are other cases where DRX was associated with the development of the typically strong textures observed in deformed magnesium (e.g. Mg-Al-Zn alloys [21,22]). The impact of DRX on the texture depends on the temperature, strain rate and applied strain. This is because these parameters dictate the operating deformation modes and the type of DRX nucleation mechanism. There are several examples in the literature documenting slight changes of texture at low deformation temperatures and an overwhelming texture randomisation at higher temperatures [7,23-25]. With respect to deformation twinning, it has been reported that DRX inside compression twins is of continuous nature, which relies on extensive dynamic recovery alongside prismatic slip in order to rotate the nucleated grains inside the twin about their c-axis resulting in subsequent texture randomisation [26,27]. In this context, the separate contributions of high temperature plasticity and DRX in unraveling the origin of texture randomisation at elevated temperatures are still to be determined. This is particularly challenging because in real cases dynamic recrystallisation cannot be treated as a superposition of deformation and static recrystallisation due to the complex dynamic interactions between responsible crystallographic mechanisms and microstructure.

For example, discerning the role that grain boundary disconnection motion has in shear-coupled grain boundary motion is only in its infancy [28-30]. To the authors' knowledge, the disconnection concept has yet to be applied to grain boundary motion in Mg, aside from the specific (and important) case of deformation twinning [31,32]. Indeed, it is well established that the low temperature deformation and crystallographic texture evolution of magnesium alloys is due to dislocation slip and deformation twinning. On the other hand, the high temperature deformation of the Mg alloy polycrystals may have contributions from intragranular slip and climb of dislocations, diffusional flow, as well as deformation twinning and other shear-coupled boundary migration processes, which may be particularly important during conditions of dynamic recrystallization.

An important change caused by alloying addition of RE or Ca is the retardation of dynamic recrystallisation, particularly the type requiring boundary migration [33]. This is considered crucial for recrystallisation texture modification, since it controls the amount of deformation-stored energy retained in the microstructure [34,35]. The retardation of dynamic recrystallisation can be due to solute segregation to grain boundaries and a resulting pinning effect [35]. It can also be attributed to a change in the recovery behaviour due to a reduction in

the I2 stacking fault energy, stabilising the extended configuration of dislocations on the basal plane [36,37]. This in turn restricts cross-slip and climb mechanisms, thereby retarding dynamic recovery. At this point, it is noted that enhanced nucleation can, in some cases, also lead to texture changes despite impeded growth, e.g. in Mg-Mn-Nd-alloys or Mg-Zn-Nd-alloys [7,38].

With respect to the role of alloying additions in modifying common basal textures during static or dynamic recrystallisation, two key aspects are usually discussed, namely their solid solubility and precipitation behaviour that could lead to solute drag and pinning effects, respectively. As an example, some alloying elements in Mg alloys, e.g. Ce, Nd, Y, Ca, were found to have a growth restriction effect on recrystallised grains. If there is a preference for these elements to form precipitates and, thus, exert a Zener pinning effect due to their relatively low solubility in magnesium, a question still remains whether they also show solid solution effects in terms of solute drag. The solubility limit of RE elements and Ca is influenced (mostly reduced) by the combined addition of other elements such as Zn [39]. From an energetic perspective, this is related to lattice misfits of solute atoms with the Mg matrix, which is believed to give rise to local solute clusters of Zn-RE atoms with drag effects [40]. Correspondingly, texture changes are more pronounced in Mg-Zn-based alloys, but not in Mg-Mn-based alloys due to the lack of stable RE-Mn compounds forming with the addition of Mn.

Finally, texture can also be weakened by appropriate processing. Some recent studies showed that texture weakening with basal poles split into the TD could be obtained in Mg-Al-Ca alloy sheets when they are produced via twin roll casting (TRC) [41]. This technique provides a feedstock having a fine and homogeneous microstructure due to the rapid cooling rate applied during the solidification stage. In addition to a fine grain size, the microstructure can be also tailored to have a weak texture with tilted basal poles away from the sheet normal direction. This has a huge beneficial impact on further steps of the process chain, particularly on finishing rolling to final gauges and subsequent heat treatments.

