



Original Article

Effect of Al–5Ti–1B grain refiner on the microstructure, mechanical properties and acoustic emission characteristics of Al5052 aluminium alloy



Amulya Bihari Pattnaik^{a,*}, Satyabrata Das^a, Bharat Bhushan Jha^b,
Nedumbilly Prasanth^a

^a Light Weight Metallic Materials Group, CSIR-Advanced Materials and Processes Research Institute, Bhopal, Madhya Pradesh, India

^b Surface Engineering Department, CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, India

ARTICLE INFO

Article history:

Received 15 July 2014

Accepted 29 October 2014

Available online 28 November 2014

Keywords:

Microstructure

Tensile strength

Grain refinement

Mechanical properties

Acoustic emission

ABSTRACT

In the present investigation, the effect of Al–5Ti–1B grain refiner on the microstructure, mechanical properties and acoustic emission characteristics of Al 5052 aluminium alloy have been studied. Microstructural analysis showed the presence of primary α solid solution. No Al–Mg phase was found to be formed due to the presence of magnesium in the solid solution. The results indicated that the addition of Al–5Ti–1B grain refiner into the alloy caused a significant improvement in ultimate tensile strength (UTS) and elongation values from 114 MPa and 7.8% to 185 MPa and 18% respectively. The main mechanisms behind this improvement were found to be due to the grain refinement during solidification and segregation of Ti at primary α grain boundaries. Acoustic emission (AE) results indicated that intensity of AE signals increased with increase in Al–5Ti–1B master alloy content, which had been attributed to the combined effect of dislocation motion and grain refinement. The field emission scanning electron microscopy (FESEM) and energy dispersive X-ray (EDX) analysis were used to study the microstructure and fracture surfaces of the samples.

© 2014 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. All rights reserved.

1. Introduction

Al–Mg alloys are a potential candidate for automobile industries owing particularly to their high strength to weight ratio, good corrosion resistance, weldability and formability [1]. The microstructure of Al 5052 aluminium alloy mainly consists of

primary α -Al phase and magnesium in the solid solution. The refinement of grains results in the formation of fine equiaxed grain structure, which not only improves the mechanical properties but also the quality and efficiency of the castings [2,3].

The Al–Ti–B ternary master alloys have been commonly used as grain refiners for most aluminium alloys [4]. After several decades of research on this field, no clear consensus has

* Corresponding author.

E-mail addresses: amulyapattnaik@gmail.com, amulyapattnaik@ampri.res.in (A.B. Pattnaik).

<http://dx.doi.org/10.1016/j.jmrt.2014.10.017>

2238-7854/© 2014 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. All rights reserved.

Table 1 – Chemical composition of Al5052 alloy.

Element	Si	Fe	Cu	Mn	Mg	Zn	Cr	Al
Wt%	0.25	0.40	0.1	0.1	2.5	0.1	0.15	Rem.

been reached yet on the mechanism of grain refinement in aluminium due to the addition of Al–Ti–B master alloys. Easton and Stjohn [5] classified the mechanism of grain refinement as nucleant and solute paradigms. The nucleant paradigm relates to the heterogeneous nucleation of primary α -Al grains on insoluble substrates, which acts as nucleation sites. The solute paradigm includes the role of solute elements on grain refinement process. Mohanty et al. [6] studied the mechanism of grain refinement in aluminium alloys by directly adding TiB_2 crystals into the aluminium melt. They observed that the TiB_2 particles were found in the grain boundaries and the Ti atoms segregate at TiB_2 /melt interface resulting in the formation of a thin layer of $TiAl_3$. This undergoes a peritectic reaction to form primary α -Al. Johnsson et al. introduced the solute theory to explain the grain refinement of aluminium alloys due to the addition of Al–Ti–B master alloys [7]. They suggested that both nucleants and solutes particles influence the grain refinement. The solute titanium atoms segregates and restricts the growth of nucleant particles thus making available larger number of nucleating sites for nucleation of primary α grains. Though a number of theories has been proposed to explain the grain refinement in aluminium alloys, none of these could clearly explain the exact mechanism.

Acoustic emission (AE) technique is a non-destructive evaluation procedure in which stress waves are generated from within the material due to the dynamic events occurring inside by the application of load [8]. The stress waves are generated by a number of dynamic events like, dislocation motion, inclusion, fracture, phase transformation, etc. [9]. Online monitoring of deformation behaviour has the potential to provide real time information in order to predict the onset of failure by AE parameters variation. Most of the previous studies on deformation of various materials have related the AE activities to the dislocation kinetics [10]. Scruby et al. [11] systematically studied the AE during deformation of aluminium alloys with various microstructures. They observed that the AE activities in the age hardened alloys were mainly due to shearing of Guinier–Preston zones by the dislocations. Jalaj Kumar et al. [12] in their recent investigation on AE during tensile deformation of near alpha titanium alloys identified twinning as a potential source of acoustic emission. They showed that AE signals were generated mainly due to combined effect of twinning and dislocation motion. In our recent work on a low stacking fault energy material (brass), we found that the AE signal intensity increased with increase in twinning density. We found a strong correlation of microstructural features (in terms of twinning density) with that of AE signal intensity [13].

In case of Al–Mg alloys, the presence of magnesium in the solid solution improves the grain refining capability. According to Birch et al. the addition of magnesium improves the wettability of aluminium melt with the nucleating sites by reducing the surface tension [14]. A great number of investigations have been carried out to study the grain refinement in Al–Mg alloys [15,16]. However, no work has been reported so

far to study the grain refinement and its subsequent effect on microstructure, mechanical properties and acoustic emission characteristics of Al5052 aluminium alloys.

