Microstructural and Mechanical Evaluations of Al/AlN Nano-Composite Surface Layer Produced via Friction Stir Processing

Maryam Samiee, Abbas Honarbakhsh-Raouf and Seyed Farshid Kashani Bozorg

Department of Materials Engineering, Semnan University, Semnan, Iran.
School of Metallurgy and Materials Engineering, University of Tehran, Tehran, Iran.

Abstract. Al/AlN nano-composite surface layer was fabricated on 6061 aluminum alloy substrate employing friction stir processing technique. To achieve this, nano-sized AlN powder was inserted into a pre-made groove on the substrate. Optical micrographs showed the presence of a fine dynamically recrystallized grain structure in the stirred zones of the Al/AlN nano-composites produced by FSP. Scanning electron microscope examinations indicated that by adjusting the process parameters such as increase in rotation speed, a uniform distribution of nano-sized AlN particles in the substrate Al can be obtained. The produced nano-composite surface layer presented enhanced hardness more than two times of as-received substrate Al. From micro-hardness measurements of the surface nano-composites, it can be inferred that the hardness of Al/AlN nano-composites enhanced as rotation speed increased.

Key words: Nano-composite, SEM, hardness, Friction Stir Processing, Al, AlN

INTRODUCTION

High physical and mechanical properties of Al/AlN composites such as high thermal conductivity and low coefficient of thermal expansion make them proper candidates in the microelectronic industry (Abdoli, 2010; Sreeja Kumari, 2011; Goujon, 2001; Goujon, 2003; Liu, 2009). As well as, AlN doesn’t react with Al in the interface (Abdoli, 2010; Sreeja Kumari, 2011; Goujon, 2001; Goujon, 2003; Liu, 2009; Elangovan, 2009) even in molten state, similar to SiC (Sreeja Kumari, 2011), and has interfacial adhesion with Al (Abdoli, 2010; Sreeja Kumari, 2011; Liu, 2009). Thus, the problems like interfacial reactions and poor interface that observed remarkably in Al/Al2O3 or Al/SiC composites, don’t exist in Al/AlN composites. AlN is more wettable to aluminum as compared to Al2O3 (Sreeja Kumari, 2011).

Aluminum alloy 6061 is a heat treatable wrought Al–Mg–Si alloys that was used as substrate in this study. Aluminum alloy 6061 has moderate strength and high welding characteristics. Therefore, this alloy is widely applied in the industry such as aircraft, pipelines, marine frames and storage tanks (Elangovan, 2009).

It is hard to disperse nano-sized reinforcement particles on metallic matrix to produce nano-composite layer by conventional methods such as powder metallurgy (Zahmatkesh, 2010), as these methods suffer from particles agglomerations.

Friction stir processing (FSP) is a severe plastic deformation approach to obtain fine grains, almost defect-free structures. This employed similar approach as friction stir welding (FSW), where a rotating tool is inserted into a fixed plate to apply high frictional heating and intensive plastic deformation to the materials to made a strong metallurgical joining (Hofmann, 2005; Woo, 2006; Woo, 2006). FSP, is used as a method for modifications of microstructures of surface like grain refinements and homogenization of structures, in recent years (Woo, 2006; Woo, 2006).

Hence, we stimulated to adapt FSP technique, for the first time, to produce a heat treatable aluminum matrix composite reinforced with nano-sized AlN powder.

MATERIALS AND METHODS

Friction stir processing was performed on 10mm thick rolled plates of commercial 6061 aluminum alloy as substrate, its chemical composition is presented in table 1. The rolled plates were cut into the required size in a rectangular shape, 50 mm in width and 100 mm in length. Nano-sized AlN powder with an mean diameter of ~50nm and a 99.9% purity were employed as particulate reinforcement.

Table 1: Chemical composition (wt%) of the base metal.

<table>
<thead>
<tr>
<th></th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Cr</th>
<th>Mn</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>1.05</td>
<td>0.63</td>
<td>0.41</td>
<td>0.32</td>
<td>0.05</td>
<td>0.07</td>
<td>0.12</td>
<td>0.01</td>
<td>Base</td>
</tr>
</tbody>
</table>

A non-consumable rotating tool of high carbon steel (hardened H-13 tool steel) was machined. Shoulder tool was 16 mm in diameter. Fixed pin tool was 5 mm in diameter and about 4 mm in length. A 3° tilt angle of
the fixed pin tool was used. The workpiece plates were held tightly by a fixture and ambient air cooling after each pass was used. To introduce the nano-sized AlN powders, one groove about 3 mm in depth and 1 mm in width was machined, in which the nano-sized AlN powder could be filled to the desired amount before FSP. To prevent the AlN powder from being displaced out of the groove(s), AlN was condensed with a modified FSP tool that had a shoulder and no pin. To achieve the optimum conditions for processing of AA 6061 plates, two passes of FSP were carried out at fixed advancing speed of 310 mm/min and rotation speed of 900, 1120 and 1800 rpm, respectively. The samples prepared in different FSP conditions are noted as Fx. Here, x represents the rotation speed (rpm). For an instance, F_{1800} represents the sample produced after two passes FSP at constant travel speed of 310 mm/min and rotation speed of 1800 rpm.

One of the samples produced in this method, is presented in Fig.1a. Several specimens were sectioned from the processed workpieces (Fig.1b) and machined to the required dimensions to prepare microstructural specimens. The samples were then polished using different grades of abrasive papers. Final polishing was done with the diamond paste (~1µm size) in the disc polishing machine. Specimens were etched employing HF 0.5% reagent to reveal the structure.

