## Synopsis

Composite materials are multifunctional materials having unique mechanical and physical properties that can be tailored to meet the requirements of a particular application. Aluminium based Metal Matrix Composites (MMC) always draw the attention of researchers due to its unique characteristics such as better strength to weight ratio, low wear rate and lower thermal expansion coefficient. There are various methods for manufacturing of MMC that can be grouped into two major categories: (a) Solid sate method such as powder metallurgy, co-extrusion and (b) Liquid state method such as stir casting. All of these methods for production of composites have their own advantages and disadvantages. Porosity, and poor wettabilty of dispersoids with matrix are few common problems in solid state route. Formations of undesirable phases, and segregation of dispersoids are common problems in liquid state processing route.

Friction Stir Processing (FSP) technique, a derivative technique of Friction Stir Welding (FSW) has emerged as a major solid state technique to produce composites. However, there are several challenges associated with it. Most of the past work has been on limited volume of material. Researchers have tried to combine FSP technique with powder metallurgy technique to overcome aforementioned challenges associated with these techniques. Where on one hand, powder metallurgy ensures the uniform dispersion of dispersoids in the matrix, on the other hand FSP on sintered billet removes the pores and other defects. The combination of these two techniques leads to a more controlled and uniform properties. However, at the same time, it can be noted that the combination of these processes is tedious and time consuming.

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In this study, an attempt is made to achieve bulk dispersion of a second phase into an aluminium matrix using FSP technique. A 5 mm thickness composite is attempted in this work. To achieve this objective proper and uniform mixing of the particles is required. To achieve this, new tools and processing steps are to be designed and analyzed for a better understanding of material flow around the tool pin and the effect of different tool pin geometries on the material flow. Keeping this objective, a detailed study is carried out on material flow during FSW process using aluminium as base metal. A marker material technique is employed to understand the material flow. A strip of copper is selected as the marker material. Material flow can be qualitatively predicted during the process by observing the distribution of marker material in the weld nugget. Three different kinds of tools, each with an additional feature are designed for this purpose (a) Plain frustum shape pin (b) threaded frustum shape pin and, (c) Triflute pin. The material flow due to the plain pin tool can be considered as primary flow during the FSP. Three different kinds of flow zones are observed in the weld nugget in the case of plain tool. It is found that higher numbers of geometrical features (threads and flutes) not only enhance the material flow but also lead to the additional flow currents and more thorough and uniform mixing.

A closer study of the weld nugget revealed that the copper marker particles and the matrix were diffusion bonded. Based on the reaction time available and temperature in the weld nugget a diffusion layer thickness of 4 nm is expected between copper and aluminium. However, the diffusion layer thickness was found to be 3.5 µm, which is nearly three orders of magnitude higher. This can be attributed to the enhancement of diffusion due to simultaneous application of strain and temperature.

As copper is soluble in the aluminium, an insoluble marker material tin was used for study of flow in the weld nugget. However, the effect of insolubility and lower melting point had some unexpected effect on the processing loads. The normal load during steady state tool traverse in conventional butt-welding is found to be around 2.7 KN while it attains an average value of 14.7 KN when a thin strip of tin is sandwiched between these plates. However, a drop in the torque of around 13.12 NM is observed when tin was sandwiched between the plates as compared to the case when no insert was present. On closer examination of the flow behavior, it is seen that the tin melted, squeezed out and formed a lubricious layer between the tool and the work piece. This reduced the torque significantly and a concomitant drop in temperature was observed. The interaction between the tool and the colder aluminium work piece would thus result in much larger normal and transverse load

Based on the expected and unexpected results of flow pattern in the weld nugget, a new FSP tool and processing steps were developed to manufacture MMC. Tungsten, which is the highest melting point metal is chosen as the dispersing phase. Further, as tungsten has high melting point, the kinetics of intermetallics formation would be low for the given FSP processing time at the processing temperature. This would lead to tungsten acting as a more ductile strengthening particle, which is expected to should give some unique characteristics to the MMC. Tungsten powder with an average diameter of 414 nm was dispersed in aluminum matrix with three different proportions after optimizing all the process parameters. It is noted that the mechanical properties are significantly influenced as the tungsten content in the matrix increases. In practice, MMC shows relatively low ductility compared to the parent metal. However in this case the composite exhibited even better

ductility than the as received aluminium plates (rolled sheets). The composite showed around 129 MPa of yield strength along with 21% ductility when tungsten content is 3.8 at.%. It is also found that the reaction between aluminum and tungsten occurs during the processing and form the Al<sub>12</sub>W intermetallic phase. Though the formation of this intermetallic phase was unlikely due to the low temperature and short time available during the process, the reaction kinetics between aluminium and tungsten would have been enhanced due to the simultaneous application of strain and temperature.

Given that the metal-metal, tungsten-aluminium composite produced by FSP had unique properties and also formed intermetallics, a study on incorporation of a highly insoluble material, graphite was carried out. Further graphite with its own unique properties and very low wettability with aluminium could possibly impart completely different properties to the system. Past work on graphite aluminium composites produced by other methods did not show promise. As FSP imposes high strains at relatively high flow stresses on the processed material, it was seen that the graphite got sheared to form multi-layer graphene composites with the aluminium. The graphene sheets are formed by mechanical exfoliation of graphite particles during its incorporation in the matrix. The formation of graphene was confirmed after separating the graphite from the processed zone and TEM studies of the composite. It is seen that most of the graphite got converted into multilayer graphene. This aluminiumgraphene composite exhibited enhanced ductility and UTS. As received aluminium plates exhibited only 11% ductility and around 100 MPa of UTS while this composite exhibited around 26 % ductility and 147 MPa of UTS. However, there is only a slight improvement in yield strength of this composite.