Materials Characterization 144 (2018) 440–447

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Effects of in-process cryocooling on metallurgical and mechanical properties of friction stir processed Al7075 alloy

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ARTICLE INFO

Keywords:
Age-hardenable alloy
Electron microscopy
Mechanical characterization
In-process cryocooling
EBSD

ABSTRACT

Friction stir processing (FSP) of heat treatable aluminum alloys is reported to deteriorate strength and hardness properties in spite of huge grain size refinement. The improvement in strength due to the grain size refinement after FSP could not be able to offset the reduction in strength due to coarsening or dissolution of the strengthening precipitate(s). To overcome this, the present work investigates an effective way of heat rejection from the FSP zone to control the precipitate evolution besides the grain size refinement during FSP of Al7075 alloy. The FSP was carried out with standardized process parameters (i.e. 720 rpm, 65 mm/min traverse speed) under two different cooling environments: (i) at normal air cooling and (ii) by rushing a chilled mixture (−30 °C) of liquid nitrogen and methanol through the bottom of the backing plate. Microstructural evolution was examined through optical, scanning and transmission electron microscopy and electron backscatter diffraction to confirm the grain size and morphology of precipitate(s) formed. The in-process cryocooling during the FSP led to 65 and 72% improvement in the Vickers hardness and tensile strength, respectively, in comparison to the solutionized base metal. The results have been analyzed in terms of precipitate interface characteristics and its strengthening effect, Hall–Petch strengthening due to grain size reduction and tradeoff between them.

1. Introduction

Age-hardenable Al 7XXX series alloys are extensively used in structural components (airframe, sporting goods and transportable bridges) railway transport systems, defense equipments and automobile sectors because of its outstanding properties such as high excellent strength to weight ratio, high stiffness and good corrosion resistant [1,2]. Moreover, the performance and efficiency of these alloys are required to enhance continually to fulfill the socioeconomic requirements and challenges. Making of an ultrafine grained (UFG) structure in the bulk alloys is an effective way to further enhance the various mechanical properties of these Al alloys [3,4]. Over the past few years, various severe plastic deformation (SPD) techniques such as cryorolling [5], multiaxial forging [6], cross accumulative roll bonding [7], cyclic extrusion compression [8], equal channel angular pressing [9] etc. have been applied to produce UFG materials. All of these studies are reported to use successfully for preparation of UFG structure in Al, Cu and Mg alloys. However, most of these SPD methods are comparatively complex and have some major limitations like expensive tooling, design difficulties, low productivity and limitation of sample size produced. These factors make most of the SPD methods challenging to scale up for commercialization.

In contrast, friction stir processing (FSP) is a single step process; while other SPD techniques require mostly multiple steps/cycles to develop fine grained structure [10,11]. FSP uses a simple inexpensive tooling; and a readily available machine such as a milling machine can be used to conduct the process. Other advantages of FSP are that it could be automated for production and a simple fixture to control the cooling rate can be designed. All these features make FSP easier and less expensive as compared to other SPD methods. Hence, it is possible to upgrade the FSP method from laboratory scale to the commercialized level. FSP is similar in principle to that of friction stir welding. FSP causes intense plastic deformation and frictional heating during the process which leads to generation of recrystallized grain structure in the nugget zone (NZ) of the processed sample [12,13]. Recovery and recrystallization occurring during FSP is mainly due to high gradient of strain, strain rate and temperature of the NZ near to the pin. Al and its alloys are effectively processed by the FSP to enhance its mechanical properties [11,14,15]. Kwon et al. [14] conducted FSP on Al1050 alloy and reported a significant enhancement in hardness (37%) and tensile strength (46%) of the processed material as compared to the starting material. They concluded that the FSP is a very promising technique for

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https://doi.org/10.1016/j.matchar.2018.08.001
Received 11 April 2018; Received in revised form 1 August 2018; Accepted 1 August 2018
Available online 02 August 2018
1044-5803/

enhancing the mechanical properties resulting from grain refinement. During FSP, degree of grain refinement is a strong function of process parameters and recrystallization kinetics of the material to be processed. Several researchers have reported that the effective optimization of the processing parameters can control the grain size and consequently the properties of the friction stir processed (FSPed) material [16–19]. However, in case of precipitation hardenable alloys, a competition between strengthening due to grain refinement and softening due to change in precipitate characteristics by FSP is observed [17,20]. Many researchers [13,17,21,22] have studied the precipitates formed after processing and concluded that, an adverse event of precipitate dissolution or coarsening during the FSP would be the major reason for showing inferior properties, despite the development of fine grained microstructure. In addition, Xu et al. [23] have found that fine grains evolved during FSP grow very quickly due to slow cooling rate after FSP. Aforementioned issues associated with the FSP hinder the real purpose of FSP of enhancing the mechanical properties. To overcome these constraints, it is necessary to use an effective way of heat rejection from the processing zone, which can control the precipitate kinetics besides the grain size refinement by controlling the thermal boundaries of the material during FSP of age hardenable alloys.

