Invited Viewpoint

Broadening the design space of engineering materials through "additive grain boundary engineering"

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ABSTRACT

Grain boundary engineering (GBE) is one of the most successful processing strategies to improve the properties of polycrystalline solids. However, the extensive thermomechanical processes involved during GBE restrict its use to selected applications and materials. In this viewpoint paper, we discuss the opportunity provided by additive manufacturing (AM) technology to broaden the applicability of the GBE paradigm and, consequently, the design space for engineering materials. By integrating specially-designed thermomechanical processing within AM, it would be possible to produce bulk, near-net-shape parts with complex geometry and GBE microstructure. We discuss the major challenges in this endeavor and propose some possible strategies to achieve this goal, which we refer to as "additive-GBE".

The promise of grain boundary engineering

Amongst the many processing strategies that have been conceived to improve the properties of polycrystalline materials, grain boundary engineering (GBE) deserves a special mention. By manipulating a small fraction of the atoms in the solid—namely, those which are located at grain boundaries (GBs)—GBE leads to dramatic changes in properties [1, 2], including ductility [3], fatigue [4], creep [5], hydrogen embrittlement [6, 7], and corrosion behavior [8].

GBE involves applying a sequence of thermomechanical processes to a target material, which typically consist of cyclic plastic deformation and high temperature treatments (Fig. 1). As such, GBE is a prototypical metal processing strategy, much like those employed in ancient times to improve the strength of metal alloys. The resulting microstructure exhibits a significantly different distribution of GBs, with higher fractions of low energy GBs [9, 10]. The change in the GB character distribution, which can be thought of as a "survival of the fittest", is the result of microstructure recovery and recrystallisation upon heat treatment [10, 11]. During recrystallisation, new, strain free grains nucleate and grow into the

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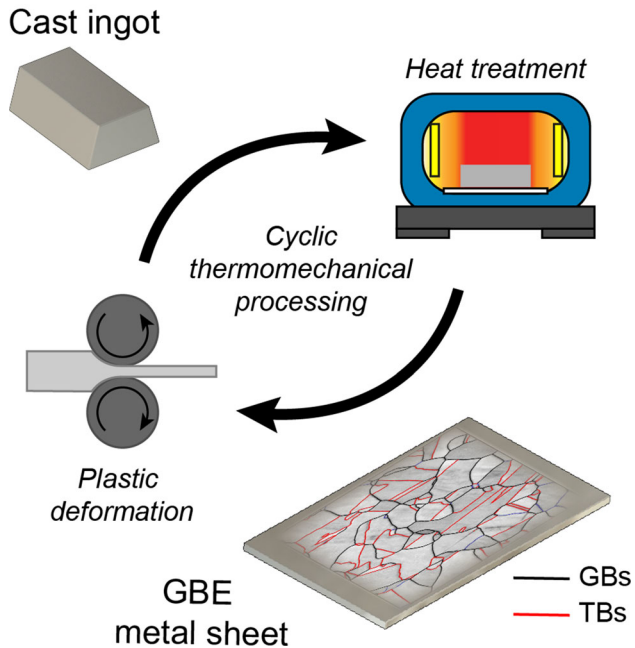
Conventional GBE

Figure 1 Schematic of conventional grain boundary engineering

surrounding microstructure, reducing the stored energy which was introduced through the mechanical deformation step in GBE. As these grains grow, they promote the formation of new GBs. Amongst those, low energy GBs tend to remain in the recrystallized microstructure because they typically exhibit reduced mobility [12, 13].

In materials that are characterized by a low-stacking fault energy, GBE yields recrystallized microstructures with copious coherent twin boundaries (TBs). Owing to their perfect atomic registry, coherent TBs have the lowest energy and the highest thermal stability amongst all GBs. As such, they are frequently observed in recrystallized microstructures and may survive the multiple strain-annealing cycles during GBE [14, 15]. Coherent TB formation is associated with stacking sequence errors that occur during GB migration upon recrystallisation [16–18]. It follows that, as a recrystallized grain grows, multiple coherent TBs can form. These highly twinned grain clusters disrupt the connectivity of general, high-energy GBs, improving the GB-governed properties of the material. Besides coherent TBs, GBE may promote the formation of other low-energy GBs, including incoherent TBs and other twin-related GBs. These GBs form as different twinned grain clusters coalesce

and interact with one another throughout the repeated strain-annealing cycles in GBE [13].

The concept of GBE was initially proposed by Watanabe et al. in the 1980s [19], convincingly demonstrated by Palumbo et al. on a variety of metal alloys a decade later [3, 5, 20, 21], and widely explained by Randle et al. in the 2000s [12, 13, 22, 23]. Since then, GBE has proliferated into a myriad of different adaptations and has been applied to a broad range of materials [8, 20, 23, 24], including non-metals [25, 26]. Despite the intense research and large number of success stories, however, very few modern industrial applications employ GBE materials [27, 28].