![Fig. 1: (a) Upper surface of Al/AlN nano-composite surface layer of the F_{1800} and (b) the cross section of the area shown in the part (a).](image)

The grain structures of the etched samples were evaluated by OM, and particle distributions of unetched specimens were observed by SEM using energy dispersive spectrometry (EDS). The Vickers hardness profiles of the specimens were measured on the traverse sections, parallel to the direction of the processing, and also on the surfaces of the specimens, perpendicular to direction of the processing, transferring the stirred zone using a Vickers indenter with a 200 gf load for 10 s.

**RESULTS AND DISCUSSION**

Optical microscopy examinations were done on the cross section of the F_{1800} (Fig.2). Transition from the stirred zone-thermo-mechanically affected zone-heat affected zone microstructure, is shown in Fig.2 a. The base metal microstructure is characterized by large elongated grains, typical of a hot rolled structure (Fig.2b). The optical micrographs indicate that the stirred zone consists of fine, uniform and equiaxed grains (Fig.2c), formed during dynamic recrystallization. The serious plastic deformation and high temperature have led to significant smaller grains compared to the base metal. The stirred zone is surrounded by the thermo-mechanically affected zone (TMAZ) (Fig.2d) and by a small heat affected zone (HAZ) (Fig.2e). Since recrystallization doesn’t occur in this region owing to low temperature, the grains of the TMAZ, are larger and less equiaxed than the stirred zone. The HAZ isn’t affected by mechanical effects and possesses similar grain structure to the base metal (Cavaliere, 2006; Hatamleh, 2007; Cavaliere, 2006).

To reveal the nano-sized AlN powder distributions in AA 6061 matrix of Al/AlN nano-composite surface layers, the surfaces of the F_{900}, F_{1120} and F_{1800} were subjected to SEM examinations, respectively, as shown in fig.3. It can be observed that decrease in rotation speed resulted in agglomeration of nano-sized AlN powder in the stirred zone. A uniform nano-sized AlN powder distributions and less nano-sized AlN powder agglomerations in the stirred zone, have arisen from the higher rotation speed (Faraji, 2010; Barmouz, 2010). From Figs.3c and 2c, it can be inferred that the presence of the nano-sized AlN powder has led to the grain refinement in the stirred zone.
Fig. 2: Cross section of the F1800: (a) the stirred zone, the TMAZ and the base metal interface, (b) the base metal, (c) the stirred zone, (d) the TMAZ and (d) the HAZ.

Fig. 4 presents the micro-hardness distribution of the Al/AlN nano-composites and Al specimens away from the centre of the stirred zone, measured vertical to the FSP direction on the surface of the F900, F1120 and F1800. As it can be seen, the hardness curves are not perfectly symmetrical, it can be explained that the plastic flow field was not uniform in the two sides of the stirred zone, regarding to the centerline of the stirred zone. This behavior can be associated to the more intensive piling of materials on the advancing side compared to the retreating side. The larger distorted grains led to higher strain-hardening (Xu, 2009; Brown, 2009; Xu, 2009). It can be seen in this fig that the hardness values in the stirred zone of the F1800, reached to 93 HV, about 2.5 times more than 36 HV, the base metal hardness value. As it can be observed obviously in fig.4, increase in the rotation speed, led to slight enhancement of the specimen average hardness value. It is due to the better distribution of the reinforcement nano-sized AlN powder (Xu, 2009; Mahmouda, 2010), as confirmed in Fig.3c, and the larger mechanical stirring volume and frictional heat, resulting in higher degree of deformation strengthening (Xu, 2009; Mahmouda, 2010). Also, the higher micro-hardness belonged to the stirred zone while lower one to the base metal. According to hall-petch relationship, higher hardness in the stirred zone than the base metal, indicates that the grain structure was finer in this zone and FSP could refine the grain structure of the processed specimens effectively (Mahmouda, 2010).

The micro-hardness profile of the stirred zone through the thickness of the plate, parallel to the FSP direction, on the cross sections of the F900, F1120, F1800 and Al specimens are shown in Fig.7. From Fig.7, the hardness of the stirred zone reduced to the hardness of the base material in the distance of about 3.5 mm from the surface of the sample. It can be concluded that FSP could harden the sample from the surface to the thickness of about 3.5 mm. As seen, similar to Fig.4, increase in rotation speed has led to enhance the average hardness value. It is related to more appropriate distribution of nano-sized powder and consequently more grain boundary pinning effect. It could refine the grain structure and harden the AA 6061 more effectively.
Fig. 3: Effect of the rotary speed on the nano-sized AlN powder distribution and aluminum nitride cluster size in the surface Al/AlN nano-composite of the: (a) F900, (b) F1120 and (c) F1800 at a magnification of 30.00 kx.

Fig. 4: The micro-hardness profile of the Al/AlN nano-composites and Al, measured parallel to the FSP direction on the surface of the F900, F1120, F1800 and Al specimens.
Fig. 5: The micro-hardness profile of Al/AlN nano-composites and Al, measured vertical to the FSP direction on the surface of the F_{900}, F_{1120}, F_{1800} and Al specimens.

**Conclusion:**

In the current research, the specimens were subjected to the different FSP conditions and the role of the rotation speed in microstructural and mechanical properties were evaluated. The achieved results could be presented as follow:

1. In the friction stir processed specimens, the stirred zone grain structure was fine, uniform and equiaxed typical feature of a recrystallized structure as compared to the base metal without visible defects.

2. Increase in rotation speed, resulted in a uniform nano-sized AlN powder distribution and smaller nano-sized powder clusters.

3. Enhanced average micro-hardness values could be obtained using higher rotation speed due to more appropriate dispersion of nano-sized powder and consequently more grain boundary pinning effect.

**ACKNOWLEDGMENT**

The authors would like to thank Semnan University and Tehran University for the financial support and the facilities.

**REFERENCES**