The present paper aims to study the effect of Al–5Ti–1B grain refiner on microstructure, mechanical properties and acoustic emission characteristics of Al5052 aluminium alloy in the light of grain refinement.

2. Experimental

Al5052 alloy was prepared using pure aluminium (99%) and magnesium (99.9%) as the starting material. First of all, pure Al was melted in an electrical resistance furnace at 750–800 °C using a 30 kg SiC crucible. Then pure magnesium was added on to the melt. Degassing of the melt was carried out using nitrogen gas for 5 min. After stirring and removal of dross, part of the molten metal (10 kg) was poured into the cast iron moulds to prepare cast fingers and plates of Al5052 alloy. The chemical composition of Al5052 alloy is shown in Table 1. The remaining part of the molten metal was grain refined using Al–5Ti–1B master alloy to produce two different grain refined alloys with 0.2 and 0.5 wt% Al–5Ti–1B respectively.

Microstructural characterizations were carried out using LEICA 5000M optical microscope and NOVA NANO FESEM 430 field emission scanning electron microscope (FESEM) which was equipped with EDX detector. The samples for microstructural characterization were taken from different position of cast ingots. These samples were polished using conventional metallographic techniques and etched with Keller's reagent. The average grain size of the specimens was measured using an image analyzer attached to the optical microscope according to ASTM E 12. The different phases present in the microstructures were identified through X-ray diffraction studies using a Bruker D8 Advance diffractometer (40 mA and 40 kV) with a scanning speed of 2°/min with a CuK_{α} ($\lambda = 1.54 \text{ \AA}$) target. The hardness values of the polished samples were measured using a Universal hardness tester. The average values of five readings for each sample are reported.

Cylindrical tensile specimens of dimensions of 20 mm diameter, 8 mm gauge width and 40 mm gauge length were cut from the cast ingots of both the as cast and grain refined alloys. Tensile tests were carried out on a floor model 8801instron servo hydraulic universal testing machine of 100 kN capacity. These tests were carried out at a strain rate of 10^{-2} s^{-1} . Impact tests of the as cast and grain refined alloys were carried out using an INSTRON make impact testing machine. Charpy V-notch impact specimens with dimension 10 mm \times 10 mm \times 55 mm were prepared according to ASTM A370. The damage mechanisms during plastic deformation were studied using scanning electron microscopic analysis of fracture surfaces.

Acoustic emission testing was carried out using a two channel PCI-DiSP with an AEwin version E2.32 AE system (Physical

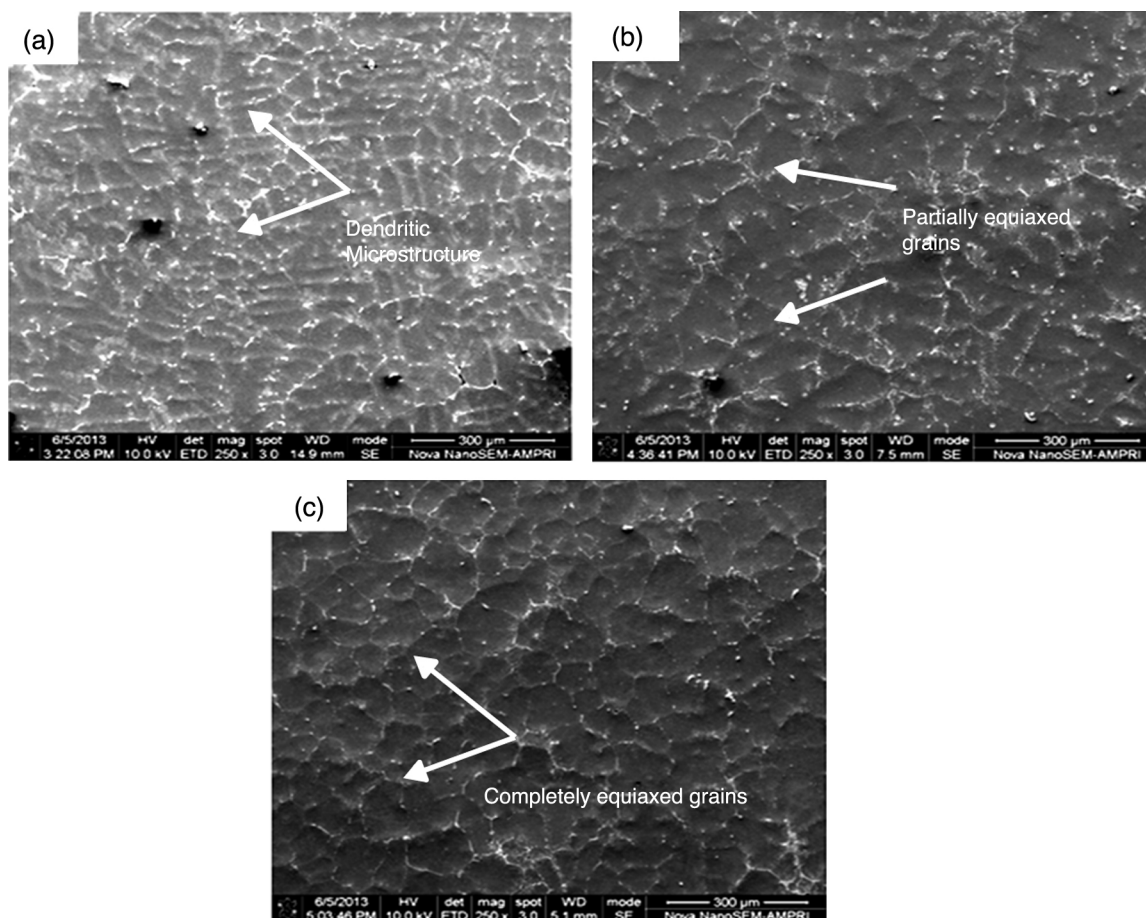


Fig. 1 – FESEM images of (a) unmodified Al5052 alloy (b) modified with 0.2 wt% Al-5Ti-1B (c) modified with 0.5 wt% Al-5Ti-1B.