What limits the broad application of GBE?

One of the possible reasons why GBE is not employed ubiquitously in industry is the limited flexibility on part geometry that it provides. Because of the large plastic strain required to trigger recrystallisation, GBE materials generally come in the form of sheets or tubes [29], as a result of the mechanical processes chosen to yield uniform and controlled deformation (e.g., rolling [30], drawing [31] and equal-channel angular pressing [32]). Thereafter, these materials require additional machining or forming to be shaped into a final product. When combined with the thermomechanical treatments required for GBE, the entire manufacturing process becomes time- and cost-intensive. Moreover, the range of parts that can be produced by sheet or wire forming is limited. For these reasons, GBE is not applied to bulk, three-dimensional (3-D) parts or components with intricate geometries. In these cases, the common practice is to rely on surface or coating technologies to minimize intergranular degradation, especially at high temperature and in corrosive environments [33].

Another drawback of GBE is the negative impact it has on materials strength, which is one of the main criteria when designing metals and metal alloys for structural applications. Since GBE relies on recovery and recrystallisation, the resulting polycrystals exhibit low densities of dislocations and low-angle GBs, as well as grains made larger by the heat treatment. Thus, the material loses both strain- and GB-hardening. Only rarely have researchers claimed an increase in material strength upon GBE. This trade-

off between strength and other GB-controlled properties further restricts the application of GBE strategies to the surface of engineering components to avoid affecting the material's bulk strength [34, 35].

Revamping GBE through additive manufacturing

Additive manufacturing (AM) is regarded as a disruptive technology owing to its unique capability of producing bulk, near-net-shape parts by stacking layers of material into complex 3-D geometries. The unprecedented design freedom provided by AM offers many advantages over traditional manufacturing routes, including part count reduction [36], incorporation of intricate internal channels and chambers [37, 38], and improved strength-to-weight ratio of structural components [39, 40]. Beside these geometry-enabled advances in part design, AM opens many new opportunities for a microstructure-based design of materials [41]. The layerwise nature of the process, in fact, makes it possible to apply materials processing strategies—such as GBE—directly on individual layers as parts are produced (Fig. 2). The benefit of a layerwise GBE, which we refer to as additive-GBE (A-GBE), is that it would enable the direct production of bulk metal parts with both GBE microstructure and near-net-shape,
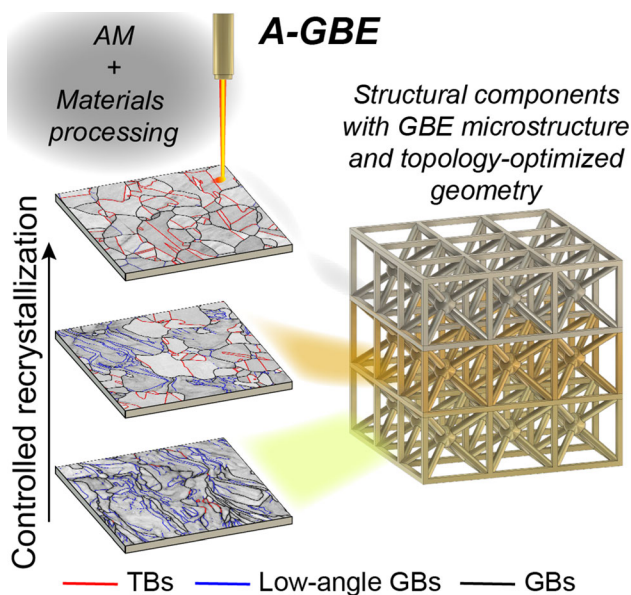
topology-optimized geometry. As a result, A-GBE parts could be endowed with lightweight and enhanced resistance to intergranular degradation. This strategy could also be more energy- and cost-effective compared to conventional GBE, owing to the reduced temperature and mechanical deformation required to activate recrystallisation on each layer, as opposed to the entire part.

Some early studies have explored the possibility of processing materials using hybrid manufacturing approaches, which concurrently combine additive technologies with tooling to do mechanical work on the build [42]. Some notable examples include in-line rolling [43, 44] and forging [45] to refine the microstructure in directed energy deposition (DED) processes, or in-situ laser or shot peening [46, 47] to produce compressive stresses and raise the strain energy in materials produced by laser powder bed fusion (LPBF). The first strategy is restricted to AM parts that tolerate low dimensional accuracy, since the repeated deformation may change the build geometry substantially. The second overcomes this limitation but may only lead to partial recrystallisation of the material due to the relatively shallow depth of the deformation zone.