In summary, it is not fully understand why certain alloying elements cause a strong, qualitative texture modification although – as discussed above – various mechanisms have been proposed. Fundamentally, new knowledge is required on the influence of alloying elements on the different deformation mechanisms mentioned above, as well as on how their type and concentration may affect the balance of hard and soft deformation modes that would, in turn, strongly impact the texture and mechanical response. On the other hand, an understanding of how the different stages of recrystallisation, namely nucleation and growth, are affected by the choice of alloying elements and the active deformation mechanisms is also required. One aspect that has been considered to a much lesser degree in these discussions of the deformation processing of Mg alloys is the possible role of dislocation cross-slip, climb and other recovery

mechanisms which are frequent precursors of recrystallization. Future work should thus combine advanced modeling and high resolution characterisation techniques to shed light on all these pending questions and stimulate further research on this exciting area.

2. Ductility and Fracture

In 1962, Chapman [42] first reported that the tensile ductility of magnesium alloys increased with a decrease in grain size, and several reports since have confirmed this experimental result, and a nice summary of this effect is shown in Figure 2 in [43]. This is the opposite trend to other alloy systems such as steel and aluminium in which smaller grain sizes often lead to a decrease in ductility. The increased ductility in magnesium alloys at smaller grain sizes is attributed to the decreased prevalence of twinning in refined structures [44,45]. The correlation between grain size and ductility is a dominant effect. Texture is another contributor to ductility [23,24,46-52], textures in magnesium are uniquely strong after thermo-mechanical processing. When normalised for grain size, magnesium alloys show higher ductilities when they have weaker textures [52]. The reasons for this have not been specifically studied. Strongly textured extrusions such as those found in AZ31 form contraction twins during tensile deformation [45], but weaker textured alloys such as those containing rare earth elements do not show this behaviour to the same extent. At present, a reduction in the activation of twinning is thought to be the most likely reason for the improvement of ductility in alloys with weaker textures. However, there are other factors that might also contribute to improvements in ductility.

Different textures have a different stress distribution within the microstructure as the “hard” and “soft” orientations respond differently to the applied strain [53,54]. The effect of texture on the development of localised stresses within the microstructure of magnesium is yet to be studied, although some modelling work has already been done [55]. Slip system activity is also now suggested to be a contributor to improve ductility in some alloys [36,37,56,57]. Magnesium suffers a large anisotropy in the stress required to activate the different slip systems [58]. Solid solution strengthening can increase the relative activity of the non-basal slip systems, and rare earth elements in particular have been shown to increase <c+a> slip [48]. This non-basal slip activity is hypothesised to improve ductility, presumably through homogenisation of the deformation among the individual grains, leading to a decrease in stress localisations during deformation. So these three microstructural factors, grain size, texture and slip activity, all have an effect on ductility. Although it is broadly understood how microstructure relates to ductility, specific studies designed to quantify these effects and rank them in order of importance have not been carried out. This is probably because it is almost impossible to de-couple the different effects, especially when the texture in particular is sensitive to alloying additions. Experimental

studies are therefore limited in their scope to answer some of these questions, and an increased reliance on modelling may be required.

Although there is likely sufficient knowledge available in the literature to generate a predictive model to adequately predict the tensile ductility, one based on texture, composition and grain size, the model would be entirely empirical. The underlying causal reasons for ductility in magnesium remains yet to be quantified, noting that it is not one, but many interrelated factors, such strain rate sensitivity (SRS) and work hardening rate (WHR) have significant effects on the formability [59,60]. SRS and WHR provide a material with a macroscopic resistance to necking. Although WHR and SRS are relatively easy to measure, what these factors truly mean within a magnesium microstructure is not as clear as it would be in, for example, an aluminium alloy. This is because the activation of twinning can contribute to higher work hardening rates, but it is not entirely clear if this has the same effect on ductility as it would have on an alloy that deforms entirely by slip. The microstructural factors causing the hardening effect in these two scenarios are markedly different, because Mg alloys do not always fail due to ductile modes of plastic instability. Rather, they can transition to more brittle fracture modes when the flow stress becomes very high. The addition of solutes may also have an effect on the SRS and WHR, but this is of course in concert with modifying both the texture and relative slip activity. Again, these effects cannot easily be de-coupled.

It is also noteworthy here that ductility is measured by the standard tensile test, but sheet formability involves more complex forming pathways. Very few studies in magnesium investigate other strain paths, but studies on biaxial stretching have shown similar indications to those found in tension: weaker textures are better for formability [61-63]. Again, there are outstanding questions to answer: Can the poor formability of AZ31 be entirely attributed to the activation of twins? Or are there other factors such as slip system activity and local stress distribution that are assisting in the better formability in the weakly textured case?

