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ULTRAFINE GRAINED MATERIALS

Nanostructured materials by mechanical alloying: new results on property enhancement

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Abstract Mechanical attrition—the mechanical alloying or milling of powders—is a very versatile and potent method of obtaining nanocrystalline or ultrafine grain structures with enhanced properties. This article presents three examples of enhanced properties obtained by materials in which the grain size has been reduced to the nanoscale or ultrafine scale by ball milling and consolidation of powders. Very high strength/hardness—the highest hardness yet reported for crystalline Mg alloys—for a ball milled $Mg_{97}Y_2Zn_1$ alloy is due in part to the nanocrystalline grain structure, along with nanoscale precipitates. A ternary Cu-base alloy with a low stacking fault energy was found to have both high strength and good ductility in a nanocrystalline material synthesized by the in situ ball milling consolidation method. This is another example that shows nanocrystalline materials need not be brittle. It is shown that bulk thermoelectric materials with superior properties can be produced by the ball milling and consolidation of powders to provide an ultrafine grain structure.

Introduction

Mechanical attrition—ball milling of powders—is a technique that has been used widely for preparation of nanostructured materials [1, 2]. As a severe plastic deformation (SPD) method, it is very versatile in terms of the classes of materials that it can prepare in nanocrystalline form. Also, along with high pressure torsion and accumulative roll bonding, it is a technique that can regularly produce nanocrystalline grain sizes (<100 nm) and typically to the finest grain sizes of any SPD method (<20 nm). The term "mechanical attrition" can be subdivided into "mechanical milling" which is the milling of single composition powders, often elements, and "mechanical alloying" which involves milling of dissimilar powders such that material transfer occurs during milling. While mechanical alloying was first developed [3] to prepare oxide dispersion strengthened superalloys, since it was found to synthesize amorphous alloys [4] it has been used as a nonequilibrium processing method to prepare many classes of metastable materials including amorphous alloys, extended solid solutions, metastable crystalline compounds, quasicrystalline materials, and nanostructured materials [1]. This has become a popular method to fabricate nanocrystalline materials because of several factors: the simplicity of the process, the relatively inexpensive equipment (on the laboratory scale) needed, and the applicability to essentially all classes of materials. A major potential advantage is the possibility for easily scaling up to tonnage quantities of material for various applications. The disadvantages that are often cited (but can be addressed by proper processing techniques) are contamination from the milling media and/or atmosphere, and the need for many applications, to consolidate the powder product without coarsening the nanocrystalline microstructure.

As a consequence of the above-mentioned advantages, mechanical attrition has become one of the most popular methods for synthesis of nanocrystalline materials, and there is a large literature on the topic [1]. Since mechanical alloying can be used to alloy, as well as to provide a nanocrystalline microstructure, it can result in significant improvements in various properties. This article will focus

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on three recent results from our laboratory on property enhancement due to the formation of nanocrystalline microstructures obtained by mechanical alloying. Related results from the literature will also be cited as appropriate.

Nanocrystalline Mg alloy prepared by mechanical alloying

There has been increasing interest in Mg and its alloys as light-weight structural materials [5]. Mg has the lowest density of any of the structural metals, except for Be. However, it is hcp and suffers from relatively low strength, ductility, and formability. A number of studies in recent years have attempted to increase the strength and ductility of Mg-based alloys by alloying, grain refinement, addition of dispersoids, or changes in texture. With a nanocrystalline matrix, the yield strength of Mg can be increased from about 120 MPa for commercial extruded Mg to about 180 MPa for nanocrystalline Mg with a 45 nm grain size [6]. The yield strength of a Mg–5wt.%Al alloy is about 500 MPa in mechanically alloyed powders compacted, sintered, and hot-extruded, with about 100 nm grain size [7]. Interestingly, at longer milling times, which resulted in smaller grain sizes($\sim$45 nm), the strength dropped to about 200 MPa consistent with that for pure ball milled Mg [6]. Surface mechanical attrition treatment (SMAT) has been carried out on a Mg alloy (AZ91D, an 8.5%Al, 0.7%Zn alloy) [8]. A nanocrystalline surface region with average grain size of about 30 nm had a hardness value of about 1.8 GPa.