Acoustic Corporation, Princeton, NJ, USA). AE pulse detection was carried out using a resonant piezoelectric transducer with a peak frequency of 150 kHz. A preamplifier with a gain of 40 dB and a compatible filter (10 kHz–2 MHz) were used to capture the AE signals. A threshold of 45 dB was set to eliminate the background noise by applying the Kaiser effect. The dummy specimens were repeatedly loaded and unloaded below the yielding region to avoid the interference of any noise during the actual experiment. A total system gain of 100 dB was used to capture the AE signals.

3. Results

3.1. Microstructural characterization

The FESEM microstructures of Al5052 aluminium alloy before and after grain refinement is shown in Fig. 1. It is clear from the figure that addition of Al-5Ti-1B master alloy resulted in grain refinement of Al5052 alloys. From Fig. 1a it was found that the microstructure of unrefined Al5052 alloy consists of interconnected and coarse dendritic microstructure. The addition of Al-5Ti-1B to Al5052 alloy resulted in changes in the morphology of the phase from coarse interconnected dendrites to fine equiaxed microstructure with homogenous distribution

of primary α -phase Fig. 1c. It was observed that grain refinement of the melt resulted in an increase in the grain boundary area per unit volume. This ensures the uniform distribution of insoluble substrates in the matrix, which acts as sites for primary α -Al nucleation. Fig. 2 shows the variation of average

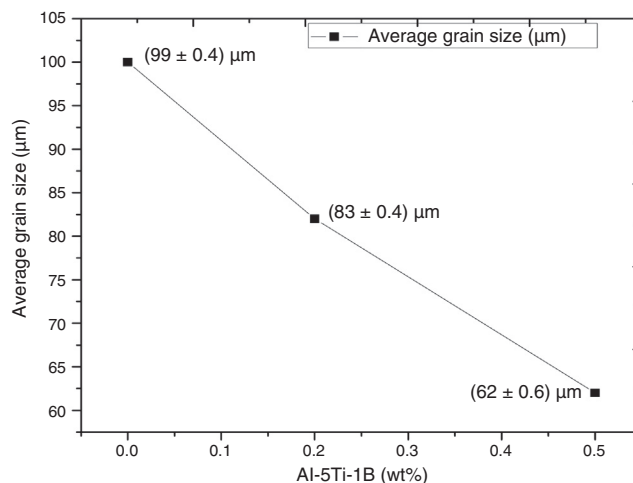


Fig. 2 – The variation of average grain size of the Al5052 alloy with different Al-5Ti-1B contents.

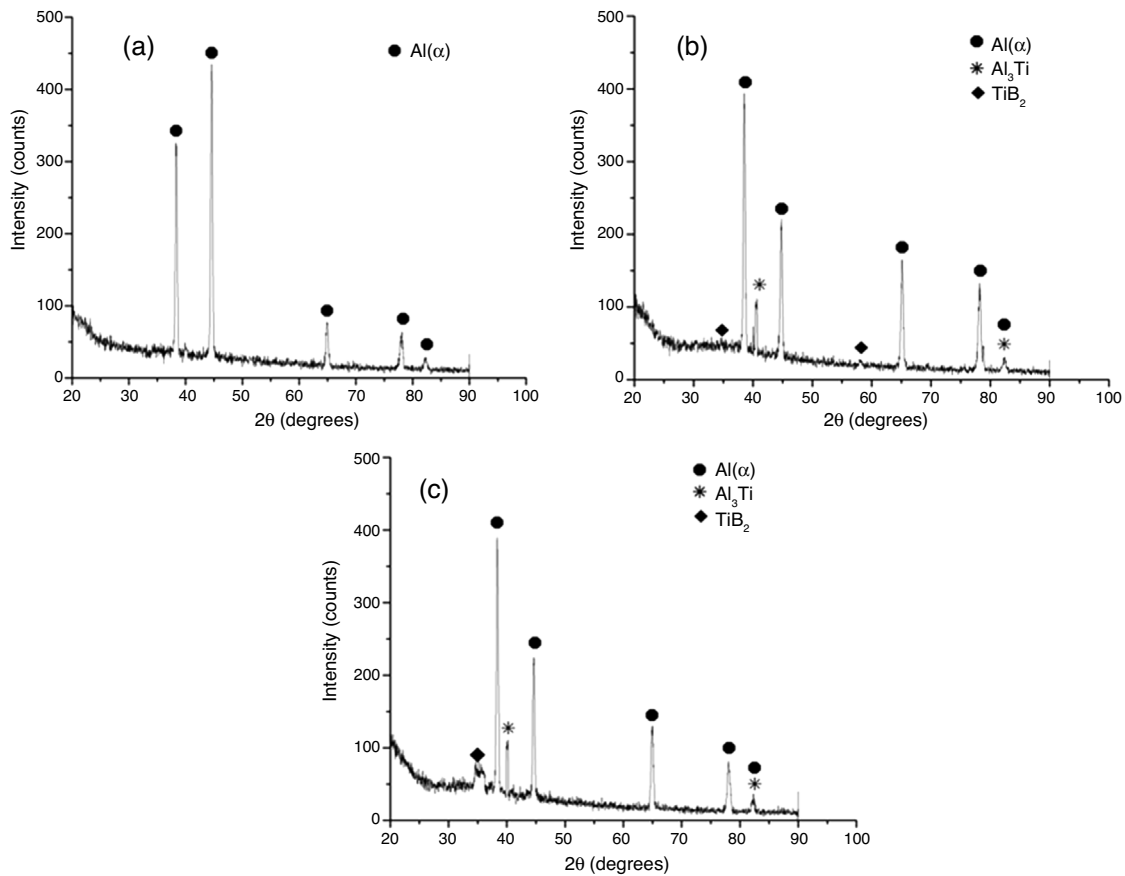


Fig. 3 – XRD plots of (a) unmodified Al5052 alloy (b) modified with 0.2 wt% Al-5Ti-1B (c) modified with 0.5 wt% Al-5Ti-1B.

grain size with addition of Al-5Ti-1B master alloy. From the figure average grain size decreased from 100 μm in unrefined Al5052 alloy to 62 μm in grain refined alloy.