One of the keys to the deformation mode – ductility relationship that is evidently so important in magnesium and its alloys is the process of void formation. This comprises their nucleation, growth and coalescence that eventually lead to ductile failure. Wilkinson, Nemcko and co-workers [64-67] have performed micro X-ray computed tomography (μXCT) experiments to examine these processes. As one would expect, the void growth and coalescence in magnesium shows peculiarities that relate to strong local gradients in strain [68]. Voids do not grow uniformly, and the nearby presence of twins, grain boundaries and particular orientations exerts a significant impact on void shape and growth rate. Void nucleation progresses steadily with strain and seem to concentrate on boundaries, particularly twin boundaries and second phase particles. In many cases, a form of the void sheeting mechanism is what facilitates final failure

[64,66]. Recent work suggests that a critical void fraction must be attained before void sheeting initiates [69]. This critical fraction will most likely be dependent upon the texture, deformation mode activity and the second phase. Prolonging the strain until this critical void fraction is attained is thus the key to extending ductility. Grain refinement tends to suppress twin formation, which lessens the contribution of twins to void nucleation, thus allowing more strain to accumulate before the critical fraction is reached. Grain refinement also reduces the length of local deformation gradients and this leads to void growth slowing down once voids attain to the grain size. Thus, in a fine-grained material, critical fractions of voiding are only attained once significantly higher number densities of voids form, which allows for more strain to accumulate.

The effect of texture on void formation requires further study, but it is evident that texture variations do indeed modify the rate of void formation at grain boundaries as well as within twins [70]. Interestingly, when wrought material is loaded in bending, the ductility of the material on the compression side of the bend can become the controlling factor [71]. Reports of cracked inner radii testify to the relevance of this phenomenon. Under these circumstances, the knowledge gained from tensile experiments has less import. Ductility in bending is still an area worthy of further research, despite the general issues having been known for a very long while.

3. Effect of particles on deformation

Particles can be effective strengtheners in magnesium alloys by acting as obstacles to dislocation motion [72]. They can therefore have a profound effect on deformation behaviour, although the strengthening obtained by precipitates in magnesium alloys is approximately 5 times less than that obtained in the strongest aluminium alloys [58], suggesting there is considerable scope for improvements through metallurgical design.

In the case of the common alloy system Mg-Al-(Zn), the precipitates that form are large and incoherent, and develop as large plates on the basal plane. These precipitates cannot be sheared, and Orowan theory [73] gives reasonable prediction of the strengthening effect. Therefore, minimising the gap between precipitates through choice of an appropriate precipitate type and distribution provides the greatest strengthening. Of the common precipitate types observed in magnesium, plates on the prismatic planes have been expected to be most effective [72,74], since they not only strengthen most against basal slip but also provide good obstacles to prismatic slip and twinning when sufficiently large to remain unsheared.

Coherent or semi-coherent (often metastable) phases are also observed in several magnesium alloys. These may be sheared by dislocations [12,75-77]. The shear resistance of a precipitate may be controlled by effects at the interface [72] or the creation of an antiphase boundary (APB) inside the precipitate [76], amongst other factors. These observations have much in common with deformation in precipitate containing aluminium alloys, but with the important difference that the different dislocation types in magnesium (e.g. basal <a>, prismatic <a>, <c+a>) have different character, and thus different shearing ability. Furthermore, precipitates in magnesium are highly anisotropic and thus may be sheared more easily on certain planes than others [72].

Evidence to date suggests that c-axis rods and prismatic plates can be sheared by basal <a> dislocations (in Mg-Zn and Mg-Y-RE alloys respectively) but non-basal dislocations do not appear to shear these precipitates [72]. Similar results were reported for coherent β''' in Mg-Nd [70,77] but the same study found that β' precipitates in Mg-Y were also sheared by prismatic and <c+a> dislocations. Therefore, although shear by basal <a> dislocations is observed to be the easiest [75-77], shear by non-basal dislocations is possible in some cases [77].

The study of precipitate shearing in magnesium remains in its infancy. Unanswered questions include the nature of the dislocations inside the precipitates, and the relative resistance to shear by the different dislocation types. Despite this, the characteristics required to maximise shear resistance of coherent precipitates can be identified and include a favourable precipitate morphology and habit [72] combined with a high APB energy [76]. First principles modelling has great potential to identify such phases.

Particles also inhibit deformation by twinning [78,79]. Precipitates do not prevent twin nucleation, and often lead to an increase in twin number density (but a decrease in size) at a near constant twin volume fraction [73,80-82]. Particles can act to block the tips of twins or inhibit twin thickening [73, 82]. Particles have only been observed to be sheared by twins when they are thin plates with a similar crystal structure to the matrix [83]. In most cases, particles remain unsheared inside the twins, in which case there is a strain incompatibility leading to a backstress that can provide the dominant strengthening effect [84,85].

