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Microwave-assisted T6 heat treating of aluminium alloy-Al2O3 nanocomposites

Microwave assisted T6 Heat Treating of aluminium alloy-Al_2O_3 nano composites

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Abstract

Blending ultra fine-grained microstructure with other added reinforcement elements that can provide good strength-microstructural integrity is the primary metallurgical target for any developed composite to exhibit its maximum strength at reduced cost and energy in aerospace application. Such nano reinforced aluminium alloy composites are processed through addition of nano alumina using powder metallurgy route followed by microwave assisted T6 heat treatment. Homogeneous nucleation by increasing the activation energy at the grain interface by addition of nano alumina resulted in ultra fine grain microstructure and significantly enhanced the mechanical properties like microhardness and strength of the fabricated nanocomposites.

1. Introduction

Aluminium alloy composites have gained the attraction of the space industry for its good strength at reduced weight and variety of processing methods available for fabrication. The advancement in the aluminium alloy composites facilitated novel researches to address the global engineering challenges by development of new novel methods for processing and fabrication. Out of all manufacturing methods, powder metallurgy technique and its range of factors to attain the good strength-microstructural integrity of the developed composites is found to be promising technique to process and fabricated aluminium alloy composites for aerospace applications [1]. Similar to other 2xxx series of aluminium alloys AA 2900 have the potential for various aerospace applications and this alloy matrix can be used to build aerospace composites by varying suitable reinforcements. Researchers in recent times have proved that adapting novel processing methods can enhance the properties of developed composites for more high strength aerospace application. "Microwave Processing" is one among novel technique known as quick processing technique that gained attraction due to combination of the unique sintering science and reduced processing duration [2, 3]. Recent literature predicts that nano alumina has registered its trend as reinforcing material, trouncing all types of ceramic material as reinforcement due to its excellent properties [4 - 6] and considered to be the more suitable material to create ultra fine-grained microstructure when microwave processed in combination with aluminium alloy matrix. Activation energy, precipitate nucleation and formation of stable intermetallic precipitates at the grain boundaries were all the challenging factors that T6 conventional heat-treating methods faced all these years for processing aluminium alloys and its composites [5]. Comparing conventional sintering methods with microwave sintering, microwave sintering exhibited enhanced strength-microstructural characteristics in aluminium composites with dominant control over the microstructure of the composites [7]. In this novel direction, the current research work was focused to study and validate the mechanism of microwave assisted T6 heat treatment on the grain boundary nucleation and formation of ultra fine-grained microstructure addressing the matrix and nano reinforcing material along with further investigation on the strength-microstructural improvement aspect scientifically.

2. Processing of AA 2900-Al_2O_3 nanocomposites

Processing and sintering mechanism of AA 2900-Al_2O_3 nano composites were fabricated through powder metallurgy route followed by microwave solution treating and T6 Microwave aging (Fig. 1a and Fig. 1b). Average particle size of 20 microns AA 2900 and 10 nm of Al_2O_3 were used. Resultant grain size and grain orientation analysis was found with Electron backscatter diffraction (EBSD) technique (Fig. 1c, Fig. 1d and Fig. 1e). Composite for EBSD were prepared by electro polishing technique using 20% perchloric acid and 80% methanol for 100 s at potential difference of 12 V. The EBSD examination was studied using FEI-Quanta 200HR- SEM following step size of 0.1 μm and with TSL OIM software [8]. The average grain size of the composite, processed with microwave heat treatment technique was found to be 10.4 μm. The mixed orientation along the [001], [101] and [111] texture for the microwave heat treated composite exhibited grain boundary precipitates and also at matrix reinforcement interfaces. From fig 1c and 1e, it is clearly evident that microwave heat treating of composite lead to dynamic grain refinement and induced grain boundary pinning by Orowan strengthening mechanism that improved strength of the composite by blocking dislocation movements. The combined effect of the processing temperature and activation energy controlled the mechanics of nucleation and growth of precipitates. AA 2900 added with nano alumina (Al_2O_3 Particle) with 2 wt% are initially ball milled for 120 min in a high energy planetary ball mill setup in acetone medium to achieve homogeneous dispersion and the mixed powder slurry is filtered to remove the acetone and dried for 60 min at 75 ^{o}C. Thus, obtained powder mixture are mechanically mixed with polyvinyl alcohol (PVA) and hot compacted in split dies at 300 MPa pressure feed with powder preheated at 150 ^{o}C to get Ø25mm x 10 mm billets which are sintered and solution treated at 495 ^{o}C for 20 min with continous flushed nitrogen gas atmosphere. Thus, sintered composite billets are cold water quenched and artificially aged for 6 hours in a microwave furnace at 190 ^{o}C as per the ASTM B308. The rate of heating during microwave solution treating was maintained at 50 ^{o}C /min and 30 ^{o}C /min for microwave artificial aging.

