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Cryogenic friction-stir processing of ultrafine-grained Al–Mg–TiO_2 nanocomposites

F. Khodabakhshi [a], A.P. Gerlich [b], A. Simchi [a,c,*], A.H. Kokabi [a]

[a] Department of Materials Science and Engineering, Sharif University of Technology, P.O. Box 11365-9466, Azadi Avenue, 14588 Tehran, Iran
[b] Department of Mechanical and Mechatronics Engineering, University of Waterloo, Waterloo, ON, Canada
[c] Institute for Nanoscience and Nanotechnology, Sharif University of Technology, P.O. Box 11365-9466, Azadi Avenue, 14588 Tehran, Iran

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ABSTRACT

Submerged friction-stir processing under cryogenic conditions was employed to fabricate ultrafine-grained nanocomposites with enhanced mechanical characteristics. Al–Mg alloy sheet with 3 vol% TiO_2 nanoparticles were processed under air (ambient temperature), a water-dry ice medium (~ -25 °C), and liquid nitrogen. It is shown that a homogenous distribution of reinforcement particles throughout the metal matrix is attained at a rotational speed of 1400 rpm and a traverse velocity of 50 mm/min after 4 passes. *In situ* formation of Al_3Ti and MgO nanophases during multi-pass processing is shown by transmission electron microscopy. Under the cryogenic cooling condition, ultrafine grains and cellular structures with sizes smaller than 1 μm and 200 nm are attained. It is shown that the formation of an ultrafine-grained structure is accompanied with significant improvement (150–200%) in the mechanical strength. The tensile yield strength of $\sim$170 MPa, elongation of $\sim$22% and Vickers hardness of $\sim$165 HV are attained. A change in the fracture mode from ductile-brittle to fully ductile is presented when the cryogenic processing is employed. A relationship between the grain size and the fracture features is demonstrated. Effects of cooling conditions on the microstructure and mechanical properties of friction stir processed Al-based nanocomposites are addressed.

1. Introduction

Ultrafine-grained (UFG) materials have attracted great attention for structural applications due to their improved mechanical and physical properties [1–3]. Submicron grain structures (typically <300 nm [4]) in these materials can provide a unique combination of strength and ductility with possible superplastic behavior in some alloys [5–10]. Various techniques have been developed to process UFG materials. In a “bottom-up” approach, bulk specimens are fabricated from nanoscale building blocks through inert gas condensation [11], electro-deposition [12], and chemical and physical deposition [13,14]. Another way to prepare UFG solids is to use coarse-grained materials and induce substantial grain refinement by severe plastic deformation (SPD) [2,15]. The main SPD methods includes equal-channel angular pressing (ECAP) [16], high-pressure torsion (HPT) [17], accumulative roll-bonding (ARB) [18], constrained groove pressing (CGP) [19], and submerged friction-stir processing (FSP) [20]. By employing these processes, various bulk UFG materials for structural applications have been prepared [21].

Recently, friction stir processing (FSP) of UFG metals, particularly Al alloys, have gained significant attention [6,22–25]. This interest mainly stems from the wide range of potential applications in the transport and aerospace industries as structural parts [26]. In this process, thermal exposure and intense plastic deformation with a strain rate of 10^0–10^3 s^{-1} and a strain of up to $\sim$40 breakup coarse secondary phases and induce severe material mixing with accelerated diffusion of elements [27]. The processed materials usually contain equiaxed recrystallized grains with relatively low dislocation densities and high fraction of high-angle grain boundaries (90%) [28]. The UFG structures render improved strength and wear resistance without a significant reduction of ductility [29,30]. Meanwhile, studies [31–33] have shown that during conventional FSP, UFG structure is difficult to achieve, since dynamic recrystallization mechanisms caused by large plastic straining and adiabatic frictional heating limit the grain refinement to 2–3 μm. To promote an UFG structure, forced cooling during the FSP technique has been proven quite effective. Several researchers have reported that the grain structure can severely be refined by applying in-process or external cooling during FSP [34–41]. For instance, investigations on 2xxx [42,43], 5xxx [44,45], 6xxx [36,46] and 7xxx [37,40] aluminum alloys have

* Corresponding author at: Department of Materials Science and Engineering, Sharif University of Technology, P.O. Box 11365-9466, Azadi Avenue, 14588 Tehran, Iran. Tel.: +98 21 6616 5261; fax: +98 21 6600 5717.
E-mail address: simchi@sharif.edu (A. Simchi).

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revealed that an UFG structure in stir zone is attained by forced cooling. Earlier work by Benavides et al. [34] showed a significant reduction in stir zone temperature for an Al–Cu alloy. The effect of cooling rate on the mechanical properties of friction stir welded AA7075-T7351 sheets has been shown by Nelson et al. [35]. Fu et al. [37] studied effects of submerging conditions utilizing cold and hot water on the mechanical properties of friction stir welded 7050 aluminum alloy. In the later case, it was peculiar that higher tensile strength and elongation values were obtained by employing hot water; however this is due to the fact that precipitate dissolution and reprecipitation dominated the properties, and not formation of an UFG structure. Liu et al. [38] reported an improved strength for friction stir welded AA2219 sheets under water compared to that of air-processed sheets. The microstructure and mechanical properties of submerged friction stir welded AA2219-T6 was investigated by Zhang et al. [39]. The results showed that the tensile strength and grain structure were sensitive to the processing parameters, especially the rotational speed. Effects of thermal boundaries on friction stir welded AA7050-T7 sheets were studied by Upadhyay et al. [40]. By using water and a liquid with subzero temperature ($-25\,^{\circ}$C), they showed hardness enhancement in the weld nugget compared to air cooling. Hofmann et al. [41] demonstrated the positive effect of submerged FSP on the grain refinement of AA6061-T6 sheets, in which grain sizes could be reduced to the 200–300 nm range.