Another possible approach is to leverage the inherent strain energy formed during AM to activate microstructure recrystallisation, especially in additive processes that involve melting of the material feedstock, such as LPBF and DED. Because of the highly localized melting, steep thermal gradients, rapid cooling rates, and repeated thermal expansion and shrinkage cycles, materials produced by these processes exhibit highly non-equilibrium microstructures containing copious dislocation densities [48–50], deformation-induced defects [51], and large residual stress [52, 53]. All these features raise the driving force for recrystallisation [54, 55], which may be activated via post-production heat treatments. Indeed, parts produced by fusion-based AM are routinely heat treated to relieve residual stresses and to homogenize the microstructure [56, 57]. However, microstructure recrystallisation in most of these materials only occurs after exposing them to very high temperatures for long times [58, 59]. The shortcoming of these extensive heat treatments is that they may coarsen the microstructure and even yield the formation of unwanted phases, which would impart below-average mechanical performance to the alloy.

Figure 2 Conceptual schematic of "additive grain boundary engineering"

While the abovementioned studies delineate a pathway towards A-GBE, we are still far from devising a systematic and robust A-GBE strategy. The major challenge is to produce the necessary energy required to activate recrystallisation without compromising the geometry of the build or introducing detrimental residual strains which could lead to part failure [60, 61]. In the following sections, we discuss alternative routes that could lead to A-GBE as well as some intriguing applications of it. We believe that both aspects will be the focus of intense research in this field in the near future.

An outlook on A-GBE in fusion-based AM

It should be noted that the density of coherent TBs and other twin-related GBs—hereafter generally referred to as TBs—in parts produced by AM is very limited in general [48, 51, 62]. Thus, A-GBE must rely on recrystallisation. As mentioned in the foregoing sections, it may be possible to activate recrystallisation and promote the formation of copious TBs by tailoring the non-equilibrium microstructure imparted by AM (and more specifically fusion-based AM). As many other phenomena that underpin the formation of materials and the evolution of their microstructure, recrystallisation requires a driving force and heat to overcome an energy barrier. In other words, the propensity of a material to undergo recrystallisation depends on how much strain energy is stored in the microstructure and how easy it is for new, recrystallized grains to nucleate and grow. In the quest for A-GBE, both aspects may be tuned concurrently through careful selection of AM processing parameters and/or by integrating layerwise mechanical treatments during AM.

Deformation-free A-GBE

Much of the strain energy required for recrystallisation is inherently generated during fusion-based AM. Indeed, the density of geometrically necessary dislocations found in metal alloys produced by LPBF ranges between 10^{13} m^{-2} and 10^{14} m^{-2} [50, 63]. In theory, this residual strain should be sufficient to activate recrystallization at temperatures compatible with industrial standards [30, 64] without any additional mechanical treatment. In practice, however, most of these AM alloys are thermally stable up to

much higher temperatures [58, 65]. This thermal stability stems from the presence of a fine solidification structure, which includes pronounced micro-segregation of solute atoms at cell or dendrite boundaries as a result of constitutional undercooling at the solidification front [62]. This structure hinders the onset and progression of recrystallisation despite the large driving force contained in the microstructure. In a recent work, we have shown how "weakening" this solidification structure by employing AM processing parameters that limit micro-segregation allows for recrystallisation to occur at progressively lower mechanically-induced strain [62].

Another microstructural feature that hinders recrystallization of alloys produced by fusion-based AM is second phase precipitates, such as oxide nanoparticles [66]. These particles are thought to originate from the melting of oxidized contaminants contained in the powder feedstock [67, 68]. Due to the rapid solidification and high cooling rates the material undergoes, these solutionized impurities precipitate and form nano-scale particles. As in the case of micro-segregation, these particles pin GB motion during recrystallisation. By reducing the oxygen contamination level during the AM process (both in the processing chamber environment as well as in the powder feedstock), or by controlling the material's cooling rate, it should be possible to limit the presence of these nanoparticles or reduce their size substantially; to a point where they would not refrain the growth of recrystallized grains.

While deformation-free A-GBE has yet to be demonstrated, we believe that devising strategies that simultaneously minimize GB pinning while raising the driving force for recrystallisation may prove successful. The latter could be achieved by employing unconventional laser sources [69], or laser processing methodologies [46] that promote higher residual strain in the as-built microstructure. Whatever the approach, a challenge will be to make such strategies scalable. Residual strains could add up and yield failure during production of large-scale parts, such as cracking, delamination, or distortion. Moreover, in these cases it may be more difficult to control the material's thermal history and thus the solidification structure.