Inoue et al. [9] prepared a $Mg_{97}Y_2Zn_1$ alloy by the warm extrusion of rapidly solidified (atomized) powders. This material exhibited a yield strength of 610 MPa and tensile elongation of about 5%. The grain size of this material was 100–150 nm. Nanoprecipitates of $Mg_{24}Y_5$ of about 10 nm size were also observed. The Mg alloy had a novel long-period superlattice structure with a 6H-type, ABA-CAB-stacking. Abe et al. [10] studied this structure with atomic-resolution Z-contrast STEM. Two kinds of grains were observed in the microstructure. One kind had the hcp structure and with a composition of about Mg–1.5%Y. The other type had the fine lamellar superlattice structure and a composition of about Mg–2%Zn–4%Y. That is, the hcp-Mg grains do not contain any Zn and the lamellar grains are significantly enriched in both Zn and Y. The lamellae consist of nanoscale hcp Mg and long-period superlattice phase layers. An 18R type layered packing sequence was also found in this alloy prepared by induction melting and Cu-mold casting [11]. This phase transforms to a 14H type phase on annealing. These long-period superlattice phases, which apparently nucleate at the grain boundaries, have increased hardness over that of hcp Mg. In order to produce bulk $Mg_{97}Zn_1Y_2$, Chen et al. [12] used equal channel angular pressing (ECAP) of cast and machined ingots. It was suggested that this method would have the advantage of lower cost than the process of Inoue et al. However, they obtained a tensile yield strength of only about 400 MPa and poor ductility of about 2% elongation. The grain size was larger in these samples (about 330 nm) than in those of Inoue et al. [9], there were large second phase particles, and cracks in the second phase particles from the ECAP process.

The objective of our present research on this topic is to prepare nanocrystalline $Mg_{97}Y_2Zn_1$ by mechanical attrition of powders to allow for the formation of a grain size in the nanocrystalline regime and to determine how the structure and properties of this alloy will be affected by the nanocrystalline grain structure. Samples with a bulk composition of Mg–2at.%Y–1at.%Zn were prepared by ball milling the pure powders together in a SPEX 8000 shaker mill in a tool steel vial with martensitic stainless steel (440) balls. The ball-to-powder mass ratio was 10:1. The ball milling was carried out at room temperature under purified argon atmosphere (<1 ppm oxygen). The milled powder samples were characterized by X-ray diffraction using a Rigaku diffractometer with CuKα radiation. Transmission electron microscopy samples were prepared by a Fischione twin jet electropolisher on compacted powders. A JOEL-2000 transmission electron microscope operated at 200 kV was used to determine the grain size distribution of the milled alloy. Microhardness of the milled powder, compacted to full density at room temperature, was measured using a Buehler Micromet microhardness tester with a Vickers indenter at 50 g load and a loading time of 15 s.

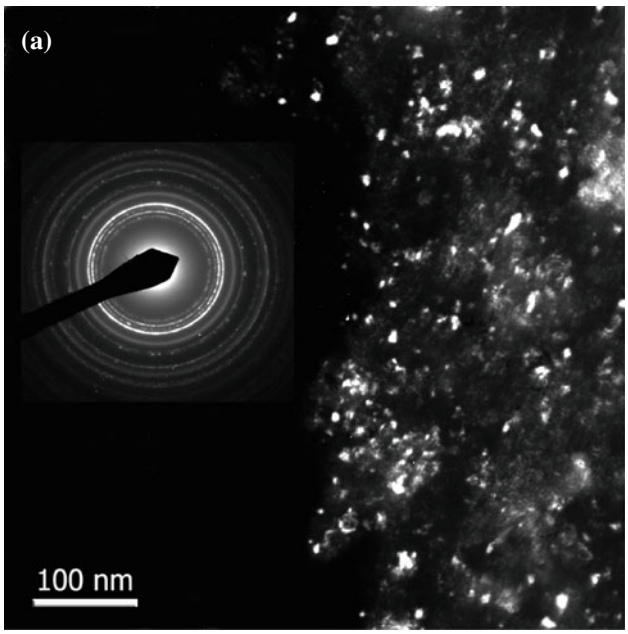
The preliminary milling of the $Mg_{97}Y_2Zn_1$ alloy resulted in agglomeration of the powder to the lower bottom side of the steel vial. This agglomeration causes inhomogeneous milling of agglomerated powders. Previous researchers encountered the same problem during milling of Mg and Mg alloys. Adding a process control agent (PCA)-surfactant- is one way to minimize powder agglomeration [13]. The PCA is usually added to prevent or minimize excessive cold welding of powder particles among themselves and/or to the milling container and the grinding medium. However, the use of the PCA can lead to contamination of the powder and also can be detrimental to subsequent powder consolidation through formation of a thin surface coating around the powders [14]. Hwang and McCormick [6] modified the SPEX milling operation by incorporating additional rotational and sliding movements to the original motion of the vial in order to minimize the powder adhesion while avoiding the use of a PCA. In our case, we realized that the agglomeration takes place after about 20 min from the start of milling. A small portion of the powder adheres to the lower part of the vial and with