The aim of this study is to develop a novel procedure to prepare aluminum matrix nanocomposites with UFG structure by employing submerged FSP. AA5052 aluminum alloy was utilized because this aluminum–magnesium alloy is very promising for structural applications in aerospace, military, and transportation industries due to their light weight and high corrosion resistance [47–49]. In order to improve the performance of the Al–Mg alloy with regard to the specific strength and wear resistance, cryogenic FSP was employed. It is shown that an UFG grain structure is developed and nanometric hard inclusions are formed upon processing. Consequently, the mechanical strength of the Al–Mg alloy is improved remarkably. A key point in this work is to use TiO_2 nanoparticles with an aim to form titanium aluminde (Al_3Ti) phase, which has low density, high Young's modulus and desirable mechanical properties at both ambient and elevated temperatures [50,51]. Although nanometric Ti particles could also be used instead of TiO_2, high activity of Ti nanoparticles to oxygen would be restrictive. Previous works on FSP of Al–Ti and Al–TiO_2 billets prepared by powder metallurgy [52–55] and friction stir processing [50,51,56–60] have shown that Al_3Ti and/or Al_2O_3 phases are formed by deformation assisted solid state chemical reactions. To the best knowledge of the authors, no report can be found in open literature on *in situ* and simultaneous composite formation and grain refinement to ultrafine range (<200 nm) for Al alloys containing Mg. It has very recently been shown that [61,62] by employing multi-pass FSP on an Al–Mg alloy with pre-placed TiO_2 nanoparticles, *in situ* Al_3Ti and MgO nanophases are synthesized while the grain structure is refined to a fine range (2–3 μm). A remarkable improvement in the hardness and tensile strength is achieved, although the ductility is deteriorated. As an extension of the previous work, submerged FSP is employed in the present work in order to refine the grain structure to an ultrafine range (<200 nm). The prepared UFG nanocomposites not only exhibit enhanced mechanical properties but also retain moderate ductility. It is worthy to mention that the advancement of this work

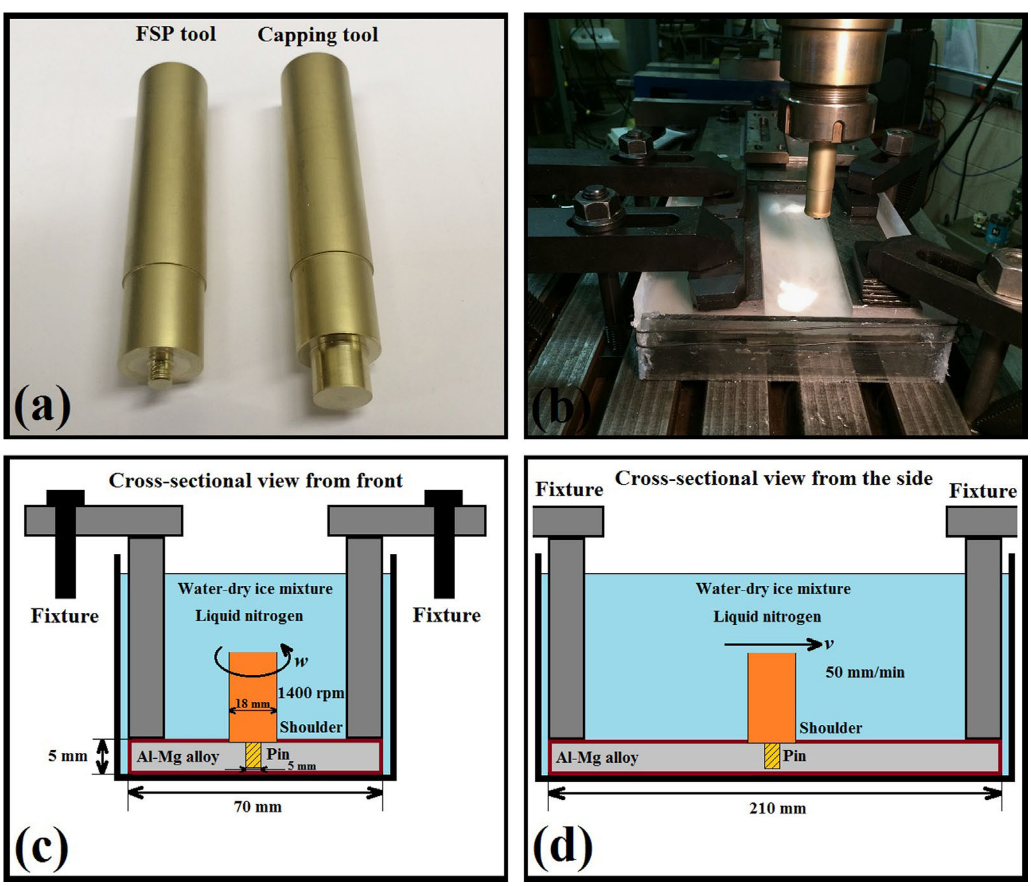

Fig. 1. (a) Tools used for capping and friction stir processing. (b) Accomplishment of submerged FSP in the lab. (c,d) Schematic design of submerged FSP.