

Cryomilled nanostructured materials: Processing and properties

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Received 8 February 2007; received in revised form 6 June 2007; accepted 6 June 2007

Abstract

Nanostructured (i.e., 1–200 nm grain size) and ultrafine-grained (i.e., 200–500 nm grain size) metals are of interest, not only as a result of their unusual combinations of physical and mechanical properties, but also because they can be readily synthesized using well-developed synthesis techniques. Cryomilling, i.e., mechanical alloying in liquid nitrogen, is representative of a class of synthesis techniques that attain the nanostructured state via severe plastic deformation. In this overview, published data related to cryomilled materials are reviewed and discussed with particular emphasis on cryomilling mechanisms; microstructure and thermal stability of cryomilled powders; primary consolidation and secondary processing methods; microstructural evolution during consolidation; and mechanical response of consolidated materials. The deformation behavior and the underlying mechanisms that govern cryomilled materials are discussed and compared with those of nanostructured materials processed via other methods, in an effort to shed light into the fundamental behavior of ultrafine-grained and nanostructured materials.

Published by Elsevier B.V.

Keywords: Cryomilling; Nanocrystalline; Strengthening mechanisms; Al alloys; Mechanical properties; Powder metallurgy

1. Introduction

Commercially significant quantities of nanostructured materials with a grain size in the range of 10–200 nm can be processed using mechanical alloying [1], which involves the transformation of plasticity-induced dislocation structures into high angle grain boundaries in metallic powders. The as-milled powder can be described as spherical agglomerates with an internal nanostructure. These 30–100 μm agglomerates can be readily consolidated via established powder metallurgical techniques, such as hot isostatic pressing, forging, or extrusion, to name a few. Interest in these materials stems from published studies which have reported that nanostructured materials consolidated from milled powder exhibit physical and mechanical properties that are superior to those of their conventional counterparts [2].

Cryomilling originally evolved as a variation of mechanical milling, and involves immersing the milling media in liquid nitrogen (approximately 77 K) and using processing parameters such that a nanostructured microstructure is attained [3–5]. Cryomilling takes advantage of the extremely low temperature of the liquid nitrogen medium, which suppresses the recov-

ery and recrystallization and leads to finer grain structures and more rapid grain refinement [1]. The processing and behavior of nanostructured materials via cryomilling have been discussed in a number of recent studies [6–9].

In this overview paper, our knowledge of the mechanisms that are responsible for the formation of a nanostructure during cryomilling is reviewed and consolidation techniques are discussed. More specifically, the present review addresses the following topics: cryomilling mechanisms; consolidation approaches; thermal stability; and mechanical behavior of consolidated materials. The mechanical behavior and the associated mechanisms of cryomilled Al alloys are discussed in an effort to shed light into the fundamental behavior of ultrafine-grained and nanostructured materials.

2. Nanostructure powders via cryomilling

The initial state of the material prior to milling is generally a powder, although flakes and other geometries can also be used. To understand the factors that influence the formation of a nanostructure during milling, one must consider two size scales. First, the overall deformation of the powder in the presence of the milling media is addressed. Second, the plasticity mechanisms that govern the formation of the nanostructure are discussed [10–13].

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Powder evolution during the milling process involves five stages, i.e., particle flattening as a result of plastic deformation, particle welding, equiaxed particle formation, random welding of powder particles and steady-state deformation, during which a balance between fracture and cold welding is established as microstructural refinement progresses. In view of the fact that severe plastic deformation during milling is a cyclic process, the milling time dominates the overall deformation strain. Hence, in order to obtain a microstructure with nanoscale grain sizes, adequate milling time is required.

Cooling of the milling media and powders is an effective approach to accelerate the fracture processes and rapidly attain steady-state conditions. There are several advantages to cryomilling as compared to milling at room temperature. First, powder agglomeration and welding to the milling media are suppressed, resulting in a more efficient milling outcome (e.g., improved yield and microstructure refinement). Second, oxidation reactions during milling are reduced under the protection of a nitrogen environment. Third, the milling time required to attain a nanostructure is significantly reduced, because the low temperature suppresses the annihilation of dislocations and the accumulation of a higher dislocation density is possible. As an example, a high density of dislocations of $1.7 \times 10^{17} \text{ m}^{-2}$ was observed in a cryomilled Al–Mg alloy [5,14], as shown in Fig. 1. In addition, many dislocations appear as dipoles, which have been reported in many heavily deformed metals. The dipoles are in fact dislocation loops with elongation along one direction so that they look like a pair of single dislocations with opposite Burgers vectors. The density of dislocations in cryomilled Al–Mg alloys is much higher than that in cold-rolled Al–Mg alloys, which is usually in the range of 10^{15} m^{-2} [5].

