



Full length article

# Coarsening kinetics of fine-scale microstructures in deformed materials



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## ABSTRACT

In this work we consider three representative continuous coarsening processes, namely subgrain growth in deformed subgrain structures, triple junction motion in deformed lamellar structures, and grain growth in deformed nanocrystalline structures, spanning a large range in structural scale and driving force. We propose a unified coarsening model, which is based on recovery kinetics and allows the apparent activation energy to change during coarsening. The model is successfully applied to the three coarsening processes in different materials of different structural morphology and scale, showing that the apparent activation energy increases during coarsening, which is verified by direct calculation. The increase in the apparent activation energy dominates the coarsening kinetics and leads to a significant decrease in the coarsening rate as coarsening proceeds. This suggests that a conventional grain growth model is not applicable in an analysis of coarsening of nanostructured materials. Our analysis also shows that an initial low thermal stability of nanostructured materials is inherently related to their large boundary area per unit volume and their high content of stored energy, providing a large driving force and, it appears, a low activation energy for structural coarsening.

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## 1. Introduction

In the design and development of strong metallic materials, plastic deformation to high and ultrahigh strains has been under rapid development in the last 20 years [1–6]. The materials owe their high strength to a very fine microstructure subdivided by boundaries on the submicrometer and nanometer scale. The energy stored in the structure is high, providing a large driving force for recovery and boundary migration [7]. These materials therefore lack thermal stability and may easily lose strength when applied or even during storage [8]. In design and development it is therefore mandatory to understand and model softening processes in these strong materials, and the present study focuses on thermal recovery preceding recrystallization.

Recovery kinetics in deformed metals has been explored extensively in the literature [9–14], and recently a thorough study of recovery in Al cold rolled to medium and high strains has been carried out [8,15–18]. These studies show that the apparent

activation energy increases continuously as recovery proceeds towards recrystallization [13,15,18]. Based on these studies, the current work widens the scope to model the general coarsening behavior of metallic materials after plastic deformation to medium-to-ultrahigh strains, where dislocations are preferentially stored in different types of boundaries, whose spacing varies on a length scale from a few micrometers to a few tens of nanometers.

When metallic materials of medium-to-high stacking fault energy are plastically deformed to low-to-medium strains, a cell-block structure typically forms, containing cell-blocks subdivided by cell boundaries [4,19]. At this strain range, the majority of deformation induced boundaries are low angle boundaries ( $<15^\circ$ ). During recovery, the boundaries become sharper and the structure may become coarser. This structure, especially after recovery to some extent, is conventionally known as a subgrain structure, where subgrain growth is an important recovery mechanism [12,20,21]. Deformation, for example by cold rolling, accumulative roll bonding and high pressure torsion, to medium-to-high strains typically leads to a finely spaced lamellar structure, containing extended lamellar boundaries parallel to each other and interconnecting dislocation boundaries in-between [4,17]. The lamellar boundaries are a combination of low angle and high angle boundaries ( $>15^\circ$ ) and the interconnecting boundaries are

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predominantly low angle boundaries. Further deformation to extreme strains, for example by ball milling, leads to an extreme grain refinement and an equiaxed deformed nanocrystalline structure, where high angle boundaries dominate. To cover a structural scale from the micrometer to the nanometer range, three representative well-characterized microstructures were chosen (Fig. 1).

- (i) A subgrain structure: Al-0.05 wt% Si single crystal channel die compressed by 70% at room temperature [22].
- (ii) A lamellar structure: commercial purity Al with a main chemical composition of 99.5Al-0.25Fe-0.15Si (wt%) deformed by cold rolling to a true strain of 5.5 [18,23];
- (iii) A nanocrystalline structure: high purity W powder ball milled at room temperature in Ar atmosphere [24].

## 2. Model derivation

The driving force for recovery and boundary migration results from the stored energy in the deformed materials. In our previous analysis of recovery kinetics, the remaining fraction of stored energy during annealing was determined by hardness indentation measurements [15,16]. In the present study, we estimate the stored energy by exclusively considering the energy stored in deformation induced boundaries since other contributions are comparatively small [25,26]. These boundaries, after initial recovery from a deformed state prior to any significant migration, have energies depending on their misorientation angles, and the boundary energy quickly approaches a constant with increasing misorientation angle according to the Read-Shockley equation [27]. During continuous coarsening of a deformation structure preceding recrystallization, the average misorientation angle typically changes marginally, and in the present work we take the average boundary energy as a constant during coarsening. Consequently, the stored energy ( $\text{J}/\text{m}^3$ ) can be simplified as [20,26]