Fig. 3 shows the X-ray diffraction (XRD) plots of unrefined Al5052 and grain refined Al5052 alloys. From Fig. 3a it was observed that the unrefined Al5052 alloy was found to be consisting of single primary α -phase, as magnesium remains in the solid solution and does not form any phase. Fig. 3b and c shows the XRD plots of grain refined Al5052 alloys with 0.2 and 0.5 wt% Al-5Ti-1B respectively. The grain refined alloys were found to be consisting of two recognizable phases i.e. Al_3Ti and TiB_2 . Fig. 4a and b shows the FESEM microstructure and the corresponding EDX spectrum of unrefined Al5052 and 0.5 wt% Al-5Ti-1B grain refined Al5052 alloy. From the EDX spectrum of the grain refined alloy it was observed that Ti segregates at the tip of the primary- α dendrites.

3.2. Hardness, impact toughness and tensile properties

Fig. 5a and b shows the hardness and impact toughness values of unmodified and Al-5Ti-1B modified alloys. The plots in Fig. 5 are the average values of five test specimens made from single castings. The absorbed energy of the unrefined Al5052 alloy was found to be 33J. After the addition of 0.5 wt% Al-5Ti-1B the absorbed energy values increased to 44J. The tensile test results of unmodified and modified Al5052 alloys tested at a

strain rate of 10^{-2} s^{-1} are shown in Table 2. The UTS and elongation values of the alloy increased from 114.7 MPa and 7.8% to 185 MPa and 18% respectively.

3.3. Acoustic emission during tensile deformation

The variation of stress and AE counts with strain of unmodified and Al-5Ti-1B modified Al5052 alloy is shown in Fig. 6. More copious AE signal was found to be generated in the region where material transform from elastic to plastic regime of deformation. Fig. 7 shows the variation of stress and amplitude of AE signals with strain of unmodified and Al-5Ti-1B modified Al5052 alloy. The AE amplitude in the yielding region for unmodified Al5052 alloy lies in the range of 45–65 dB, and the same for Al-5Ti-1B modified alloy was found to be in the range of 45–85 dB. Fig. 8 shows the variation of AE cumulative counts during deformation of unmodified and modified Al5052 alloys. This is a more quantitative way of representing the AE data.

4. Discussion

4.1. Effect of grain refiner on microstructure

The FESEM micrographs, Fig. 1, clearly show that the microstructural morphology changes from coarse interdendritic structure to fine equiaxed microstructure due to the addition