To resist twinning, precipitates should be shear resistant in the direction of the twinning shear. They should also be elastically stiff to maximise the backstress effect [85]. The should present sufficiently large obstacles to be capable of blocking twin tips [85], whilst being closely spaced on the twin plane to maximise the bowing stress for the twinning dislocations [85]. Alloy design has yet to focus on developing twinning resistant microstructures, but such an approach has significant potential benefits to increase strength or control anisotropy and potentially improve ductility.

4. Where to from here?

In the last 20 years, understanding of wrought magnesium alloys' behaviour has improved markedly, and this can largely be attributed to the application of various microscopy techniques (for example [86-89]) along with the rapid uptake of elasto- and visco-plastic self-consistent (EPSC and VPSC) and other polycrystal plasticity modelling approaches to quantify deformation behaviours (for example, [53,54]). Although the field has come a long way, there are still some areas ripe for study.

Second phase particles. It is apparent from the work done on both slip and twinning that the inclusion of particles into a microstructure can have the dual beneficial effects of strengthening the alloy and potentially alleviating the tension-compression asymmetry. The recently discovered shearability of particles by basal dislocations [75] helps to explain the rather modest strengthening potential of these particles, and the development of non-shearable particles could lead to greatly enhanced strength. An increase in the number density of particles is also a key factor for improvement in age hardening of magnesium-based alloys [90]. Closely linked to design of the particles is understanding of how they interact with twins. Although the case for slip is relatively well studied in many systems, magnesium is one of the first metallurgical system in which the interaction of precipitates and twins has been closely examined. A deeper understanding of this will be critical in determining how best to design precipitates for improved wrought behaviour.

Solute behaviour. Extensive experimental investigation of the effect of solutes on recrystallisation texture development have been carried out, and there is a fairly extensive empirical understanding of the different solutes, particularly the rare earth elements, which modify the texture (for example, see [1-5,7,8]). What is lacking is an understanding of why this is the case. The current inability to predict the effect of solute species on the recrystallisation texture development is not unique to magnesium. Other well studied systems still rely on empirical models to design processes, the classical case being control of cube texture in aluminium sheet processing to limit earing [91]. Any research which allowed us to understand and predict texture development would be of great benefit to the broader metallurgical community.

The effect of solutes on the deformation behavior is also an area ripe for study. It is known that solutes can affect the non-basal slip behaviour [36,37], and this has been linked to changes in fracture and tension-compression asymmetry. Again here there are some studies, but there is not yet a full library of information on how different solute species and concentration levels

affect the different mechanisms. One very interesting area yet to be examined closely is the interaction of multiple solute species within the matrix. Different species can co-segregate to twin boundaries [87], and this implies that they will also co-locate at dislocations, or even bind with each other in the lattice. The effect of these aspects on solute strengthening and work hardening is yet to be considered. Clearly there are some extremely fertile research questions still to be answered in the field of wrought magnesium alloy behaviour, and the authors are hopeful that this collation of thoughts will be of benefit to the research community.

Advanced computational techniques. Modern developments in high performance computing have enabled the study of computationally intensive problems. In particular, this has allowed us to study atomistic behaviors that cannot be measured experimentally. This led to the discovery of solute effects on SFE and dislocations [36,37], probably the first of many similar observations yet to be made. Molecular dynamics has also been used to study the atomic-scale behavior of twins in 3-dimensions [92], with these first studies being the very beginning of this new field of endeavor. The most novel application of computational modelling is its combination with other techniques, such as the use of the similarity index [93], to search for new alloys. The CALPHAD approach was actively applied to the study of casting alloys [94], but the approach has been less frequently invoked in studies of wrought alloys. Finally, the application of artificial intelligence and machine learning is ubiquitous in research at this time [95]. The complexity of wrought Mg alloy behavior seems ripe for the application of these methods. Indeed, there is an ever-increasing scope for the utilization of computational methods, and the coupling of computation and experiment are likely to spear-head many new discoveries into the future.

RE-free alloys. As the world's resources dwindle, eliminating high cost and limited resources like RE elements from magnesium alloys will become necessary. In addition, narrowing the range of compositions on the market may also become a necessity to enable optimum alloy recycling at the end of component lifetime. For these reasons, and of course reasons of cost, there is likely to be great benefit in developing alloys that do not require RE elements.

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