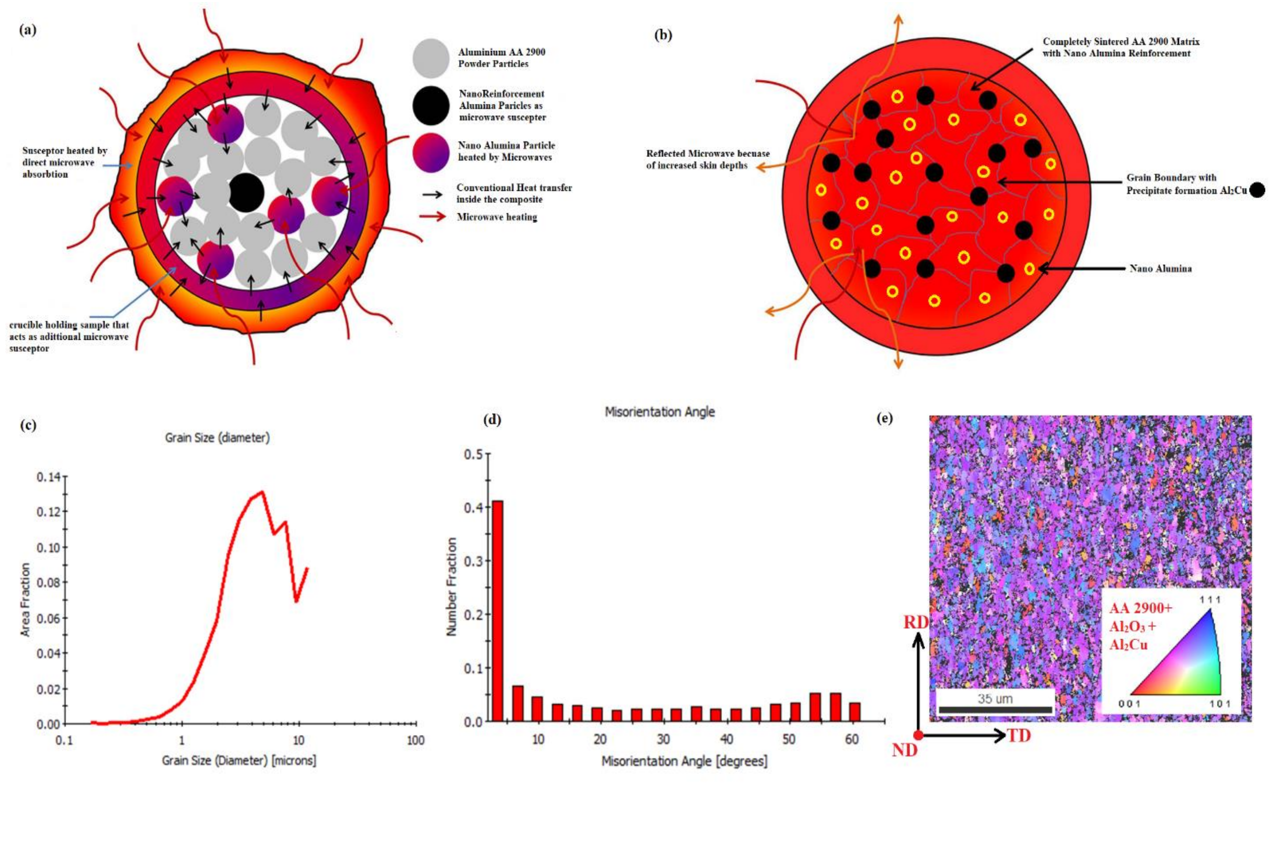

Fig. 1. (a) Microwave processing mechanism (b) microwave T6 heat treatment mechanism (c) grain size distribution (d) misorientation angle chart (e) EBSD maps

3. Result and discussion

3.1 X-ray diffraction (XRD) and micro-hardness validation

X-ray diffraction validation (Fig. 2) was performed to confirm the internal phase constituents of the T6 microwave heat treated AA 2900-alumina nano composites. Profound peaks of aluminium identified at ~38.5^o (111), ~44.7^o (200), ~65.9^o (220) and ~78.4^o (311). Out of all peaks high intensity peaks are recorded at ~38.5^o (111) and ~44.7^o (200). This represents the introduction of residual stress induced in fabricated composites due to blending of nano alumina particle, microwave assisted heat treating process and generation of compressive residual stresses.

