



Grain size softening effect in intermetallics



M. Raviathul Basariya^a, N.K. Mukhopadhyay^a, Sriharsha Sripathi^b, K.A. Padmanabhan^{c,*}

^a Department of Metallurgical Engineering, Indian Institute of Technology, Banaras Hindu University, Varanasi, 221 005, India

^b School of Engineering Sciences and Technology, University of Hyderabad, Prof. C.R. Rao Road, Hyderabad, 500 046, India

^c Centre for Nanotechnology, University of Hyderabad, Prof. C.R. Rao Road, Hyderabad, 500 046, India

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ABSTRACT

Inverse Hall–Petch/grain size softening effect is a phenomenon, which when present, leads to a decrease in flow stress with a decrease in grain size. The manifestation of this phenomenon at room temperature in nanostructured intermetallics, including some of our new results, is discussed here. Following earlier work, the IHP effect is attributed to the loss of intra-crystalline dislocation motion control to mesoscopic (\geq a grain diameter) grain/interphase boundary sliding controlled flow, which evidently is confined to the grain/interphase boundary regions. Equations for estimating the free energy of activation (same as the activation energy) for the rate controlling process, the free volume fraction present in a basic sliding unit and the average number of grain boundaries that align to form a plane interface during superplastic deformation, derived in earlier papers, are used to account for this phenomenon quantitatively in the present case also.

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1. Introduction

Nanocrystalline intermetallics are produced by different techniques [1–6]. The observed transition in material response from hardening to softening with decreasing grain size in the nanometer range (the so called “inverse Hall–Petch (IHP)” or “grain size softening effect”) in this class of materials at room temperature is an interesting phenomenon and appears to be independent of the processing route so long as contamination and significant changes in density due to processing are not present. In this paper, we have examined the phenomenon of inverse Hall–Petch effect at room temperature in some nanostructured intermetallics. Here, in view of the ambient temperature employed, grain size stability in the nanostructured intermetallics, after processing, is assured. In addition, so far as we could see, contamination and related issues were absent. Therefore, to the best of our judgment, the inverse Hall–Petch relationship observed in this class of materials is genuine. In our earlier works [7–13], optimal structural superplasticity was attributed to the onset of a mesoscopic (\sim a grain diameter or more) grain/interphase (in duplex or multi-phase materials) boundary sliding flow process. The usefulness of this

model was demonstrated earlier [8,10] for explaining the IHP effect in some metallic and nanostructured, quasi-crystalline materials. As stated above, the same model is used here to explain the IHP effect in intermetallics. While doing this, those aspects of the model, so far not emphasized, will be brought into focus. In addition, some new results on IHP in a nanocrystalline intermetallic, Al_5Fe_2 , obtained by us (unpublished) will be presented and shown to obey the same relationship. A full account of the mesoscopic grain boundary sliding controlled flow process and the validation procedures used are presented elsewhere [7–9,12,13]. For completeness, in the next section, a brief description of the mesoscopic grain/interphase boundary sliding controlled flow process is presented.

2. A brief description of a mesoscopic grain boundary sliding controlled flow model

It is well known that when deformation mechanisms operate in sequence, the slowest process will control the strain rate of deformation. In a coupled process like grain boundary sliding (GBS) – GB diffusion, which one of the two processes is regarded as rate controlling is a matter of choice on the part of the author [14,15]. The final aim is/should be to account for the experimental observations in as many systems as possible with the same minimum set of assumptions. In our model, it is assumed that during optimal superplastic flow, GBS, accompanied by diffusion in and/or

* Corresponding author.

E-mail addresses: kapse@uohyd.ernet.in, ananthaster@gmail.com (K.A. Padmanabhan).

dislocation emission from the sliding grain/interphase boundaries develops to a mesoscopic scale. (The grain interior is considered to be quasi-rigid, with practically no deformation present in this region, except for what is needed in the grain boundary neighborhoods, to ensure coherency of deformation). The former (GBS) is assumed to be the slower and hence the rate controlling process. The actual accommodation process (diffusion in, dislocation or partial dislocation emission from the sliding grain boundary regions) will depend on the nature and strength of the obstacles present in the grain boundaries and the experimental conditions like strain rate, grain size and temperature.

Several experiments have demonstrated that high-angle grain boundaries are very conducive to superplastic flow (for a summary, see Refs. [16,17]). With increasing strain, low-angle boundaries favorably oriented to the stress axis, will gradually be transformed into high-angle grain boundaries. Those that are unfavorably oriented will be bypassed by grain rotation, arising from the unbalanced shear stresses at the grain boundaries which slide at different rates that depend on the resolved shear stresses acting on these boundaries [18]. In other words, in this model superplastic deformation is rate controlled by mesoscopic grain/interphase boundary sliding along high-angle grain boundaries that are favorably oriented to the stress axis. The repeated observation that superplastic tendency is far less in materials with microstructures in which high-angle grain boundary fraction is rather low [19] vindicates this viewpoint. After a detailed survey of literature [13,20] it has also been concluded that solitons like vacancies, dislocations etc. get delocalized in high-angle boundaries and, therefore, it is not necessary –in fact, not meaningful– to invoke dislocation glide (based on crystallographic notions) along high-angle grain boundaries as a concomitant of the GBS process.