further milling time, more powder accumulates and results in excessive agglomeration. In order to resolve the agglomeration problem without adding a PCA or modifying the rotation of the SPEX mill, the milling process was interrupted after every 15 min and the vial was rotated 90° and then the milling was continued. This simple procedure yielded over 90% of the initial mass of the alloy powders in loose form with no significant agglomeration or welding of the powders to the balls or vial.

X-ray diffraction patterns were determined as a function of milling time on the milled powders. The peaks for the pure Zn and Y disappeared after 1 h of milling and only peaks for hcp Mg were observed. Close examination of the XRD patterns reveals that new peaks appeared after 5 h of milling. Some of these peaks could be identified as belonging to the $Mg_{24}Y_5$ structure, consistent with the previous observations on this alloy after rapid solidification and extrusion, from electron diffraction measurements [9, 10]. The grain size of the $Mg_{24}Y_5$ phase calculated from XRD line broadening in our alloys was about 12 nm.

The milling process also causes the XRD peaks to broaden and decreases their intensities due to refinement of the grain size and the introduction of lattice strain. The integral breadth analysis was used to calculate the grain size and lattice microstrain from the XRD line broadening [15]. The analysis presumes that the grain size broadening and strain broadening profiles can be approximated by Cauchy and Gaussian functions, respectively. The average crystallite size and the rms lattice strain estimated by this method for our alloy milled for 8 h was calculated to be 27 nm and 0.24%, respectively. Figure 1a shows a dark-field TEM micrograph and the corresponding electron diffraction pattern, see inset, of the alloy mechanically milled for 8 h. The grains appear to be equiaxed and randomly distributed within the structure. The grain size distribution based on measurement of 210 grain diameters on dark field images showed a mean grain size of 21 nm and a monotonic grain size distribution as shown in Fig. 1b. This grain size is comparable to that (27 nm) estimated from the XRD line broadening calculations.

The variation in microhardness as a function of milling time is shown in Fig. 2. The hardness increases with milling time, more gradually at the longer times. After 8 h of milling the hardness reaches a value of 2.13 GPa. This is the highest value reported for this alloy, with references 9 and 12 reporting hardness (or strength $\times 3$) as about 1.8 and 1.4 GPa, respectively. Our hardness for the $Mg_{97}Y_2Zn_1$ alloy is comparable to the high hardness values for more highly alloyed Mg-based alloys which form bulk metallic glasses and are then extruded and crystallized to nanocrystalline structures with intermetallic precipitates [16]. Thus, mechanical alloying of the $Mg_{97}Y_2Zn_1$ alloy appears to be a viable approach to synthesizing this potentially useful material. Further studies of the structure and properties of this material are being conducted in the authors' laboratory.



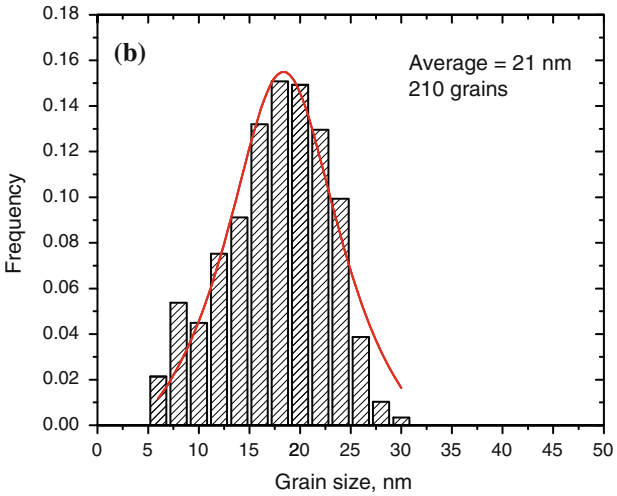


Fig. 1 Dark-field TEM micrograph (**a**) and grain size distribution (**b**) for $Mg_{97}Y_2Zn_1$ milled for 8 h