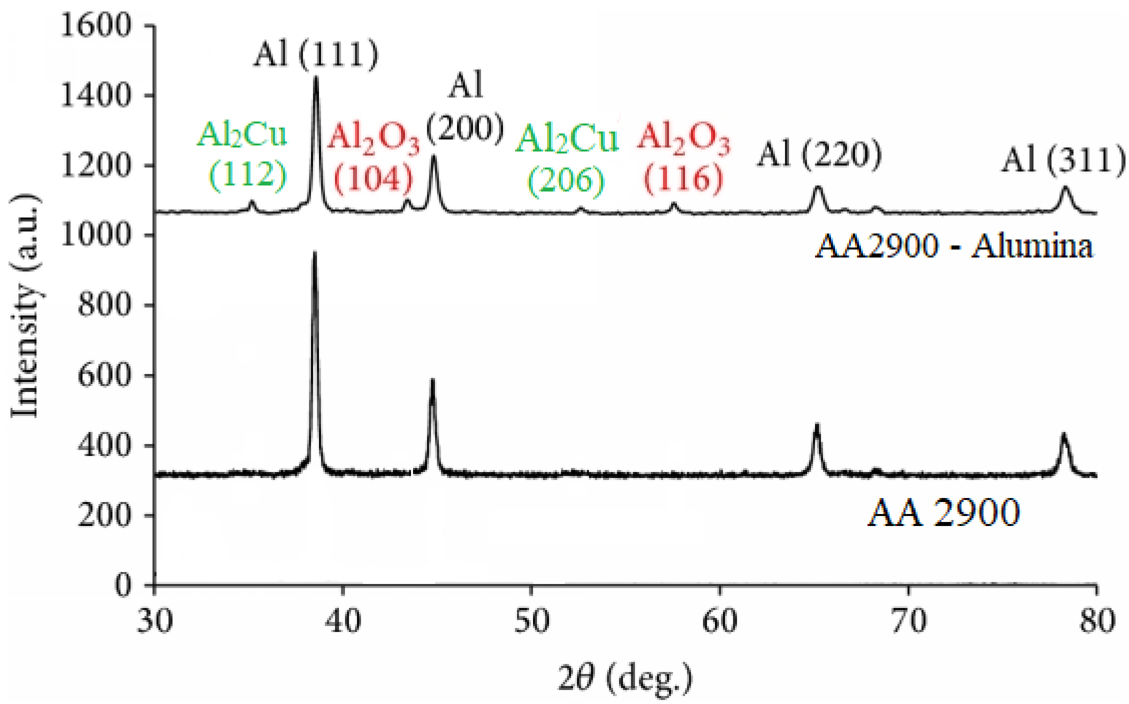

Fig. 2. XRD of different crystalline planes of AA 2900 – alumina nanocomposites.

Vickers microhardness (HV) experiment was performed as per ASTM E-384 standard with (Matsuzawa MMT-X, diamond indenter) maximum indentation load of 400 g and holding duration of 12 s. The surface average microhardness of the nanocomposites was increased (HV - 183) due to the microwave assisted T6 treatment which provided fast and homogeneous nucleation of Ɵ-precipitates like Al_2Cu in combination with the reinforced nano Al_2O_3 compared with parent 2900 aluminium alloy (HV - 131). This propensity of improvement is in good agreement with strengthening principles like the improved activation energy for the grain boundary nucleation of precipitates, Orowan looping, precipitation hardening, particle strengthening and grain boundary strengthening.

3.2 High-resolution transmission electron microscope (HRTEM) grain interphase analysis

The ultra fine grain structured AA 2900-alumina nanocomposites are prepared through milling followed by electro polishing, dimpling and analyzed for the grain boundary interphase through high-resolution transmission electron microscopy (Fig. 3a). HRTEM image clearly indicated the grain interphase between the aluminium matrix and the nano-Al_2O_3 particle and the interphase is nucleated with the Ɵ-Al_2Cu precipitate. This integrity of ultra fine grain with aluminium-alumina-precipitate is mainly due to the microwave T6 heat treatment and microwave solution treatment of nanocomposite.