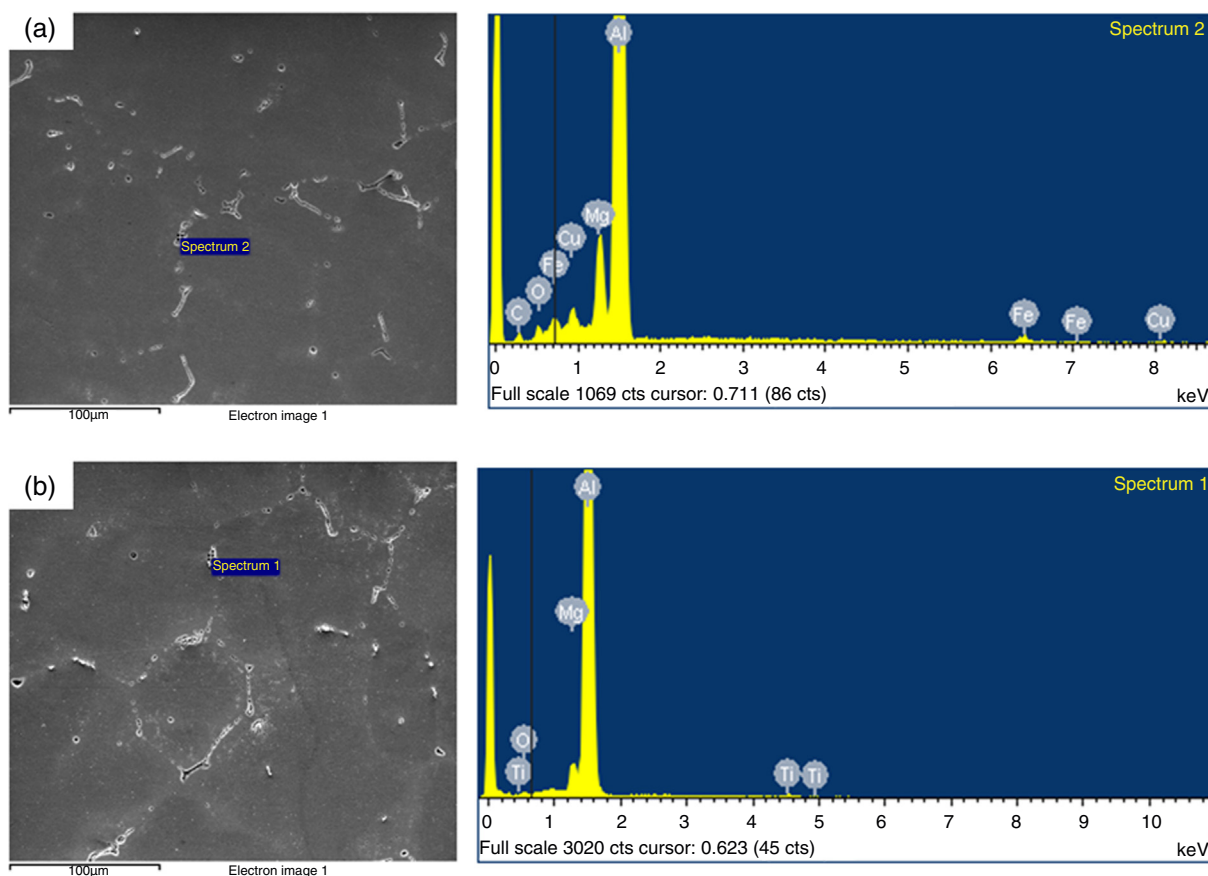


Fig. 4 – FESEM image and corresponding EDX spectrum of (a) unmodified Al5052 alloy (b) modified with 0.5 wt% Al-5Ti-1B.

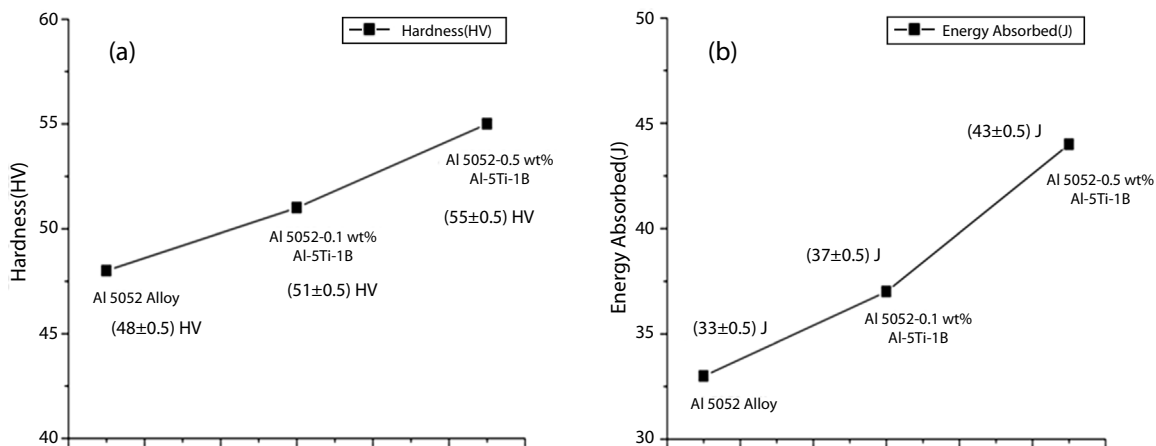


Fig. 5 – Variation of (a) Hardness (b) Impact toughness values with Al-5Ti-1B content.

Table 2 – Tensile test results of unmodified and modified Al5052 alloys tested at a strain rate of 10^{-2} s^{-1} .

Microstructural condition	0.2% offset YS (MPa)	UTS (MPa)	Total elongation (%)
Al5052 alloy (unmodified)	55 ± 0.40	115 ± 0.79	8 ± 0.87
Al5052 – 0.1 wt% Al-5Ti-1B (modified)	58 ± 0.30	160 ± 0.24	14 ± 0.7
Al5052 – 0.5 wt% Al-5Ti-1B (modified)	58 ± 0.40	185 ± 0.92	18 ± 0.5

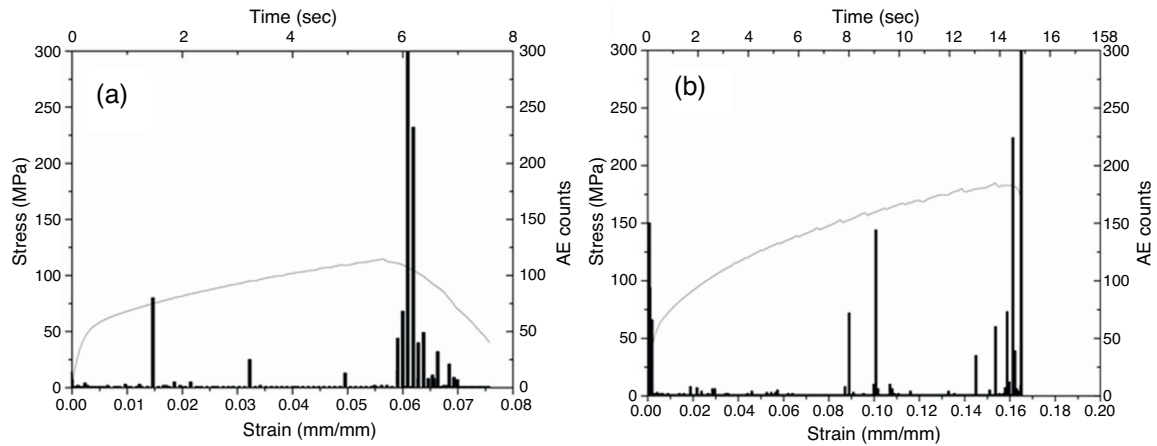


Fig. 6 – Variation of stress and AE counts with strain of (a) unmodified and (b) 0.5 wt% Al-5Ti-1B modified Al5052 alloy.