Mechanical properties and structure of in situ consolidated nanocrystalline alloys

A problem with using "two-step" processes such as the mechanical attrition of powders and their consolidation for synthesis of nanocrystalline alloys is the need for successfully consolidating the powders to complete theoretical density and bonding. Typical methods for doing this involve both pressure and temperature and the latter

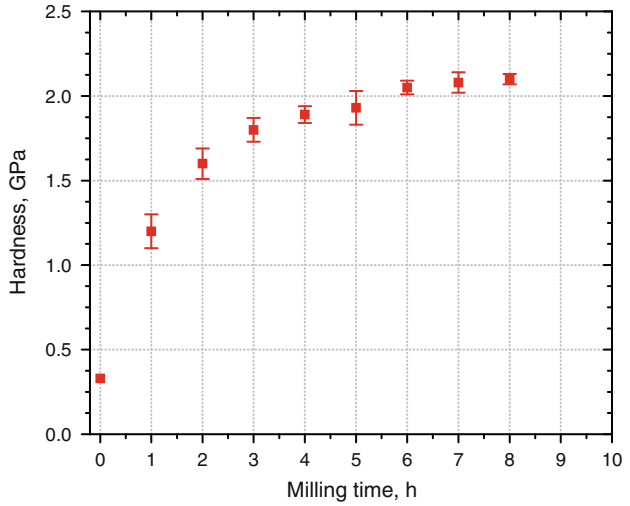


Fig. 2 Hardness as a function of milling time for $Mg_{97}Y_2Zn_1$

necessarily leads to coarsening of the nanocrystalline microstructure. This topic has been reviewed, for example, by Groza [17] and Mayo [18]. These reviews document research on consolidation of powders by both conventional (e.g., hot pressing, hot isostatic pressing, extrusion, etc.) and innovative methods (e.g., microwave sintering, plasma activated sintering, etc.). Even when theoretical densities have been obtained, mechanical tests in tension often reveal brittle behavior that appears to be due to incomplete particulate bonding rather than intrinsic mechanical behavior [19]. The in situ consolidation ball milling method to be described below, with recent results using this method, is one method to provide artifact-free samples for the measurement of intrinsic mechanical behavior of nanocrystalline materials.

The first example of in situ consolidated ball milled nanocrystalline alloys was in Zn [20]. We had found that milling Zn at 77 K produced powder with a fine nanocrystalline grain size (~30 nm average diameter), but was difficult to consolidate to complete density and bonding by hot pressing. Conversely, we found that by milling at room temperature, spherical balls of Zn, 5–10 mm in diameter were formed, but had grain sizes in the submicron range, that is, typically several hundreds of nanometers. It was found that a combination of first milling at 77 K followed by room temperature milling was effective in providing artifact-free samples for subsequent mechanical testing while still maintaining a nanocrystalline microstructure. The room temperature milling appears to sharpen the broad grain size distribution obtained for the cryomilling times used into a narrower peak by removing the largest and smallest grains from the distribution. This was attributed to enhanced grain growth of the finest grains and grain size reduction by recovery processes in the larger grains—both due to the higher thermal activation available at room temperature. The in situ consolidated ball milled Zn, with nanocrystalline microstructures, in the form of spherical balls, could be pressed into disks. Dog-bone shaped tensile samples were prepared from these disks for tensile tests, and the measurement of good ductility showed that the samples were indeed artifact-free and providing inherent properties.

