Making ceramic–metal composite material by friction stir processing

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Abstract: An innovative method to add ceramic particles in the metal matrix to make ceramic metal matrix composite was experimented and proved with alumina powder as particles and AE42 magnesium alloy as matrix. The alloy was subjected to friction stir processing and alumina particles were added through the processing tool. AE42 magnesium alloy has primary α-Mg phase of 100-150 micron grain size and secondary phase of 10-50 micron size as precipitates. Al12RE, Al11RE3 and Al17Mg12 are main secondary phases in the form of precipitates. Alumina powder was selected with average particles size of 5 micron. For processing parameters of 300-400 rpm tool speed, 15-20 mm/minute traverse speed and a threaded pin geometry; composites with fine distribution of second phase precipitates and alumina particles in the matrix were observed. Mechanical and microstructural characterization revealed uniform properties in longitudinal and transverse directions. Composite material has superior mechanical properties than the magnesium alloy. Distribution of particles was up to the length of tool pin. Tool pin geometry, feed rate and volume percentage of alumina particles, processing speed and tool rpm on the effect of mechanical and micro-structural properties were analyzed in detail.

1. Introduction:
Friction Stir Processing (FSP) is one of the advanced material processing methods based on friction stir welding. It was invented and patented by TWI – The Welding Institute [1]. This process is used for changing and improving the cast microstructures of metallic materials and many studies were carried out in last 20 years [2]. In FSP, a rotating tool made up of comparatively stronger metal is plunged inside the job and traversed to make processed zone. Many researchers worked on this topic and optimized the process parameters for various materials. Also, literatures are available for FSP of composites [3]. But very few attempts were made to use FSP itself for making composites. These attempts resulted in success for making composites up to a small thickness [3].
For making Metallic Matrix Composites (MMC), various methods including powder sintering, melt addition (squeeze casting, spray deposition, stir casting), diffusion bonding, electroplating are followed [4]. But the limitation of each method results in innovating new methods. All these conventional methods lag the possibility of applying it in-situ or making composites by secondary processes.

For an innovative idea, FSP was experimented to make MMC with alumina as particles, AE42 magnesium alloy as matrix. As this method is expected to make improved surface properties, functionally graded composite and metallic materials, this study was conceptualised and carried out. Due to its light weight, alloys and composites based on magnesium are of special interest for much application including aerospace [5]. But magnesium alloys suffer from brittleness and low strength [6]. Magnesium alloy composites improve them; FSP improves further. So, in the study both were combined. When the composite making by this method is successful, it can be used in-situ composite making, surface modification, and to make functionally graded materials etc. AE42 magnesium alloy was selected for the study as it was specially developed for automotive applications, with better creep resistance.

2. Experimental Details:
Experimental set up for making magnesium-alumina MMC using FSP is shown in figure 1. For powder insertion, a hole was made in the FSP tool and it was inserted by a pressure delivery method. This is shown in figure 2. After powder (alumina particles) filling, the tool is fitted and made to do processing as in conventional FSP. The spring presses and delivers the powder which results in a stir zone (of magnesium) with alumina particles. Alumina particles used in the experiment are of 5-6 micron size. Percentage of alumina in the matrix is controlled by the hole diameter of tool. For comparative purpose, processing of magnesium alloys were carried out with and without alumina (with alumina is composite).

AE42 alloy with 3 mm thickness was used for FSP experiments. Properties and composition of the alloy used are shown in Table 1. Experimental parameters are given in Table 2. After the experiments, microstructural analysis by Tescon Scanning Electron Microscope (SEM), hardness testing by Shimadzu Vickers Hardness machine with load of 2 kg, tensile testing by Shimadzu UTM as per ASTM E8 at cross head speed 2 mm per minute were carried out. Samples were prepared for all these analyses as per standards from the processed zone by cutting.

| Property | Y.S (MPa) | UTS (MPa) | Elongation (%) | Hardness (VHN) | Composition (Wt %) | Form |
|----------|-----------|-----------|----------------|----------------|-------------------|------|
| Value    | 130       | 225       | 6              | 72             | Al :3.9, Ce :1.2, Mn :0.3, La :0.6, Nd :0.4, Pr :0.1, Th :0.2, Mg :Balance | Die cast |

| Parameter | Tool speed (rpm) | Traverse speed (mm/minute) | Tool pin length and diameter | Alumina powder feed (Volume) |
|-----------|------------------|----------------------------|----------------------------|-----------------------------|
| Value     | 300-600          | 15-40                      | 3 mm pin length and diameter | 500 mm³                     |
3. Results and Discussions:

3.1. Tensile Testing:
Mechanical properties of magnesium alloy improved by FSP, and further improved for composites. Processed zone with alumina (composite) had 15% higher strength than the processed alloy. Notably elongation of composite was in comparison with processed alloy (without alumina). Table 3 shows the tensile and hardness results of composite achieved for processing parameters of 400 rpm tool speed, 20 mm/minute traverse speed; the strength value was higher for 20 mm/minute traverse speed, which may selected as optimum.

3.2. Hardness:
Hardness of the composite increased to 93 VHN from 72-74 of base metal (magnesium alloy). Increase in hardness may be attributed to refined microstructure and tiny and uniform distribution of second phase particles, and the alumina particles that were highly oriented along with matrix [7]. It is to be noted that the hardness value of processed zone without alumina particles was lower than 93.

| Material condition                          | YS (MPa) | UTS (MPa) | Elongation % | Hardness (VHN) |
|--------------------------------------------|----------|-----------|--------------|----------------|
| Processed zone without alumina (300 rpm, 20 mm/minute) | 101      | 245       | 11.4         | 83             |
| Processed zone with alumina (MMC) (300 rpm, 20 mm/minute) | 115      | 281       | 10.9         | 93             |
| Processed zone without alumina (400 rpm, 20 mm/minute) | 104      | 255       | 11           | 82             |
| Processed zone with alumina (MMC) (400 rpm, 20 mm/minute) | 122      | 291       | 10.2         | 90             |
| Processed zone without alumina (600 rpm, 20 mm/minute) | 103      | 251       | 10.5         | 80             |
| Processed zone with alumina (MMC) (600 rpm, 20 mm/minute) | 120      | 286       | 9.9          | 88             |
3.3. Microstructure:
Microstructural results are shown in figure 3 and 4. From the microstructures it is evident that the grain size of the processed zone was refined to 4-5 micron, second phase particles along with alumina particles were distributed evenly to the matrix. Flow of the alumina was as per the tool flow direction. Energy Dispersive X-ray Spectroscopy (EDS) from figure 4 confirmed the presence of alumina particles. Secondary phases and alumina particles were in the range of 1-10 micron in the matrix.

Figure 3. SE Microstructures of (a) PM, (b) processed zone (without alumina) and (a) MMC

Figure 4. EDS results of MMC, spectrum 2 defines alumina particle
4. Conclusions:

- A new novel method was experimented and proved to make metal matrix composites by FSP.
- The composite resulted in improving the mechanical properties of magnesium alloy.
- Microstructural results revealed grain refinement, equally distributed alumina particles were confirmed by SEM and EDS.
- This process makes composites up to the pin length of FSP tool, can be used to make MMCs from secondary processes.

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