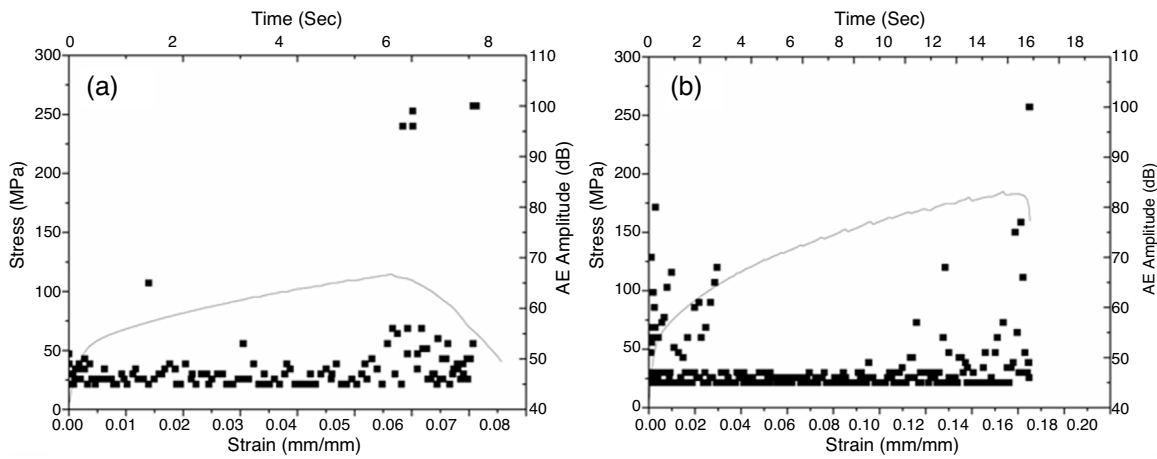


Fig. 7 – Variation of stress and amplitude of AE signals with strain of (a) unmodified and (b) 0.5 wt% Al-5Ti-1B modified Al5052 alloy.

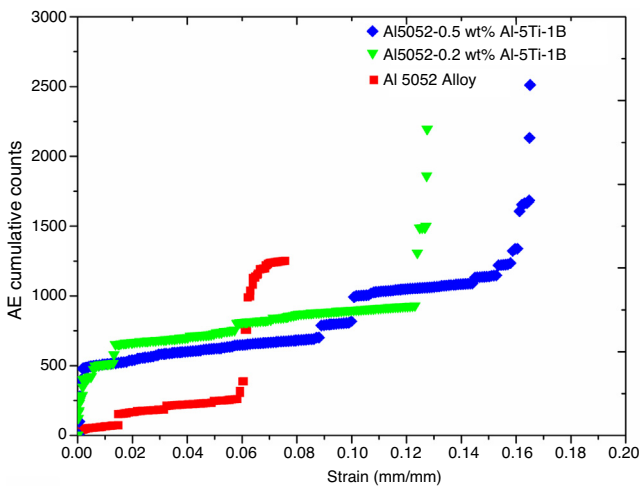


Fig. 8 – Variation of AE cumulative counts during deformation of unmodified and Al-5Ti-1B modified Al5052 alloys.

of Al-5Ti-1B grain refiner. The X-Ray diffraction plots confirm the presence of Al_3Ti and TiB_2 phases, which acts as potential nucleant for primary- α phase nucleation. Several researchers have explained grain refinement in aluminium alloys due to the addition of Al-5Ti-1B master alloy in terms of different theories such as carbide/boride theory [17], phase diagram/peritectic theory [18,19], peritectic hulk theory [20,21], duplex nucleation theory [22,23], and solute theory [7,24]. Cibula et al. [19,24] observed that the use of Al-5Ti-1B as grain refiner, introduces both titanium and boron in to the melt in the form of AlB_2 , TiB_2 and Al_3Ti . They suggested that TiB_2 particles act as insoluble substrates for primary α -phase nucleation. In comparison to TiB_2 , Al_3Ti was found to be a better nucleant mainly due to its good orientation relationship with aluminium [20]. Johnsson and Bakrued proposed the solute theory, which suggested that both addition of solute atoms and nucleant particles are vital for grain refinement of aluminium alloys [7]. The EDX spectrum of Al-5Ti-1B modified alloy confirms the presence of solutal titanium at the tip of the primary- α grain boundary. Thus, the mechanism of grain refinement of Al5052 alloy by the addition of Al-5Ti-1B master alloy can be attributed to the formation of Al_3Ti and

