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Rapid synthesis of an extra hard metal matrix nanocomposite at ambient temperature

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ABSTRACT

The strengthening of metals is essentially controlled by the microstructures of the metal solids and it is well understood that smaller grain sizes lead to higher hardness and increased strength. Nevertheless, true bulk nanostructured materials are difficult to produce using established engineering techniques, especially when considering the practical and societal needs of materials selection. Lightweight Al and Mg are conventional metals having excellent physico-chemical and mechanical properties and with good strength/weight ratios in the finished products. However, the fabrication of high-strength metals consisting of these elements, using mechanical alloying and milling and cladding-type metal working, generally involves long-term processing conducted under extreme conditions using special facilities. The present study demonstrates the very rapid synthesis of a metal matrix nanocomposite (MMNC) of the Al–Mg system which was achieved by stacking metal disks of the two pure metals and processing by high-pressure torsion at ambient temperature for 10 turns. An exceptionally high hardness was achieved, similar to many steels, through rapid stress-induced diffusion of Mg and the simultaneous formation of intermetallic nano-layers and a nanostructured intermetallic compound with a supersaturated solid solution. This unexpected result suggests a potential for simply and expeditiously fabricating a wide range of MMNCs.

1. Introduction

The synthesis of new materials is now driven by technological issues combined with the restrictions imposed by ecological considerations in a variety of industrial applications [1]. The lightweight metals of aluminum and magnesium are widely used for structural applications in the automotive, aerospace and electronic industries but improvements in the mechanical properties of these metals would be attractive for enhancing their future use. In practice, a recent study reported an increase in the strength limit of an aerospace-grade Al-7075 alloy by high-pressure torsion (HPT) processing while maintaining reasonable formability [2]. Nevertheless, it is reasonable to anticipate that there is probably an upper limit on the enhancement in mechanical properties that may be achieved when the processing is conducted directly on the alloy. This suggests the potential for achieving superior properties by bonding dissimilar metals and synthesizing new metal systems. In practice, however, Mg alloys generally have less weldability due to their high Zn content and conventional methods for joining metals are often not practical for magnesium-based alloys.

Mechanical alloying and mechanical milling [3] are widely used to process the promising Al–Mg alloy system but these methods require precisely measured concentrations of powders to produce a system in a metastable state and then several procedural steps in order to make semi-final solid products. The bonding of dissimilar metals is an alternative approach for directly producing the bulk Al–Mg alloy system and this includes fusion welding [4] and compound casting [5] requiring metals in a liquid state or roll cladding/bonding of a solid-state welding [6–9]. In all of these methods, the metals are diffusion-bonded under a relatively limited pressure at an elevated temperature for a long period of time so that it is not feasible to

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produce an exceptionally fine microstructure as required for applying Hall–Petch (HP) strengthening [10–12].

Bulk nanostructured materials (BNM) having true nanometer grains can be fabricated by severe plastic deformation (SPD) which is a promising technique for achieving grain refinement in bulk metals [13]. A growing interest in the field of nanostructured materials has arisen in the last two decades due to reports of unusual properties in nanostructured materials containing ultrafine grains with extremely large fractions of grain boundaries [14]. In the SPD process of accumulative roll bonding (ARB), two sheets of the same material are stacked, heated, rolled for bonding, and then the processed sheet is cut in half, the surfaces are degreased and wire brushed and the two bonded sheets are stacked again and the process is repeated [15]. Using the principles of ARB, sets of dissimilar metals with Al have been processed to make multi-layered microstructures: for example, in the Al–Cu [16], Al–Mg [17–19] and Al–Zn [20] systems. However, inspection of the processed fine-grained metal plates after ARB revealed anisotropic plastic behavior, including strength and ductility, which was dependent upon the rolling direction and the through-thickness direction [21].

Among the various available SPD techniques, high-pressure torsion (HPT) provides the potential for achieving true nanostructures [22]. In this procedure, a disk is strained under a high compressive pressure with concurrent torsional straining and the processing is usually conducted at room temperature (RT) which is effective even for difficult-to-deform materials such as Mg alloys [23]. Also, the processed metals generally demonstrate an enhancement of the physical and mechanical characteristics through significant grain refinement and the intensive introduction of point and line defects [24].

