

ABSTRACT

Metal matrix nanocomposites (MMNCs) are superior to conventional alloys in terms of mechanical and thermal properties and are adaptable for current industrial requirements. The low density with adequate strength, ductility, and the flexible processing capability makes Aluminium (Al) and its alloys the most investigated matrices for nanocomposites. Solidification processing methods such as casting are cost-effective for large-scale fabrication of MMNCs. However, particle agglomeration, severe interfacial reaction of *ex-situ* added nanoparticles and fluidity reduction individually or collectively can deteriorate the mechanical properties of solidification processed MMNCs and these issues are not yet unravelled completely. Hence, in the present work, an attempt has been made to address the above stated challenges in MMNC fabrication by *in-situ* synthesis of nanocomposites and their semisolid processing. Ultrasonic treatment (UT) is employed to synthesize thermally stable *in-situ* nanoparticles which can eliminate the interfacial reaction issues and also improve the particle dispersion. The semisolid processing of nanocomposite is primarily aimed to alleviate the fluidity related issues. On the application of shear force in semisolid conditions, the viscosity of metal will be apparently reduced and thus it can overcome fluidity related issues during the processing of MMNCs. In addition, the globular microstructure development during semisolid processing helps in retaining the matrix ductility of the composites.

To comprehend the scope of work, pure Al was selected for optimization of UT processing parameters to synthesise *in-situ* nanocomposites. A356.0 and Al 2014 alloys were selected as the matrix materials and reinforced with *in-situ* TiB₂ particles. A comprehensive study on the effect of reinforcement particles on age hardening behaviour of A356.0 and Al 2014 matrices was conducted. An attempt to understand the effect of UT on strontium based eutectic modification of A356.0 alloy was also made. The effect of ultrasonic treatment

and semisolid processing parameters on the microstructure and mechanical properties of the nanocomposite was evaluated. The structure-property correlation is also investigated and discussed in detail.

Pilot studies during optimisation of UT process parameters revealed that the UT of composite melt during the *in-situ* reaction of fluoride salts and UT after completion of the *in-situ* reaction can generate nanoparticles in the melt. Nevertheless, UT of composite melt during the *in-situ* reaction contaminates the composite melt by sonotrode dissolution while post *in-situ* melt treatment method can alleviate the melt contamination. Hence, post *in-situ* reaction ultrasonic treatment of conventional salt-melt route Al/TiB₂ composite was used to fabricate and disperse nanoparticles effectively in the matrix of the composites. Post reaction UT of A356.0/TiB₂ and Al 2014/2TiB₂ composites reduced the size of TiB₂ particles from 950 ± 80 and 800 ± 110 nm to 34 ± 16 and 24 ± 13 nm respectively with improved dispersion. The age hardening studies of composites with and without UT revealed that the presence of both micron and nano sized reinforcement particles increased the aging kinetics of both Al 2014 and A356.0 matrices. Age hardening of Al 2014/TiB₂ micron and nanocomposites enhanced hardness and age hardening rate when compared with monolithic alloy. The age hardening studies added that the aging rate of composite samples with nano sized reinforcement particles is higher than micron composites. However, the interaction of fluorine from salt flux and Mg at various stages of composite preparation resulted in a loss of Mg content that led to the reduction in peak hardness of composites compared to A356.0 alloy. To compensate the mechanical property reduction due to Mg loss and associated issues with age hardening, an attempt has been made with eutectic modification of A356.0 alloy and composites. It is found that the addition of Sr to A356.0 alloy reduced the length of eutectic Si from $15.6 \mu\text{m}$ to $0.65 \mu\text{m}$ with a simultaneous increase in porosity from 0.84 % to 3.59 %. UT of Sr modified A356.0 alloy reduced the specific porosity volume to 0.86 % and retained the eutectic modification. Ultrasonic cavitation assisted degasification and bi-film

breakage is proposed as the governing mechanism for porosity alleviation. Thermal analysis of unmodified and Sr-modified A356.0 alloys with and without UT confirmed that the ultrasonic treatment of Sr modified A356.0 alloy retains the quality of eutectic modification. UT of the Sr added alloy and composites improved tensile properties due to the effect of microstructure modification and porosity mitigation.

The non-dendritic feedstock for thixoforming was prepared by liquidus casting route and the microstructural evolution during the semi-solid isothermal holding of liquidus cast samples was conducted in detail. It was observed that the liquidus casting is effective in producing non-dendritic microstructure. Isothermal holding of the samples at semisolid condition enhanced the sphericity of samples for initial few minutes of holding and further increase in holding time resulted in grain coarsening and reduction in sphericity. The grain coarsening rate (K) of samples are analysed with Lifshitz-Slyozov-Wanger (LSW) equation. The semisolid processing (thixoforming) of alloy and composite samples performed using the parameters identified from the isothermal holding studies and thixoforming induced a microstructural transformation into globular type. As the combined effect of ultrasonic assisted *in-situ* synthesis of TiB_2 nanoparticles, eutectic modification and thixoforming, the yield strength of A356.0 alloy improved by 64 MPa with concomitant ductility enhancement from 4.5 to 7.4 %. Similarly, the yield strength of as-thixoformed Al 2014 alloy is increased by 102 MPa with ductility enhancement from 17.1 to 18.7 %. The peak aged yield strength of thixoformed Al 2014 alloy is enhanced by ~130 MPa with ductility improvement from 19.7 to 20.1 %. Structure-property correlation studies show that particle-dislocation interactions in as-cast condition and combination of particle-dislocation interactions and shearing of precipitates by dislocations in precipitation hardened condition enhance the strength. These results have a significant impact in the field of Aluminium MMNCs.