$$P = \gamma S_V \quad (1)$$

where  $\gamma$  is the average boundary energy ( $\text{J}/\text{m}^2$ ) and  $S_V$  is the boundary area per unit volume ( $\text{m}^{-1}$ ), which can be expressed as

$$S_V = \frac{\alpha}{D} \quad (2)$$

where  $\alpha$  is a geometrical constant depending on the morphology

and  $D$  is a characteristic size parameter: (i) the subgrain size in a subgrain structure, (ii) the lamellar boundary spacing in a lamellar structure, and (iii) the grain size in a nanocrystalline structure. The morphology of a deformation structure may change slightly during continuous coarsening, but for simplicity we ignore this small change, i.e.  $\alpha$  is fixed. Therefore as a first approximation, the stored energy  $P$  is reciprocally related to the characteristic size parameter  $D$ .

In order to account for a near logarithmic dependence of recovery kinetics, Kuhlmann [9] first suggested an increasing apparent activation energy during recovery. Later, Borelius and coworkers [10] suggested a similar rate equation for recovery, considering the zero recovery rate at the end

$$\frac{dP}{dt} = -K_0 P \exp\left(-\frac{Q_0 - \beta P}{RT}\right) \quad (3)$$

where  $t$  is the annealing time,  $T$  is the annealing temperature,  $R$  is the gas constant,  $Q_0$  is the activation energy at the end of recovery, and the three fitting parameters  $K_0$ ,  $Q_0$  and  $\beta$  are associated with the operative recovery mechanisms. Eq. (3) may be recognized as a first-order reaction rate equation with an apparent activation energy depending linearly on the extent of recovery already occurred. The apparent activation energy is a key parameter in our study, and in the following the word *apparent* will be omitted for simplicity.

Combining Eqs. (1)–(3) gives the following expression

$$\frac{dD}{dt} = k_1 D \exp\left(\frac{k_2}{DT}\right) \quad (4)$$

where  $k_1$  is temperature dependent and  $k_2$  is a constant, written as

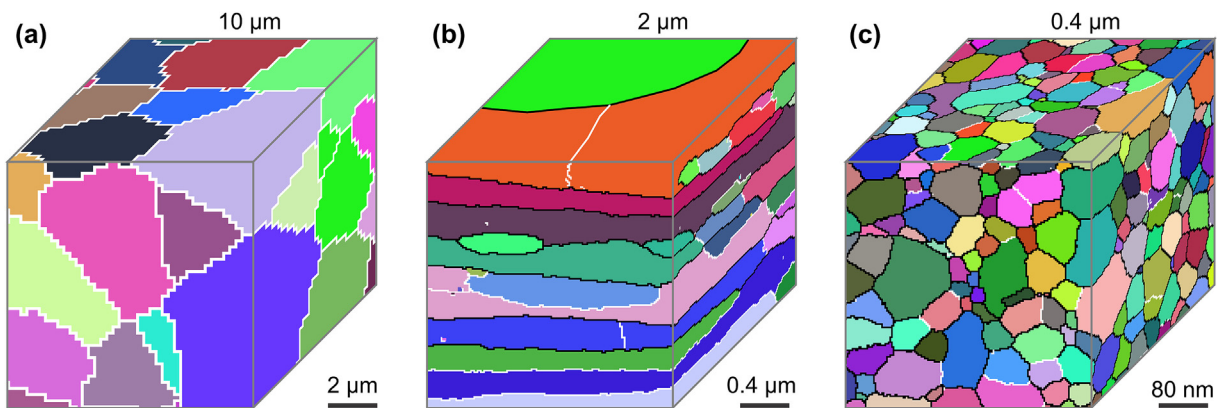
$$k_1 = K_0 \exp\left(-\frac{Q_0}{RT}\right) \quad (5)$$

$$k_2 = \frac{\alpha\beta\gamma}{R} \quad (6)$$

Eq. (4) can be solved with the aid of exponential integrals, resulting in the following relationship ( $D = D_0$  at  $t = 0$ )

$$Ei\left(-\frac{k_2}{DT}\right) - Ei\left(-\frac{k_2}{D_0T}\right) = -k_1 t \quad (7)$$

where  $Ei(\ )$  is the exponential integral of the quantity inside the bracket.



**Fig. 1.** Illustrations of three deformation structures of different length scales. (a) A subgrain structure; (b) a lamellar structure; (c) a nanocrystalline structure. White and black lines represent low and high angle boundaries, respectively. Different colors represent different grains/subgrains of different crystallographic orientations. The stored energy in the three structures spans almost two orders of magnitude. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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