Further, nano alumina has extensively bonded throughout the circumference signifying that microwave T6 process did generate an enough temperature at the interface of both matrix and reinforcement. Generation of such strong bonds are mainly due to rise in the activation energy at the grain interfaces because of microwave T6 treatment creating ultra fine grains [9]. Further, grain growth is also stopped because of Ɵ-Al_2Cu nucleation at the grain boundary (Fig. 3a and Fig. 3b). The respective ring diffraction pattern (Fig. 3c) indicates ultra fine grains (UFG) are polycrystalline aluminium and increased cluster on region attributed to accrual of dislocations [10]. Additionally, diffraction rings are in strange shapes (disturbed rings) and a discontinuous spot in the ring diffraction pattern corresponds to the arbitrary orientation of various crystals planes owing to grain boundary strengthening and precipitation strengthening mechanism. This is due to existence of nano alumina, which influences the grain boundary nucleation of Al_2Cu as alumina acts as a microwave recipient leading to formation of ultra fine-grained structure. This behaviour is a proof for the effect of pinning on added nano alumina, by blocking the crystalline growth in the nanocomposites leading to UFG structure.

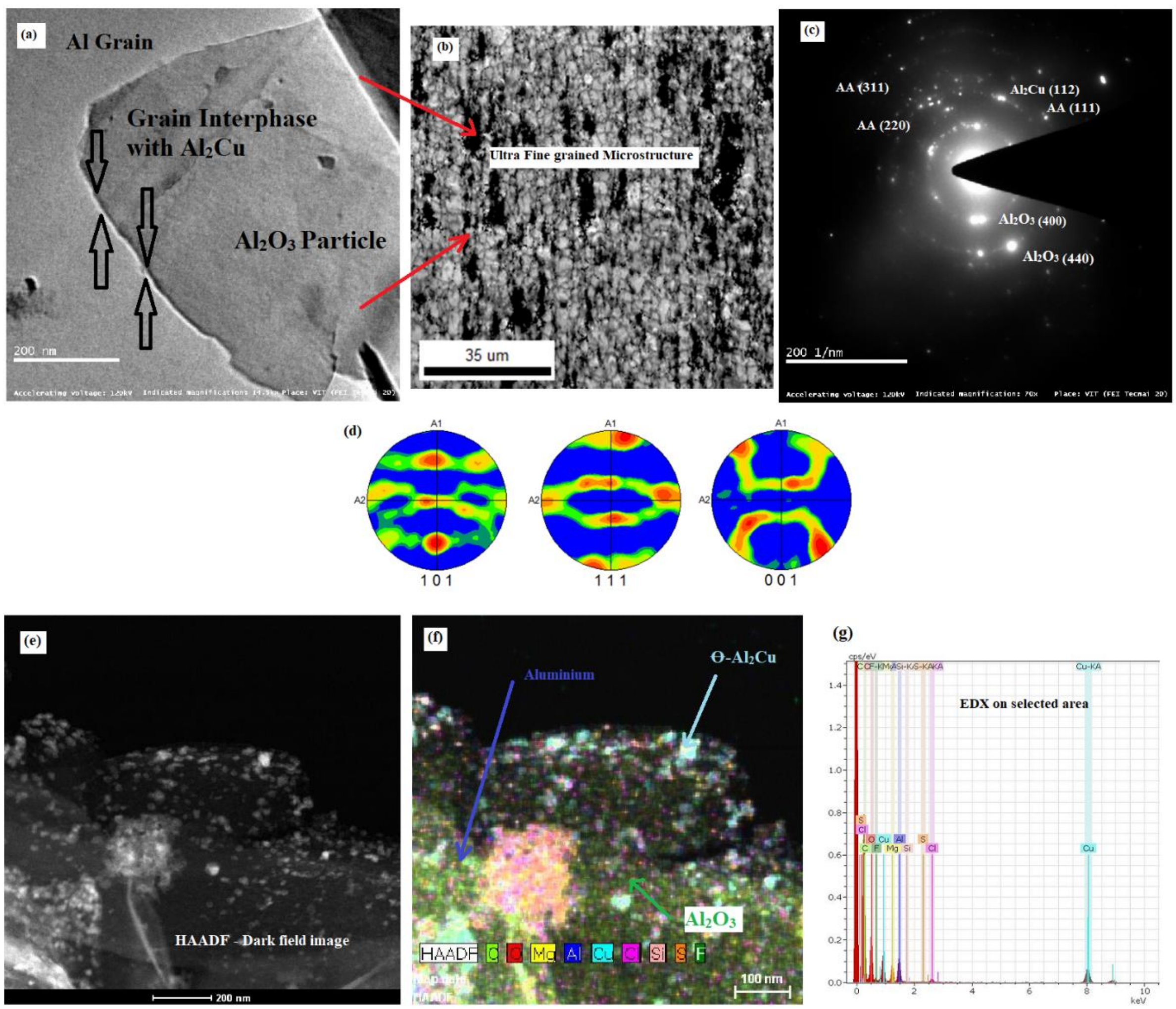

Fig. 3. (a) HRTEM Grain interphase (b) HRSEM Ultra grained structure of nanocomposite (c) diffraction pattern of Microwave T6 nanocomposite (d) pole figures (e) HAADF-STEM of ultra fine grain (f) element map of nanocomposite and (g) EDX of AA2900-alumina nanocomposite

EBSD (Fig. 3d) confirms the ultra-fine-grained structure and found higher grain orientation along mixed (101), (111) and (001) composite microstructure with average grain size of 10.4 μm (Fig. 1d). The existence of nano alumina and its blend with Ө-Al_2Cu at the interface is confirmed using high-angle annular field imaging (HAADF-HRTEM) (Fig. 3e) and all the elemental maps (Fig. 3f). This behavior confirms that nucleation of Ө-Al_2Cu achieved at the grain