In this model, GBS is analyzed as a two-scale process: (a) atomistic, and (b) mesoscopic.

For mathematical development at the level of atomistics, the basic sliding unit present in the boundary is assumed to be an oblate spheroid of about 5 atomic diameters in the boundary plane and 2.5 atomic diameters in height (\approx average grain boundary width, see below) in a direction perpendicular to the boundary plane [7,9]. This way, it has been possible to explain superplastic flow in crystalline materials as well as bulk metallic glasses on a common basis [11]. This atomic ensemble is located symmetrically about the plane of the boundary with one half falling in each of the two grains that meet to form the boundary (Fig. 1 (a), (b)). The choice of shape (oblate spheroid) is due to the fact that the strain field inside a deformed oblate spheroid is uniform, with the help of which many useful engineering properties can be determined [21]. As has been pointed out, any deviation present in the real shape of the atom ensemble from the idealized (oblate spheroid) shape is taken care of by a form factor, α , which has a magnitude, ~ 1.0 [7]. The average shear strain associated with a unit sliding event (Fig. 1 (c) – (e)) is ~ 0.1 (arrived at based on bubble raft experiments and molecular dynamics simulations [22–25]), when the ensemble moves from one stable/metastable position to the next. It must be noted, however, that the actual shear strain associated with a unit boundary sliding event will be a function of the nature and strength of the interatomic forces present among the atoms that constitute the boundary. However, the bubble raft experiments reveal the order of magnitude value that could be associated with the unit shear strain (~ 0.1). (An examination of Fig. 1 (d), (e) reveals that after unit sliding shear strain, a circular dislocation loop (in the Volterra sense) of Burgers vector zero, will be left on the grain boundary plane, although crystallographic notions cannot be associated with such a deformation process (as the atom shuffling will involve only a very small fraction of the inter-atomic distance). Thus, a formal (not physical) case can be made for the propagation

of extrinsic dislocations along the boundary during the sliding process to reconcile this approach with that of Rybin, Nazarov and Valiev (for a summary, see Ref. [26]).

Based on an extensive analysis of experimental data concerning many metals, the grain boundary width is assumed to be 2–3 atomic diameters [25]. The elastic energy of the shear and momentary volumetric distortions (as the basic sliding unit is embedded inside a solid matrix) accompanying the unit shear event, ΔF_0 , constitutes the free energy of activation for the GBS process (arrived at by considering the deforming unit and the surroundings as a whole; same as the activation energy for the rate controlling process, often discussed in the literature, which only looks at the deforming unit [12]). Sliding at an individual grain/interphase boundary described above is rendered ineffective by steric hindrance, e.g. the presence of a triple junction.

For large scale sliding, the process has to develop to a mesoscopic scale (Fig. 2(a)) and form a plane interface (Fig. 2 (b)). When such plane interface formation is present simultaneously at different locations in a test specimen, inter-connection of such plane interfaces will result in large-scale boundary sliding and significant specimen elongation, i.e., superplasticity is observed. The length of the plane interfaces so formed is likely to be a strong function of grain size and temperature. It (the length of the plane interface) can be of the order of one or more grain diameters. (See later for a mathematical expression.)

During mesoscopic plane interface formation a portion of the energy supplied by the applied stress is spent in rearranging matter in the grain boundary region (Fig. 2 (b)). This gives rise to a long-range threshold stress, τ_0 , needed for the onset of mesoscopic boundary sliding. The driving force for plane interface formation is the minimization of the total free energy of the deforming system and the fact that for this configuration the work done by the external stress is a maximum, as required by Taylor's principle of maximum work [7,13]. (In Fig. 2 (c) it is very easy to show using Herring's equation for equilibrium at a triple junction that extending the boundary and increasing the magnitude of the included angle at the triple junction leads to a lowering of the total free energy of the system [9]). The long-range threshold stress necessary for plane interface formation is calculated assuming the grain shape to be rhombic dodecahedron (Fig. 2 (a)), the shape considered to be the closest to real crystals in polycrystalline materials [7,8]. As the grain boundary area per unit volume increases with a decrease in grain size, τ_0 increases with decreasing grain size (the number of boundaries aligning to form a plane interface increases with decreasing grain size). In contrast, as the inter-atomic bonds become weaker with increasing temperature, the rearrangement of matter in the boundary regions becomes easier and τ_0 decreases with increasing temperature. Experimental support for these two inferences is available [16,17].

A survey of literature reveals that this model has received considerable positive attention in the literature dealing with the deformation of nanocrystalline materials. This is because, in view of the very fine grain size, the grain boundary regions constitute a very significant fraction of the total volume of a material and thus, the idea of viewing the grain interior as a strong phase A and the grain boundary region as a weaker phase B is very much in vogue. Recently, the significance of this idea has been recognized beyond the domain of nanostructured materials. For example [27], have observed grain boundary sliding at room temperature in severe plastic deformed aluminum. They have suggested that the grain boundary region constitutes a weaker, high wetting component (component B) between high strength Al–Al grains (component A). In other words, the presence of thin GB layers is considered to serve as an inter-granular lubricant. Such GB layers can also form due to the so-called pseudo-incomplete GB wetting (see, for example [28],

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