FSP with active/in-process cooling setup suppresses precipitate coarsening/overaging and arrests the grain growth by reducing the peak temperature, increasing cooling rate and limiting holding time. This tactic can combine the precipitation strengthening and grain size strengthening along with other strengthening mechanisms in age hardenable Al alloys. In recent years, various researchers have applied means of external cooling effect (in-process and submerged) during FSP/FSW and they concluded that external cooling during FSP is an effective way to enhance the metallurgical characteristics and thereby mechanical properties of the processed materials. For example, Fratini et al. [24] used a different cooling medium (water, forced air, and free air) flowing on the surface of the specimen (AA7075 T6) during FSW and reported that the tensile strength of the joint was superior in the sample processed in water cooling medium. In order to take full benefit of water cooling, Tokisue et al. [25] conducted submerged FSW on Al6061 alloy and concluded that it is practicable to achieve sufficient torque for welding during underwater FSW. Hofmann and Vecchio also [26] carried out submerged (underwater) FSP on Al6061-T6 alloy. They found that submerged FSP could attain more grain size refinement due to faster cooling rate compared to those of samples FSPed under ambient conditions. Upadhyay and Reynolds [27] investigated the influence of submerged FSW on the weld properties of Al7050-T7451 joints welded in air, underwater and under sub-ambient liquid medium of −25 °C temperature. They reported that there is an increase in the torque required (power consumption/machine capability) for the submerged/sub-ambient welding conditions though tensile strength of the FSPed samples is improved. In order to obtain rapid heat sink during FSP, Chang et al. [28] designed an efficient cooling system and concluded that UFG structure with a drastic increase in the microhardness (increment factor of 2.4) could be achieved in Mg-Al-Zn alloy by FSP attached with a rapid heat sink.

In recent years, FSP with external/in-process cooling has attracted significant attention although the studies are still limited as compared to normal FSP at room temperature. Furthermore, investigation on the influence of in-process cryocooling during FSP of the Al7075 alloy is rarely reported. Hence, the present work aims to investigate the effect of in-process cryocooling during FSP on the microstructural evolution and mechanical properties of the solutionized Al7075 alloy. Normal FSP (at room temperature) was also carried out for comparison of the in-process cryocooling FSP results. Detailed analysis of mechanical properties (such as tensile strength and hardness) and microstructural features (grain size and precipitate characteristics) was carried out to establish the effectiveness of the in-process cryocooling and mechanisms of microstructural evolution during FSP of Al7075 alloy.

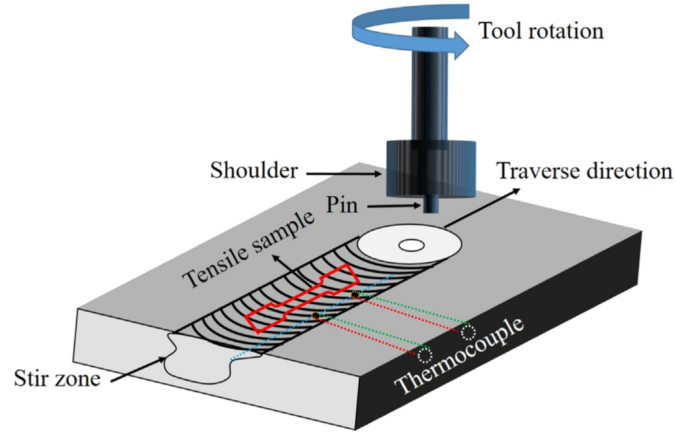

Fig. 1. Schematic of FSP.

2. Experimental

Al7075 alloy plate with a thickness of 6 mm was procured from M/s. Hindalco Industries, India, in T651 condition. Chemical composition (in wt%) of the material is as follows: 5.93% Zn, 2.26% Mg, 1.43% Cu, 0.23% Cr, 0.18% Si, 0.18% Fe, and balance is Al. The FSP tool used for the present experiments was designed to have a concave shoulder (diameter = 25 mm) and cylindrical pin (diameter = 6 mm & length = 4 mm) (Fig. 1). The FSP experiments for the present study were carried out only using a rotational speed of 720 rpm and at a traverse speed of 65 mm/min with a constant tilt angle of 1.5° to the vertical axis. These parameters were optimized in our previous studies [17] where the FSP experiments were conducted at room temperature. Several combinations of traverse speeds (25, 45, 65, 85 and 100 mm/min) and rotatory speeds (508 and 720 rpm) were used to obtain defect free processed zones. For both the conditions, i.e. in-process cryocooling (IPC) and normal air cooling (NAC), the optimized results in terms of defect free processed zone with highly refined microstructures were obtained only for the traverse speed of 65 mm/min at an rpm of 720. Rectangular plates of dimension 100 mm × 30 mm × 6 mm were cut from the procured plate and then solutionized at 480 °C for 6 h followed by water quenching. FSP of solution treated samples was carried out at two different conditions: (i) NAC and (ii) IPC. For IPC during FSP, an indigenously designed and fabricated fixture was used (Fig. 2). The fixture has the configuration of a hollow rectangular chamber below the top surface of the backing plate. The backing plate of the fixture was made of copper because of its high thermal conductivity. A chilled mixture (−30 °C) of liquid nitrogen and methanol ($LN_2 + CH_3OH$) was used to flow continuously through the hollow rectangular chamber of the backing plate during FSP. Temperature profile of the NZ during FSP was measured using the K-type thermocouple (marked in Fig. 1). The FSP parameters were kept constant for both NAC and IPC conditions for better comparison of the effects of two different cooling rates on microstructural evolution.

For metallographic characterization, samples were sliced from cross section perpendicular to the FSP direction. The specimens for optical and scanning electron microscopy (SEM) were polished with different grades of emery papers and then polished with MgO suspension on a velvet cloth. Modified Keller's reagent was used to etch the mirror polished surface of the samples for metallographic observation. For electron backscattered diffraction (EBSD) studies, the samples were electropolished in a solution of 20% perchloric acid +80% methanol for 90 s at −30 °C and at a potential difference of 12 V. The EBSD scan was conducted on FEI-Quanta 200FE-SEM with a step size of 0.2 μm. Further the scan was analyzed by using TSL OIM software. In addition, FEI Technai 20 G2S-Twin HR transmission electron microscope operated at 200 kV was used to characterize the microstructural details in