interface. Ө-Al_2Cu at the grain interface and the excellent interface bonding between aluminium and nano alumina are achieved by microwave T6 treatment process which is confirmed by element map analysis (Fig. 3f) and EDX describes the constituents present in the nano composite (Fig. 3g). Thus, HRTEM, element mapping and EDX evaluation confirms nanocomposite interface, excellent bonding of aluminium and nano alumina and grain boundary nucleation of Ө-Al_2Cu as a result of microwave T6 treatment. This phenomenon leads to achieving an ultra-fine-grained microstructure of the nanocomposite with enhanced properties [12].

The tensile experiment was performed in a universal tensile testing rig (Instron - 8801) as per ASTM – E08 standards (sample thickness 6mm) with strain rate of 0.5 mm/min. Fig. 4 shows the stress-strain relation for the AA 2900 - alumina nanocomposite and microwave T6 treated nanocomposite specimens. AA 2900-alumina nanocomposites processed with microwave T6 treatment with UFG structure exhibited tensile strength of 460 MPa which was 28.2 % development in UTS compared to the AA 2900-alumina nanocomposite without microwave T6 treatment (330 MPa) by reinforcing 2 wt% of nano-Al_2O_3. An enhancement in the UTS due to the grain boundary nucleation of Ө-Al2Cu and obtaining UFG due to microwave T6 treatment in the AA 2900-alumina nanocomposites is good agreement with the early researchers working on pure aluminium [11]. This significant enhancement with very short processing time, lesser reinforcement wt% of nano alumina has technology edge over compared to other conventional heat treatment techniques. Thus, microwave assisted T6 heat treatment of nanocomposites are potential techniques which makes the method novel to achieve ultra fine grain microstructure in the developed nanocomposites [12]. Such momentous enhancement is due to targeted nucleation of the ϴ-Al_2Cu at the grain boundary interface which forms throughout the bulk of the nanocomposite which is processed by microwave T6 treatment followed by powder metallurgy fabrication.