Along with an investigation of the in situ consolidation ball milling of Zn, Al and several Al alloys were also studied [21]. A sphere of Al–3%Mg was prepared which had a diameter of 8 mm. When uniaxially compressed at room temperature into a disk of about 11 mm diameter, the Al–3%Mg alloy had a density greater than 98% of theoretical as measured by the Archimedes principle method. However, these samples were all milled at room temperature and found to contain voids in the spheres. While pressing to disks may close these voids, in order to eliminate any questions regarding artifacts for mechanical property measurements, subsequent studies combined milling at 77 K with milling at room temperature to obtain porosity-free spheres. A bulk nanocrystalline Al–5%Mg alloy was synthesized by the in situ consolidation ball milling method [22]. The ball milling was first carried out at 77 K in order to produce a nanocrystalline grain structure and the appropriate flake-like morphology which subsequent ball milling at room temperature can form into dense, porosity-free spheres. The milling was carried out in a Spex 8000 shaker mill with the ball-to-powder mass ratio of 10:1. The solid spheres formed by the combination of cryomilling and room temperature milling were pressed into disks about 1 mm in thickness and 10–12 mm in diameter. Characterization studies were made on these disks including XRD and TEM. The XRD results indicated that a metastable solid solution of Mg in Al had been formed. No diffraction peaks for extra phases were observed, and precise lattice parameter measurements were consistent with the formation of a solid solution of Mg in Al. X-ray diffraction line broadening analysis gave an average grain size of 32 nm which was in reasonable agreement with the results of dark-field TEM measurement which gave a mean grain size of 26 nm. The disks were cut into dog-bone shaped tensile specimens with a gauge length of 2 mm and a width of 1 mm. The tensile samples were polished to a mirror finish to eliminate the effect of surface flaws. The final thicknesses of the tensile samples were in the range of 460–600 μm. Tensile tests in a miniature tensile machine at room temperature resulted in the stress–strain curves as illustrated in Fig. 3 (curve C) in comparison with annealed coarse grain Al-5083 alloy, and Al-5083 alloys made by the cryomilling of powder and consolidation by hot isostatic pressing (HIP) and extrusion at 523 K [23] (curve B) or consolidated by cold isostatic pressing (CIP) and extrusion [24] (curve D). It is clear that

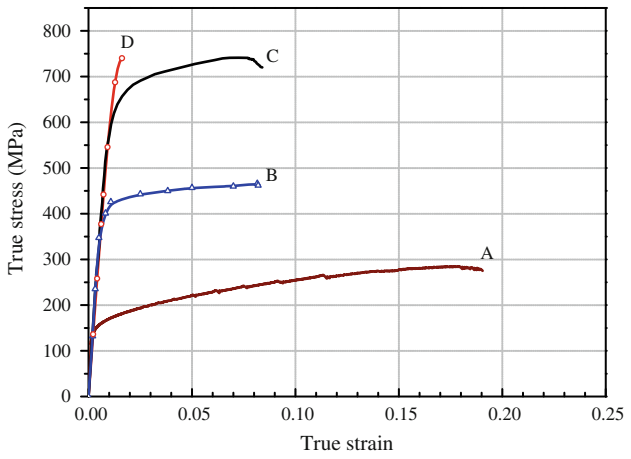


Fig. 3 Typical tensile stress–strain curves for tested samples [22]: curve A, annealed, coarse-grained (55 μm) Al-5083 alloy; curve B [23], Al-5083 alloy (bimodal grain size, nc +μm) produced through cryo-milling and consolidation by HIP and extrusion at 523 K; curve C, bulk in situ consolidated nc (26 nm) Al–5% Mg alloy; curve D[24], nc (30 nm) Al-5083 alloy produced through cryo-milling and consolidation by CIP and extrusion

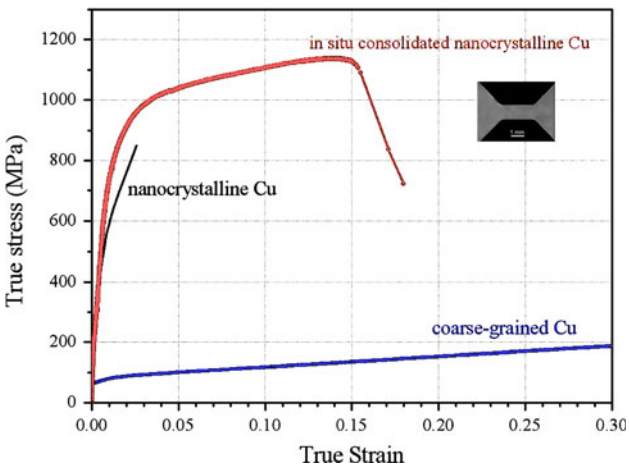


Fig. 4 A typical tensile stress–strain curve for the bulk in situ consolidated Cu sample in comparison with that of a coarse-grained polycrystalline Cu sample (an average grain size larger than 80 μm) and a nanocrystalline Cu sample prepared by an inert-gas condensation and compaction technique [from ref. 26]

the nanocrystalline sample made by the in situ consolidation ball milling method has the best combination of strength and ductility. This sample had more than 4 times the yield strength of the commercial Al–Mg alloy, Al-5083, along with good ductility of 8.5% elongation in tension. The observed ductility is consistent with the substantial strain hardening observed which implies that the deformation is controlled by creation and interactions of dislocations.