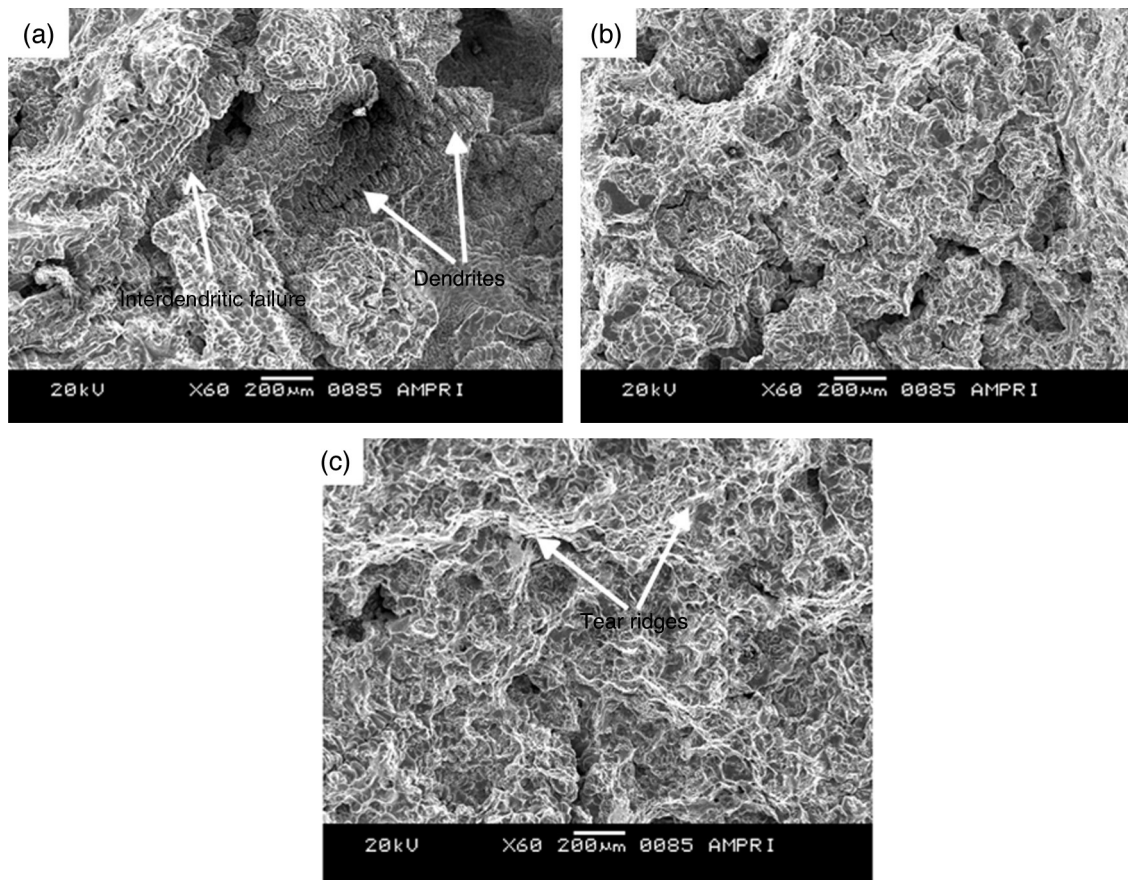


Fig. 9 – SEM micrographs of fracture surfaces of (a) unmodified Al5052 alloy (b) modified with 0.2 wt% Al-5Ti-1B (c) modified with 0.5 wt% Al-5Ti-1B.

TiB₂ phase, which provides more nucleation sites for heterogeneous nucleation [5,21,22]. It can also be attributed to the presence of solutal titanium at the tip of the primary- α dendrites which restricts the growth of primary α -phase, hence providing more nucleation sites [23].

4.2. Effect of grain refiner on mechanical and acoustic emission properties

The hardness and impact toughness values were found to increase with increase in Al-5Ti-1B content, which is mainly attributed to the refinement of grains. The low impact toughness value in unrefined alloy is mainly attributed to the presence of coarse primary- α dendritic structure. The improved impact toughness due to the addition of Al-5Ti-1B master alloy is mainly due to the change in morphology of primary α -phase from coarse dendritic structure to fine equiaxed grains. The energy absorption increases mainly due to the increase in grain boundary area per unit volume. The energy during the dynamic impact test is mainly absorbed by the grain boundaries. Thus, the increase in the grain boundaries increases the energy absorption during impact [23,24]. From the microstructural observation, it is evident that the addition of Al-5Ti-1B to Al5052 alloy resulted in finer grain size and improvement in morphology from coarse dendritic structure to fine equiaxed grains. The reduction in grain size resulted in

improvement in tensile properties of Al5052 alloy. The presence of magnesium in the solid solution and solutal titanium at primary- α grain boundary contributed to the solid solution strengthening of modified Al5052 alloy.

The intensity of AE counts was found to increase with increase in Al-5Ti-1B master alloy addition. Most of the acoustic emission signals were generated in the micro plastic and yielding region of the stress-strain curve. Sudden bursts in the AE signals were detected at the time of fracture. The AE cumulative counts were found to increase with increase in Al-5Ti-1B master alloy addition. The AE signals generated in the micro plastic and yielding stage could be attributed to the generation and motion of dislocations from Frank-Read and grain boundary sources [13,25]. The increase in AE activity (counts and amplitude) due to the addition of Al-5Ti-1B master alloy is mainly attributed to the change in morphology of primary- α grain from coarse dendritic to fine equiaxed structure. This fine equiaxed microstructure increases the grain boundary dislocation interaction which results in higher strengthening with increased AE signal intensity. The presence of solute titanium at the primary- α grain boundaries and the presence of magnesium in the solid solution contributes significantly towards pinning and unpinning of dislocations during deformation thus making it inhomogeneous. This fact has been clearly demonstrated by the presence of serrations in true-true strain curve of these modified alloys. Higher numbers of AE

counts and their large amplitudes could possibly be attributed to inhomogeneous plastic deformation of modified Al5052 alloy with 0.2% and 0.5% Al-5Ti-1B grain refiner. The increase in AE activity in the whole range of deformation could therefore be due the combined effect of dislocation motion, solid solution strengthening and inhomogeneous plastic deformation.

4.3. Fracture surface observation

Fig. 9a shows the fracture surface of the unrefined Al5052 alloy associated with large voids and macro cracks resulting in interdendritic failure, which corresponds to brittle mode of fracture. The coarse dendritic structure acts as a source of stress concentration and crack initiation. Fig. 9b and c illustrates the fracture surfaces of the alloy with 0.2 and 0.5 wt% Al-5Ti-1B respectively. The fracture surfaces were found to be associated with fine dimples or tear ridges, which correspond to ductile mode failure. The grain refinement and the improvement in the morphology of the primary α -phase due to the addition of Al-5Ti-1B grain refiner results in a transition of fracture surface from brittle to ductile mode of failure.

5. Conclusions

In the present work, the effect of Al-5Ti-1B grain refiner on microstructure, mechanical properties and acoustic emission characteristics of Al5052 alloy was studied. The following conclusions can be drawn based on the experimental results.