The essential principle in HPT processing is that the strain introduced within the HPT disk sample is markedly inhomogeneous. Specifically, when a disk is strained by torsion in HPT, the shear strain, γ, is given by a relationship of the form [25]

$$\gamma = \frac{2\pi N r}{h} \quad (1)$$

where N is the number of HPT revolutions and r and h are the radius and height (or thickness) of the disk, respectively. Therefore, it is apparent that the torsional straining imposed within the disk sample is dependent upon the distance from the center of the disk and at the center where $r=0$ it follows from Eq. (1) that the strain is theoretically zero. This implies that there is an inevitable inhomogeneity both in the microstructure and in the hardness in disk samples processed by HPT. Nevertheless, it was demonstrated experimentally that high numbers of HPT revolutions and high applied pressures are both effective in producing reasonably homogeneous microstructures and homogeneous values for the microhardness throughout the disks [26–28]. Because of the introduction of intense plastic straining during processing, HPT has been applied also for the bonding of machining chips [29] and the consolidation of metallic powders [30–32]. Nevertheless, these processes require a high processing temperature [32] and a two-step process is needed for cold/hot compaction prior to consolidation by HPT [31].

To date, there is only a single practical demonstration of a solid-state reaction in an Al–Cu system through the bonding of semi-circular half-disks of Al and Cu using HPT at ambient temperature for up to 100 turns [33]. However, a recent report demonstrated at least the potential for forming a spiral-textured Al–Cu hybrid material though HPT where four quarter-disks, including two of pure Cu and two of an Al-6061 alloy, were positioned to make a complete disk and then processed by HPT at room temperature for 1 turn [34]. A limitation of this latter work was that it described only the computational calculation of the distribution of equivalent stress in the processed disk using a finite element method and there was no detailed microstructural analysis of the processed disk.

Accordingly, the present research was undertaken specifically to explore the alternative possibility of achieving direct diffusion bonding of Al and Mg disks through HPT processing. The specific aim was to synthesize an Al–Mg system as a multi-layered BNM through the bonding of separate Al and Mg disks using HPT and then to undertake a detailed examination of the deformed structure. As will be demonstrated, the results provide a direct confirmation of the potential for using HPT for the formation of an intermetallic-based metal matrix nanocomposite (MMNC) through diffusion bonding at room temperature.

2. Experimental materials and procedures

The experiments were conducted using two materials: a commercial purity (99.5%) aluminum Al-1050 alloy containing 0.40 wt% Fe and 0.25 wt% Si as major impurities with <0.07 wt% Zn and <0.05 wt% of Cu and Mg as minor impurities and a commercial ZK60 magnesium alloy containing 6.03 wt% Zn and 0.72 wt% Zr with <0.01 wt% of Fe, Si, Cu and Mn as minor impurities. Both alloys were received as extruded bars having diameters of ~10 mm. These bars were cut into billets with lengths of ~65 mm, a series of disks was sliced from the billets with thicknesses of ~1.2 mm and the disks were polished to final thicknesses of ~0.83 mm.

Conventional HPT processing was conducted at room temperature under quasi-constrained conditions [35,36] following the general processing procedure described earlier [37] except that the pressing involved more than one disk. Thus, separate disks of the Al and Mg alloys were placed in the depression on the lower anvil in the order of Al/Mg/Al where the Mg disk was positioned between the two Al disks but without using any glue or metal brushing treatment. Three separate piles of three disks in the order of Al/Mg/Al were processed through HPT under an applied pressure, P, of 6.0 GPa at RT for total numbers of revolutions, N, of 1, 5 and 10 turns using a constant rotation speed of 1 rpm. The configuration of the disks for HPT processing is illustrated in Fig. 1, where (a) shows the piled disk samples on the lower anvil of the HPT facility before processing and (b) is a schematic illustration of the experimental set-up.

Following processing, each disk was cut vertically along the diameter to give two semi-circular disks. One vertical cross-section from each disk was polished, chemically etched using Keller's etchant and examined by optical microscopy. Subsequently, values of the Vickers microhardness, Hv, were recorded over the vertical cross-sections of each disk using a Shimadzu HMV-2 facility with a load of 50 gf. These measurements recorded the individual microhardness values following a rectilinear grid pattern with an incremental spacing of 0.2 mm in both the horizontal and vertical directions. The microhardness data were then used to construct color-coded contour maps displaying the hardness distributions within each disk.

The horizontal disk surfaces of the semi-circular disks processed by HPT for 5 and 10 turns were polished mechanically and then finished by vibratory polishing. An X-ray diffraction (XRD) analysis was performed using a high-resolution XRD system, Rigaku Ultima III, in selected edge regions on the polished surfaces of the disks processed through 5 and 10 turns. The examination used a Cu Kα radiation with a scanning speed of 1° min^{-1} and a step interval of 0.01°. Microstructural cell parameters and phase percentages were quantified by means of the XRD data analysis software, Materials Analysis Using Diffraction (MAUD), which is based on a full pattern fitting procedure (the Rietveld method).

Detailed microstructural analyses by transmission electron microscopy (TEM) were conducted at the edges of semi-circular disks after HPT through 5 and 10 turns using a spherical aberration