

ABSTRACT

Aluminium (Al) alloys are used in transportation, defense and aerospace applications owing to their light weight and high specific strength. However, low surface hardness, wear and corrosion resistance limit the use of these alloys in various strategic applications. Microstructural modification by surface composite formation can overcome these limitations. Metal matrix surface composites possess the combined properties of the base alloy and the reinforcement. Surface composites can be fabricated through liquid and solid state processing routes. Liquid state processing may result in the formation of different intermetallics which lead to deteriorated mechanical properties. Solid state processing can avoid the drawbacks of liquid state processing such as reduced wettability and interfacial reaction between the matrix and the reinforcements. Friction stir processing (FSP) is an effective solid state processing route to develop surface composites. Formation of intermetallics can be avoided as FSP is done at temperatures lower than the base material T_m . The processing generates fine grained microstructure due to severe plastic deformation. Presence of reinforcement particles in the fine grain matrix enhance the surface hardness and reduce the thermal effects. Molybdenum (Mo) is a suitable metallic reinforcement for the surface composite owing to its high melting point, better wear and corrosion resistance compared to aluminium. Understanding the various parameters and their impact on the Al surface composites is important, yet not known. Here we present the effect of the friction stir processing parameters such as tool shoulder diameter, tool rotation speed, number of grooves, and number of processing passes as well as the reinforcement particle distribution on the resultant surface properties of Al surface composites.

The groove method is used to fabricate the materials using friction stir processing. The experiments are carried out at different process parameters such as reinforcement content, number of processing passes, tool rotational speed, tool shoulder diameter and type of reinforcement particles. The microstructural, mechanical and electrochemical properties of the base alloy are compared with the friction stir processed materials without reinforcement particles and surface composites fabricated after incorporating the reinforcement particles. The microstructural characterization is performed using optical microscope with image analyzer and Scanning Electron Microscope (SEM). The compositional analysis is carried out using Energy Dispersive X-ray Spectroscopy (EDS) and X-ray powder diffraction (XRD) techniques. Surface hardness of all the processed samples and the unprocessed base alloy in

the top surface and the cross-section surface of the processed region is measured using a Vickers microhardness testing machine. The post-processing particle size and distribution is analyzed using ImageJ analysis software. The electrochemical behavior of the base alloy, friction stir processed materials and the surface composites are studied using Potentiodynamic polarization and Electrochemical Impedance Spectroscopy. The preliminary analysis of tensile behavior of the materials is carried out using an Instron Universal tensile testing machine and the wear analysis is done using reciprocating wear testing equipment.

Single and double groove FSP experiments are carried out to study the effect of number of grooves on the hardness of surface composites fabricated using 22, 20 and 18mm tool shoulder diameter. The introduction of a second groove increases the Mo content in the surface composite. The incorporated Mo particles enhance the surface hardness of Al 1050 base alloy. The surface hardness of unprocessed material is 26 HV 0.1 and the hardness increases to 42 HV 0.1 on the top surface and 44 HV 0.1 on the cross-section surface of SGC (Single Groove Composite). The top and cross-section hardness for DGC (Double Groove Composite) are 46 HV 0.1 and 49 HV 0.1, respectively. Microhardness value of 51 HV 0.1 on the top surface and 52 HV 0.1 on the cross section surface is observed in DGC sample processed with 18mm shoulder diameter tool. The amount of Mo particles incorporated in the composite material is increased with decrease in tool shoulder diameter. The amount of reinforcement is about ~8%wt Mo in SGC and ~12%wt in DGC samples processed with 22mm shoulder diameter tool, respectively according to the microstructural analysis. The amount of Mo particles in SGC and DGC is ~17%wt and ~22%wt, respectively for samples processed with 20mm shoulder diameter tool whereas SGC and DGC fabricated using a 18mm shoulder diameter tool possessed ~27%wt and ~31%wt Mo particles. Mechanical shearing through severe plastic deformation during the friction stir processing reduces the average Mo particle size in the surface composites. Molybdenum is distributed in its elemental form in the Al-Mo surface composites. The grain size in the processed samples reduces with decrease in tool shoulder diameter. Surface hardness improves due to the combined effect of Mo reinforcement and grain refinement induced by the friction stir processing