The in situ consolidation ball milling method was used to prepare bulk nanocrystalline Cu that could be used for mechanical testing [25, 26]. The Cu powders were first milled at 77 K for 3 h. The starting Cu powders were flattened and welded together to form thin small rounded flakes about 1 mm in diameter. Further combinations of milling at room temperature and 77 K up to 10 h produced fully dense and spherical–shaped balls with sizes up to 5–8 mm diameter. Density measurements and SEM observations of sectioned spheres indicated that they were free from any porosity. The grain sizes of these Cu spheres as determined by both XRD and dark-field TEM were about 23 nm with a fairly narrow grain size distribution such that no grains had diameters >50 nm. The Cu spheres were pressed into disks from which several types of mechanical tests could be performed. Disk samples were prepared for microhardness measurements and the miniaturized disk bend test (MDBT) [25]. The hardness of the nanocrystalline Cu samples was measured to be 2.3 GPa. The yield strength determined from the MDBT data was 770 MPa. This is about 10 times higher than the yield strength for conventional grain size Cu and is consistent with the hardness value and the approximate Tabor relation of hardness = 3× (yield strength). Along with the high values of strength and hardness, the samples exhibited significant ductility in the MDBT. While quantitative ductility values are not possible, the disks showed substantial ductility by deforming into large "hat" shaped disk morphology. Subsequently, tensile tests were carried out on the nanocrystalline Cu made by the in situ consolidation ball milling method [26]. Dog-bone shaped tensile specimens were machined from the nanocrystalline Cu disks which had gauge lengths of 2 mm, widths of 1 mm, and thickness of about 0.5 mm. The stress–strain curve for this nanocrystalline Cu is shown in Fig. 4 in comparison with data for the best nanocrystalline Cu made by the inert-gas condensation and compaction method [27] and conventional grain size Cu. The nanocrystalline Cu made by in situ consolidation ball milling shows both very high strength along with good ductility.

To try to understand the high strength and good ductility in this nanocrystalline Cu, in situ TEM tensile testing was carried out [26]. Dislocation activity was observed in grains as small as 20 nm. Dislocation pile-ups were observed as well as individual dislocations which remained after the stress was removed. These results suggest that dislocation plasticity is responsible for the mechanical behavior.

Cheng et al. [28] also used in situ consolidation ball milling to prepare nanocrystalline Cu. The processing variables were somewhat different than those used in [25, 26], and the results were therefore also different. The vial temperature at nominally liquid nitrogen temperature was between 93 and 113 K, which is a little higher

temperature than used in [25, 26]. This may be critical since the large dense spheres were not obtained but large plates, the largest piece usually found under the vial cover. The best results in this study were for milling times at liquid nitrogen temperature for 3 h followed by 5–6 h milling at room temperature. The plates so formed were then cold rolled at room temperature by a thickness reduction of 50–100%. This procedure produced samples for tensile tests with 3 mm gauge length and width in the 1–2 mm range and thicknesses from 150 to 300 μm. The grain sizes of these materials had a broad distribution with an average of about 40–60 nm and largest grains about 150 nm. The tensile yield strengths were about 600–700 MPa and elongation to failure of about 6%. The as-milled samples contained some porosity, flaws, and were very brittle. After cold rolling, the flaws were apparently eliminated, at least in part, such that the ductility in tension (6% elongation) was observed.

The differences in the results of references [26] and [28] illustrate how critical the processing variables are for attaining artifact-free nanocrystalline materials with a narrow grain size distribution by the in situ consolidation ball milling method. In turn, this points to our lack of a detailed understanding of the mechanism for this process. Aiken and Courtney [29] modeled the particle size evolution during ball milling. Their model was framed in the context of the balance between particle fracture and welding. They derived probabilities for fracture and welding, and for Cu these were 0.0332 and 0.06, respectively. This would suggest that welding would dominate and Cu particles would grow with milling time. Milling Cu at room temperature in an attritor mill indeed showed an initial increase in average particle size with some particles in the 1–2 mm range. However, after 8 h of milling the average size began to decrease. The authors suggest this is due to work-hardening which would increase the fracture probability with respect to the welding probability. Clearly, more modeling combined with experimental studies are needed if the mechanism(s) responsible for in situ consolidation ball milling are to be understood. This in turn could allow for a wider variety of materials to be prepared in this manner.