- (1) The addition of Al-5Ti-1B master alloy reduced the grain size of Al5052 alloy from 100 μm to 62 μm .
- (2) The morphology of primary α -phase changes from coarse dendritic structure to fine equiaxed grains which is attributed to the grain refinement due to the addition of Al-5Ti-1B and segregation of Ti at the tip of primary α dendrites, which restricts the growth of primary α grains.
- (3) The mechanical properties of Al5052 alloy were improved by the addition of Al-5Ti-1B master alloy. The ultimate tensile strength and elongation values were increased from 114 MPa and 7.8% to 185 MPa and 18% respectively.
- (4) The increase in AE activity in the whole range of deformation is mainly attributed to the combined effect of dislocation motion, solid solution strengthening and inhomogeneous plastic deformation.
- (5) Fracture surface analysis of both unmodified and Al-5Ti-1B modified alloy showed a transition from brittle to ductile mode of failure.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

The authors would like to thank Director, CSIR-Advanced Materials and Processes Research Institute, Bhopal, India for his constant encouragement and support. The authors would

also like to acknowledge the financial support provided by CSIR under the project ESC 0101.

REFERENCES

- [1] Carle D, Blount G. The suitability of aluminium as an alternative material for car bodies. *Mater Des* 1999;20:267-72.
- [2] Murty BS, Kori SA, Chakraborty M. Grain refinement of aluminium and its alloys by heterogeneous nucleation and alloying. *Int Mater Rev* 2002;47:3-29.
- [3] Cibula A. Mechanism of grain refinement of sand castings in aluminium alloys. *Inst Metals J* 1949;76:321-60.
- [4] Easton MA, Stjohn DH. A model of grain refinement incorporating alloy constitution and potency of heterogeneous nucleant particles. *Acta Mater* 2001;49:1867-78.
- [5] Easton MA, Stjohn DH. Grain refinement of aluminum alloys: Part I. the nucleant and solute paradigms—a review of the literature. *Metall Mater Trans A* 1999;30-34:1613-23.
- [6] Mohanty PS, Gruzleski JE. Mechanism of grain refinement of aluminium. *Acta Metall* 1995;43:2001-12.
- [7] Johnsson M, Backerud L, Sigworth GK. Study of mechanism of grain refinement of aluminium after addition of Ti- and B-containing master alloys. *Metall Trans A* 1993;24A:481-91.
- [8] Liptai RG, Harris DO, Tatro CA. An introduction to acoustic emission. In: *Acoustic Emission*. ASTM STP 505; 1972. p. 3-10.
- [9] Beattie AG. Acoustic emission principles and instrumentation. *J Acoust Emission* 1983;2:95-128.
- [10] Schofield BH. Research on sources and characteristics of acoustic emission. In: *Acoustic Emission*. ASTM STP 505. American Society for Testing and Materials; 1972. p. 11-9.
- [11] Scruby CB, Wadley HNG, Rusbridge K, Stockham-Jones D. Influence of microstructure on acoustic emission during deformation of aluminium alloys. *Metal Sci* 1981;15:599-608.
- [12] Kumar J, Punnose S, Mukhopadhyay CK, Jayakumar T, Kumar V. Acoustic emission during tensile deformation of smooth and notched specimens of near alpha titanium alloys. *Res Non-destruct Eval* 2012;23:17-31.
- [13] Pattnaik AB, Jha BB, Sahoo R. Effect of strain rate on acoustic emission during tensile deformation of α -brass. *Mater Sci Technol* 2013;29:294-9.
- [14] Birch MEJ, Fisher P. Mechanism of fade in grain refining of aluminium with titanium boron aluminium. *Solidification processing*. London: The Institute of Metals; 1987. p. 500-2.
- [15] Backerud L, Gustafon P, Jhonsson M. Grain refinement mechanism of aluminium as a result of addition of titanium and boron. *Aluminium* 1991;67:910-5.
- [16] Mohanty PS, Samuel FH, Gruzleski JE, Kosto TK. Studies on the mechanism of grain refinement in aluminium. *Light Metals* 1994;103:9-1045.
- [17] Mohanty PS, Guthrie RIL, Gruzleski JE. Studies on addition of inclusions to molten aluminium using a novel technique. *Metall Mater Trans B* 1995;26B:103-10.
- [18] Johnsson M. On the mechanism of grain refinement of aluminium after additions of Ti and B. In: Das SK, editor. *Light metals*. 1993. p. 769-77.
- [19] Cibula A. Discussion of the mechanisms of grain refinement in dilute aluminium alloys. *Metall Trans* 1972;3:751-3.
- [20] Davies IG, Denies JM, Hellawell A. The nucleation of aluminium grains in alloys of aluminium with titanium and boron. *Metall Trans* 1970;1:275-80.
- [21] Emamy M, Daman AR, Taghiabadi R, Mahmudi M. Effects of Zr, Ti and B on structure and tensile properties of Al-10Mg alloy (A350). *Int J Cast Metals Res* 2004;17:1-17.
- [22] Kumar GSV, Murthy BS, Chakraborty M. Development of Al-Ti-C grain refiner and study of their grain refining

-
- efficiency on Al and Al-7Si alloy. *J Alloys Compds* 2005;396:143-50.
- [23] Easton M, Stjohn D. Grain refinement of aluminium alloys: Part II. Confirmation of, and a mechanism for, the solute paradigm. *Metall Mater Trans A* 1999;30:1625.
- [24] Cibula A. Grain refinement of Al-alloy casting by addition of titanium and boron. *Inst Metals J* 1951-1952;80: 1-16.
- [25] Dieter George E. *Mechanical metallurgy*. SI Metric ed. Tata MacGraw Hill; 1928.