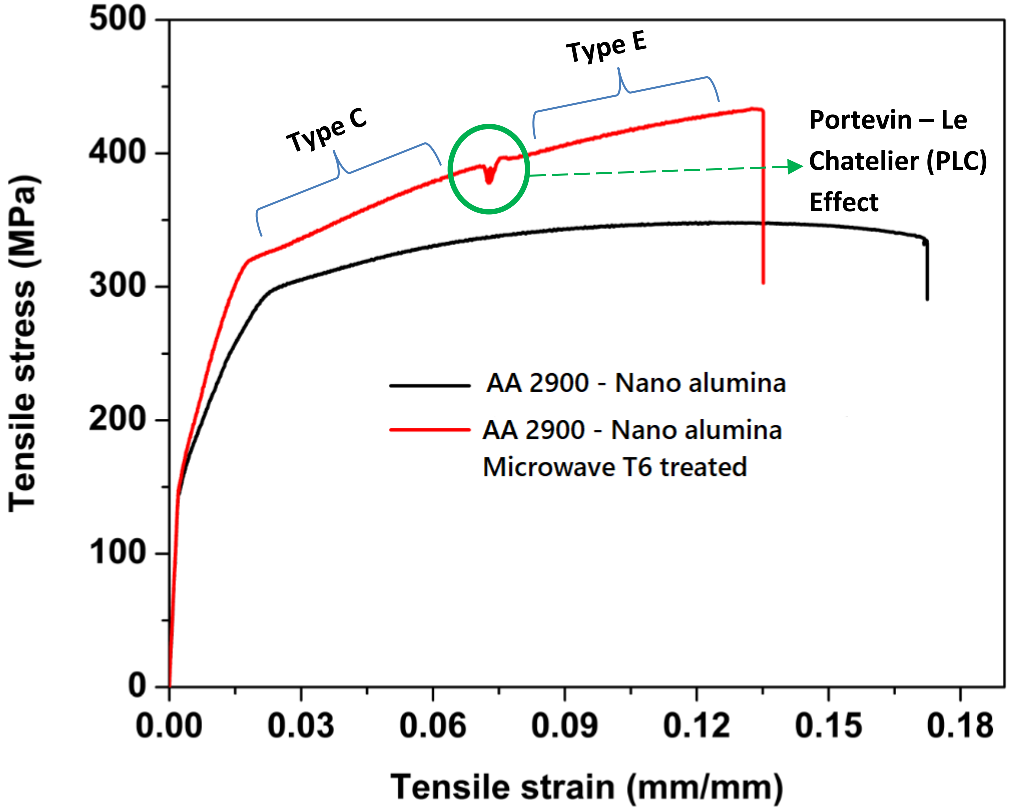

Fig. 4. Stress-strain curves of nanocomposite and (UFG) microwave T6 nanocomposite.

From fig 4 it is recorded that serrated type or Portevin–Le Chatelier (PLC) effect of type C was observed in the engineering stress-strain curve right after yield point, tailed by type E beyond a strain value of ~3.5% as a result of Dynamic Strain Aging (DSA) [14]. The PLC effect noticed is mainly produced by DSA primarily resulted from the interaction of diffusing solute atoms (mainly Mg-Cu-Sn in this case) and dynamic dislocations, therefore increasing the required activation energy for driving the slip planes through the plastic deformation [15]. From fig 4 it is clearly evident that a stress dip induced at a point when the composite reached a level to trigger the motion of pinned dislocations, followed by the concentrated dislocation movement until the dislocations are re-pinned [15]. Thus, load transfers from matrix to reinforcement are solely an interfacial bonding among them and inter particle shear stresses. Nevertheless, notable increase in the UTS of the microwave T6 nanocomposite gives a higher strength than nanocomposite AA2900-alumina samples furnace cooled with marginal decrease in ductility [13]. This phenomenon of increased strength of the microwave T6 treated nanocomposites is ascribed to form ultra fine-grained microstructure in combination with the grain boundary nucleated Θ-Al_2Cu. Comparable trend of ultra fine-grained microstructure is observed in samples that are post processed with surface treatment either mechanically or chemically, which exhibits higher strength after such processing (i.e., Friction stir processing, laser treatment, chemical etching and ion diffusion). So, ultra fine-grained microstructure plays an important role in strengthening nanocomposites, which was achieved by employing microwave T6 treatment technique which is identified as precipitation strengthening and grain boundary strengthening mechanism by Orowan theory of strengthening.

4. Conclusions

Microwave assisted T6 treatment on AA2900-alumina nanocomposites exhibited well structured integrity with ultra fine-grained microstructure and presence of nano alumina enabled a novel dimension of sintering and strengthening mechanism when exposed to microwaves. Subsequently, considerable improvement in strength was achieved on the nanocomposites through novel heat treatment mechanism in agreement with general theory of composites strengthening.

Ultimately, the combination of microwave T6 treatment and addition of nano-alumina direct to ultra fine-grained microstructure significantly influences mechanical and other properties of the nanocomposites. So, this research study is conveying the principle of achieving ultra grain microstructure in a powder metallurgy fabricated and microwave assisted T6 processed nanocomposite without even processing for secondary operations which eventually reduces the cost and energy for manufacturing composites. Such kind of novel techniques can be employed to process light weight alloys to serve a high strength application in aerospace gears application.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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