Our recent work on the deformation behavior of nanocrystalline materials includes a study of low stacking fault energy materials. An example of these is a Cu–12at.%Al–4at.%Zn alloy which has a stacking fault energy of about 7 mJ/m^2 [30]. We have been able to produce a nanocrystalline microstructure in bulk, artifact-free samples of this alloy by the in situ consolidation method. X-ray diffraction of this alloy showed only an fcc structure. Transmission electron microscopy revealed a nanocrystalline structure with an average grain size of about 22 nm. More detailed high resolution TEM showed extensive faulting. This work

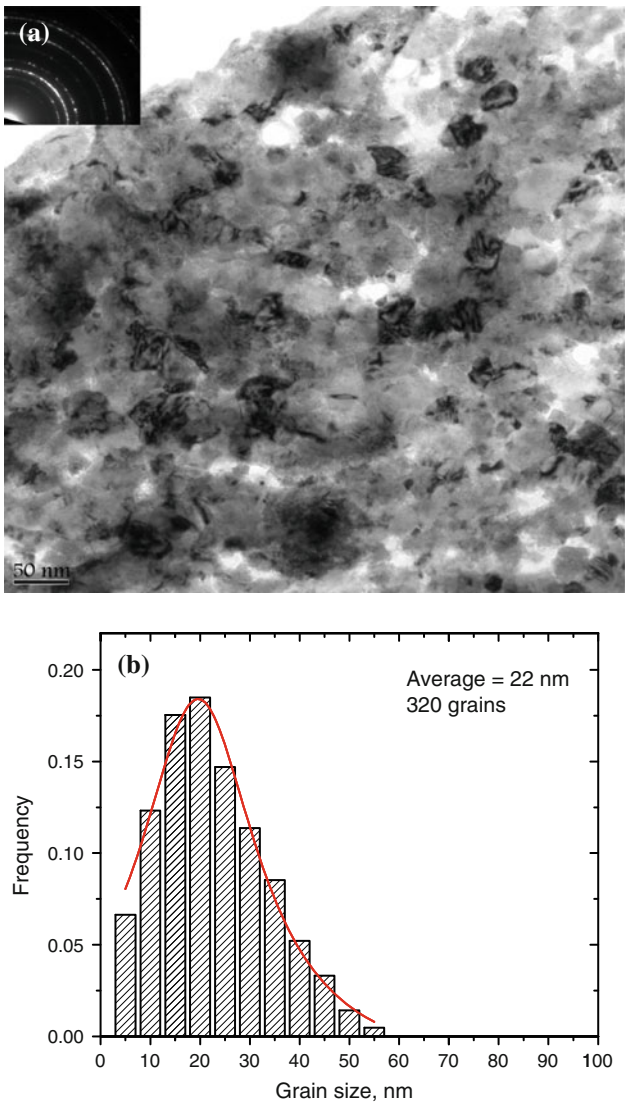


Fig. 5 TEM micrograph (**a**) and grain size distribution (**b**) for nanocrystalline Cu–12at.%Al–at.% Zn

will be described elsewhere. The TEM micrograph and the grain size distribution are given in Fig. 5a, b, respectively. The spherical balls that resulted from the in situ powder consolidation were pressed into disks. Dog-bone shaped tensile specimens were milled from these disks and tested in our miniature tensile testing machine. The resulting stress–strain curve is shown in Fig. 6. This material has excellent mechanical properties for a Cu-based nanocrystalline material with a fine grain size. While the yield strength of 1067 MPa and ultimate strength of 1198 MPa might be expected for nanocrystalline Cu alloys with ~20 nm grain size, the good ductility of about 10% elongation in tension is unusual. This may be due to the artifact-free structure, and the microstructure which allows for significant strain hardening.

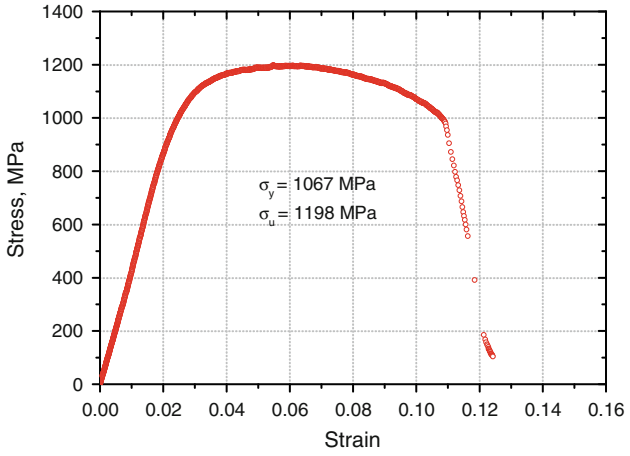


Fig. 6 Stress–strain curve for nanocrystalline Cu–12at.%Al–4at.%Zn with a 22-nm grain size

Enhanced thermoelectric properties by ball milling

While most of our research has been directed toward the mechanical behavior of nanocrystalline materials, nanocrystalline grain size can have major influence on many other properties such as magnetic, electrical, corrosion resistance. Here we describe the beneficial effects of nanocrystalline microstructures on thermoelectric materials prepared by ball milling of powders.