The grain refinement process during milling is accomplished via the formation of shear bands under localized deformation

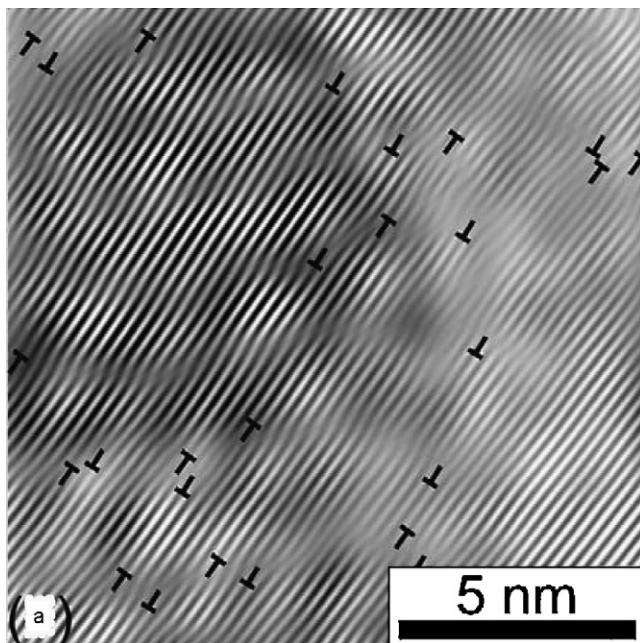


Fig. 1. Dislocations in a cryomilled Al–Mg alloy [5,14].

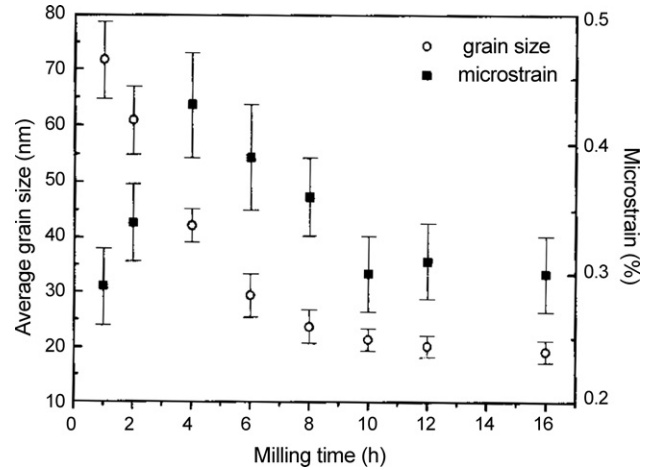


Fig. 2. The grain size and microstrain as a function of milling time in a cryomilled Al–7.5% Mg alloy [15].

and then the extension of shear bands to the entire sample [10]. The presence of elongated grains in milled powders is often associated with insufficient milling time. Therefore, it is not surprising to find microstructural features that manifest different relative degrees of deformation, and not simple lognormal distributions of randomly oriented equiaxed grains. With an increase of milling time, the grain size in a cryomilled Al–7.5% Mg alloy tends to decrease slowly to a saturated value, as shown in Fig. 2 [15].

Experimental and theoretical studies suggest that for each material, there is a minimum grain size that is obtainable by milling, and that its value is related to the intrinsic properties of the material, such as crystal structure [16]. Essentially, it has been proposed that the minimum grain size obtainable by milling scales inversely with melting temperature or bulk modulus. Mohamed recently developed a dislocation model to quantitatively describe the minimum grain size obtainable during milling [17]. According to this model, the minimum grain size is governed by a balance between the hardening rate introduced by dislocation generation and the recovery rate arising from dislocation annihilation and recombination. By balancing the rate of grain size decrease and the rate of grain size increase, the minimum grain size ($d_{\min}$) is given by [17]:

$$\frac{d_{\min}}{b} = A_3 \exp\left(\frac{-\beta Q}{4RT}\right) \left(\frac{D_{\text{PO}} G b^2}{v_0 k_B T}\right)^{0.25} \left(\frac{\gamma}{G b}\right)^{0.5} \left(\frac{G}{\sigma}\right)^{1.25} \quad (1)$$

where b is the magnitude of the Burgers vector, A_3 a dimensionless constant, β constant, Q the self-diffusion activation energy, R the gas constant, T the absolute temperature, D_{PO} the diffusion coefficient, G the shear modulus, v_0 the initial dislocation velocity, k_B Boltzmann's constant, γ the stacking fault energy and σ is the applied stress. The model predicts that the minimum grain size scales inversely with hardness, proportionally with the stacking fault energy and exponentially with the activation energy for recovery.

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