The combined effect of number of passes and grooves on the corrosion resistance is also analyzed. Aluminium surface composites are prepared using single pass (SP) and double pass (DP) FSP groove method. Potentiodynamic corrosion testing of Al-Mo surface composites is

carried out to study the change in the corrosion resistance. Surface composites exhibit better corrosion resistance with higher corrosion potential (E_{corr}) and lower corrosion rate (i_{corr}) compared to the unprocessed base alloy and friction stir processed materials without Mo addition. Amount of Mo particles is increased by the addition of a second groove. A second processing pass homogeneously distributes this higher amount of Mo particles. Double groove double pass sample exhibits best corrosion resistance. Electrochemical Impedance Spectroscopy (EIS) analysis confirms the higher corrosion resistance of the surface composites. Surface composites with Mo particles show a charge transfer controlled corrosion behavior whereas the Al 1050 base alloy exhibits a mixed corrosion behavior. The initiation stage of pitting corrosion is restricted by the presence of evenly distributed fine Mo particles on the surface composites. The second processing pass on the double groove composite also increases the hardness of Al-Mo surface composite due to the uniform distribution of reinforcements in the grain refined Al matrix.

Different tool rotation speeds and tool shoulder diameters are used to fabricate defect-free Al-Mo surface composites. Molybdenum particles are uniformly distributed in the aluminum matrix without formation of any intermetallics with various tool shoulder diameters (18, 20 and 22mm) and tool rotation speeds (780, 900 and 1200rpm). Higher amount of Mo is observed in the surface composite processed at higher tool rotation speed and lower tool shoulder diameter. Dynamic recrystallization during FSP and pinning effect due to the Mo particles results in the grain refinement. The hardness of surface composites increases with increase in tool rotation speed and decreases with increase in tool shoulder diameter. The surface composite hardness enhances due to the coupled effect of grain refinement and Mo reinforcement. A two fold increase is observed in the average hardness of surface composites compared to the unprocessed Al 1050 base alloy.

The corrosion behavior of aluminium surface composites with metallic, ceramic and hybrid reinforcement particles are also analyzed in this study. Molybdenum, possessing higher corrosion resistance compared to aluminium, is used as the metallic reinforcement and the third hardest known material boron carbide (B_4C) is used as the ceramic reinforcement. The effect of hybrid reinforcement addition on the corrosion behavior of Al surface composites is studied by the combined addition of boron carbide and molybdenum particles. The electrochemical behavior of the developed composites is analyzed through potentiodynamic polarization and EIS. The reinforcement particles distributed in the Al matrix affect the

pitting corrosion behavior of the surface composites. The base alloy and the hybrid surface composites show diffusion controlled corrosion mechanism whereas the Mo and B₄C reinforced composites exhibit charge transfer controlled behavior. Single reinforcement addition enhances corrosion resistance of the base alloy whereas the hybrid reinforcement addition induces more corrosion. Post-corrosion microscopy analysis reveals the severity of pitting in hybrid composites. The surface composites developed with metallic, ceramic and hybrid reinforcements are free from general intermetallic phases possible based on the equilibrium phase diagram.

Al-Mo surface composites exhibit improved tensile strength and wear resistance compared to the unprocessed base Al 1050 alloy. The improvement in the wear resistance and tensile strength in the surface composites are attributed to the combined effect of reinforcement particles and grain refinement. The improved ductility in the surface composites compared to the unprocessed base alloy corresponds to the better interfacial bonding between the matrix and the reinforcement particles. The knowledge, regarding the effect of processing parameters and type of reinforcements on the aluminium surface composite properties by FSP, will be very useful in developing optimized surface composites for strategic applications.