Heat treatment-free A-GBE

Another interesting feature that may facilitate A-GBE in fusion-based AM processes is the intrinsic heat treatment resulting from the repeated melting and solidification of individual layers. As the high-energy source (either a laser or an electron beam) scans the layer, it generates a heat affected zone that starts from the fusion boundary and extends into the solid material surrounding the melt pool [70]. By selecting different processing parameters, the heat affected zone may be tuned to positively affect the microstructure of the solidified material; for instance by triggering phase transformations [71], or activating recrystallisation [51, 72]. Recently, Laleh et al. [73] found high fractions of TBs in the as-built microstructure of stainless steel and attributed this unusual phenomenon to poor heat dissipation during LPBF. Although their GB character distribution is dominated by high-mobility incoherent TBs, their work showcases the possibility to capitalize on the cyclic intrinsic heat treatments to activate dynamic recrystallization or recovery during the AM process. The advantage of this approach is that the parts produced would not require a GBE-specific heat treatment to activate recrystallisation, which would decrease production time and cost. Moreover, dynamic recrystallisation could also mitigate long standing problems related to the large residual stresses found in as-built AM parts [61].

Site-specific A-GBE

Because material and geometry are formed concurrently, point by point, during AM, parts may be produced with dissimilar microstructures using processing parameters that vary site-specifically. When controlled, this microstructure heterogeneity may impart additional functionalities to the build and have positive effects over parts performance. Some notable examples of this strategy can be found in the realm of surface engineering [74] or thin films technology [75], where such a heterogeneity can produce additional strengthening mechanisms and even help overcome the strength-ductility trade-off in metallic materials [76–78]. These "microstructure architectures", however, are typically restricted to small scale materials because of the limitations associated with the respective manufacturing processes. With AM, these microstructure designs may be

extended to bulk materials containing site-specific textures [41], directional solidification structures [79], dissimilar grain structures [80], composition gradients [81], and multiple phases [71]. In the context of A-GBE, site-specific microstructure control could be used to engineer the density of nucleation sites for recrystallisation across the build—for instance by selectively weakening the solidification structure. A low nucleation density would lead to the growth of large twin-related grain clusters separated by a sparse and disconnected network of high-angle GBs [82]. These high TB-density microstructures could exhibit properties comparable to those of materials that undergo several strain-annealing cycles following conventional GBE processes.

By controlling these microstructural features site-specifically, A-GBE could also enable the production of materials that integrate completely different GB character distributions [72], which would be impossible to attain via conventional GBE routes. One possible approach to achieve this goal is to tune the thermal stability of metal alloys site-specifically to alternate between regions that undergo recrystallisation and regions that do not. Alternatively, mechanical work could be applied only on specific regions of the build during hybrid manufacturing processes [72]. One benefit these microstructures could bring is to overcome the trade-off between enhanced GB-controlled properties and material strength in GBE materials. By designing the optimum fraction of recrystallized (i.e., soft) and non-recrystallized (i.e., hard) microstructures as well as their spatial distribution, parts could be made with high corrosion resistance and high strength at locations that best suit the constraints imposed by the target applications. We believe that these designs could be of interest for applications that require engineering alloys to operate in harsh environments.

Beyond twin-related GBE

This viewpoint focuses on TB-related GBE. However, TB multiplication through recrystallisation is restricted to materials with low stacking fault energy. While many engineering alloys fall under this category, including nickel, iron, and titanium alloys, others such as aluminum alloys are excluded from it. However, there are other types of GBs which could improve the properties of polycrystalline solids. For instance, some recent studies pinpointed the

beneficial effects of low-angle GBs on intergranular corrosion of aluminum alloys [83–85] and on the strength of stainless steel [86, 87]. The possibility offered by AM to control the crystallographic texture and local crystallographic misorientation [41] in the build opens the path to tailoring the occurrence of different types of GBs to improve the properties of any material. Moreover, this capability would significantly expand the design space of A-GBE materials to include alloys with site-specific regions dominated by high- or low-angle GBs arbitrarily distributed across the build.

Besides controlling the character distribution of GBs, AM may be pivotal to engineer their chemical composition, which provides an additional route to enhancing GB-governed properties of polycrystals. Raabe et al. [88] demonstrated that solute segregation at GBs may improve boundary cohesion, lower the boundary energy, and even promote local phase transformations. Manipulation of GB segregation during AM has been shown effective at mitigating hot cracking in nickel-based superalloy [89] and high entropy alloys [90]. These strategies involve, for instance, designing novel AM alloys that contain solute elements with low solute solubility and high strengthening power [91], or adjusting the AM process parameters to manipulate the cooling rate and thus tailor the GB segregation level [92, 93].

For now, the materials that may be produced via these unconventional processing routes may not have obvious applications or functionalities that can be easily envisaged. However, it is only a matter of time before researchers in academia and industry start considering how to capitalize on these untapped opportunities to address the problems of tomorrow.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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