As part of the increasing interest in energy savings, thermoelectric devices have received attention for their potential applications in waste heat recovery, air-conditioning, and refrigeration. The efficiency of thermoelectric devices is determined by the material dimensionless figure of merit, ZT, defined as, $ZT = (S^2\sigma/\kappa)T$, where S, σ, κ, and T are the Seebeck coefficient, electrical conductivity, thermal conductivity, and absolute temperature, respectively [31]. A number of approaches have been used to try to improve ZT but the maximum ZT of the important commercial materials based upon Bi_2Te_3 and its alloys, such as $Bi_xSb_{2-x}Te_3$ (*p*-type) and $Bi_2Te_{3-x}Se_x$ (*n*-type), has remained at about 1 for the *p*-type material, and less than 1, typically about 0.9 for the *n*-type material. A strategy that has been proposed by several groups [32, 33] is to reduce the dimensionality of the material by introduction of nanoscale constituents that would allow for quantum-confinement effects to enhance the power factor, $S^2\sigma$, and at the same time the large interfacial area in nanostructures would be designed so that the thermal conductivity would be reduced more than the electrical conductivity due to the differences in their respective scattering lengths. A recent breakthrough in increasing ZT was reported by Poudel et al. [34]. They prepared the *p*-type BiSbTe material by ball milling the bulk ingots into powder which was determined to be nanoscale. The powders were consolidated by direct-current hot pressing. The bulk consolidated material had a range of grain sizes from the nanoscale up to about 1 μm and contained "nanodots" which were Sb rich, and some nanoscale Te precipitates. This material exhibited a ZT value at room temperature of 1.2 and a peak value of 1.4 at 100 °C. At room temperature the power factor of their material was similar to that of a state-of-the-art ingot of the same composition, but the thermal conductivity was lower, thus providing the enhanced values of ZT.

We have recently prepared nanocrystalline $Bi_2Te_{2.7}Se_{0.3}$ by ball milling the elemental powders at either 77 K or room temperature. The as-milled powders had fine nanocrystalline grain sizes as determined from X-ray diffraction line broadening of between 10 and 20 nm. The powders were consolidated by hot pressing under pressure of 2 GPa at temperatures ranging from 200 to 410 °C, the latter near the melting point of the material. The density of the compacts increased with increasing compaction temperature and the electrical resistivity, presumably reflecting the decrease in porosity, also decreased with increasing temperature. Thus the best thermoelectric properties were measured on the samples compacted at 410 °C. The room temperature value of ZT determined was about 1.7, about double the best existing value for this *n*-type material. The microstructure of the samples with the optimum ZT values was determined by TEM. Thinning for TEM was carried out by focused ion beam machining. The average grain size of this sample was found to be about 400 nm. More detailed studies of the microstructure and continued processing to obtain a finer grain size are in progress in the authors' laboratory and will be reported in due course.

Summary

Mechanical attrition—the mechanical alloying or milling of powders—is a very versatile and potent method of obtaining nanocrystalline or ultrafine grain structures with enhanced properties. It is a severe plastic deformation technique that can provide the finest nanoscale grain sizes. Compaction to bulk material remains a challenge. This article presented three examples of enhanced properties obtained by materials in which the grain size has been reduced to the nanoscale by ball milling of powders. Very high strength/hardness—the highest hardness yet reported—for a Mg-based alloy is due in part to the nanocrystalline grain structure, along with nanoscale precipitates. A ternary Cu-base alloy with a low stacking fault energy was found to have both high strength and good ductility in a nanocrystalline material synthesized by the in situ ball milling consolidation method. This is another example that shows nanocrystalline materials need not be brittle. Finally, it is shown that bulk thermoelectric materials with superior

properties can be produced by the ball milling and consolidation of powders to provide an ultrafine grain structure.

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