

INFLUENCE OF MECHANICAL ALLOYING AND LEAD CONTENT ON MICROSTRUCTURE, HARDNESS AND TRIBOLOGICAL BEHAVIOR OF 6061 ALUMINIUM ALLOYS

M. Paidpilli*, K. Verma, R. Pandey and A. Upadhyaya

* maheshpaidpilli@gmail.com

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Department of Materials Science and Engineering, Indian Institute of Technology Kanpur, India.

Abstract: In the present work, one batch of prealloyed 6061Al powder was processed by mixing and another one was ball milled with varying amount of lead content (0-15 vol. %). These powders were compacted at 300MPa and sintered at 590°C under N₂. The instrumented hardness and the young's modulus of as-sintered 6061Al-Pb alloys were examined as a function of lead content and processing route. The wear test under dry sliding condition has been performed at varying loads (10-40 N) using pin-on-disc tribometer. The microstructure and worn surfaces have been investigated using SEM to evaluate the change in topographical features due to mechanical alloying and lead content. The mechanically alloyed materials showed improved wear characteristics as compared to as-mixed counterpart alloys. Delamination of 6061Al-Pb alloys decreases up to an optimum lead composition in both as-mixed and ball-milled 6061Al-Pb alloys. The results indicated minimum wear rate for as-mixed and ball-milled 6061Al alloy at 5 and 10 vol. % Pb, respectively.

Keywords: 6061Al-Pb alloys, Mechanical Alloying, Microstructure, Wear and Young Modulus.

1. INTRODUCTION

In recent years, aluminium lead alloys have been widely used as bearing materials in automobile industry [1]. Aluminium contribute strength, low density and corrosion resistance while lead provides good thermal conductivity and ductility. In addition, lead enhances wear resistance by forming lead rich film and yield lubrication between the sliding surfaces [2, 3]. The thickness, composition and properties of the lubricating film formed during sliding are largely unknown [4]. The manufacturing of Al-Pb alloys through traditional casting route is problematic due to the following: Firstly, lead segregation occur during solidification. Secondly, no mutual solid solubility between Al and Pb alloy. Thirdly, wide miscibility gap exist in liquid state. Finally, high density difference between the constituent phase [5, 6]. To overcome the above drawbacks, several other techniques have been developed such as rapid solidification [7], stir casting [8] and powder metallurgy [9] to produce Al-Pb alloy. However, powder metallurgy have advantage over other technique due to near net shape processing, tailoring of composition and microstructure.

Powder metallurgy route includes mixing, pressing and sintering to consolidate the 6061Al and lead powder. The mixing of powder is a crucial stage as it determines the porosity level and homogenization of the Al-Pb system, which influence the tribological properties of 6061Al-Pb alloys [4]. Zhu et al. [10, 11] have reported that mechanical alloying of high density difference powders resulted homogeneous microstructure. More recently, Sastry and Ranga [12] concluded that attrition milling has enhanced the densification of Al-Pb alloy.

Zhang and Alpas [13] described wear mechanism with respect to load. They reported that initially at lower load, formation of iron-oxide layer took place and as load increases delamination starts and finally at higher load severe damage was observed. Wear occurred due to adhesion, abrasion and delamination mechanisms are directly depends on parameters such as normal load, sliding distance and sliding velocity [4]. Elsewhere, Wang and Rack [14] investigated the tribological response of metal matrix composite with respect to applied load, sliding distance, sliding velocity and material properties. They reported that wear rate abruptly increased with respect to these parameters. In

another study, Gurcan and Baker [15] demonstrated the effect of volume fraction of reinforced particle on wear behaviour of 6061Al alloy and reported improved wear resistance with volume fraction of particles. The relationship between wear rate and hardness is difficult to derive. Wear test performed under lubricated condition exhibited enhancement of wear resistance with increase in hardness. Whereas, non-lubricated sliding condition showed opposite behaviour [13, 16]. Albert et al. [17] observed the effect of mechanical alloying on Al-Pb alloy and reported wear resistance has improved with addition of 10 wt. % Pb. An et al. [3] documented better wear and friction characteristic for Al-Si-Pb alloy with increase in Pb content. Similar results were also reported by Zhu et al. [5] and showed improved tribological behaviour by mechanical alloying.

To the best of our knowledge, wear behaviour of prealloyed 6061Al-Pb system fabricated by conventional press and sinter route was rarely documented. In the present work, 6061Al-Pb alloys were obtained through ball-milling and mixing route with unique microstructure. This investigation examined the effect of lead content, processing route and applied load on the wear rate of 6061Al-Pb alloys. Moreover a direct comparison between as-mixed and ball-milled 6061Al-Pb alloy consolidated using persistent liquid phase sintering is made on the basis of microstructure, hardness, wear rate and lead content.

2. EXPERIMENTAL PROCEDURES

2. 1. Material Preparation

Prealloyed 6061Al powder (supplier: Cegedur Pechiney; France) was mixed with lead powder (supplier: Sigma-Aldrich) in different compositions (5, 10, 15 Vol. %). Another set of same composition powder were prepared by ball milling in a planetary mill (speed: 300 rpm, time: 5h). The ball to powder ratio was kept 10:1. The powders prepared by both processing route were compacted at 300MPa and sintered inside tube furnace under N₂ atmosphere at temperature 590°C for 1h. To prevent oxidation, the sintering

was performed in nitrogen atmosphere.

2. 2. Microstructure

Metallographic examination was carried out on sintered samples by polishing with SiC paper of varying grid size (320, 500, 1000, and 1600). Thereafter, the samples were polished using an alumina suspension (1µm, 0.3µm, 0.05µm) and final polishing was performed with 0.04µm colloidal silica. The specimens were characterized using scanning electron microscope (QUANTA 200, FEI, The Netherlands).

2. 3. Hardness and Wear Test

The instrumented indentation was conducted on all sintered specimens using Vickers diamond indenter (CSM instruments). The maximum load applied was 5N with 10s dwelling time. The instrumented hardness and young's modulus was calculated using Oliver and Pharr method [18]. In all specimens five indentations were made and the average value reported. Dry sliding wear tests was performed on the specimens with varying amount of load (10-40N) using pin-on-disc type wear testing machine (DUCOM Triboinnovators; Bangalore). Before each test, surface of sample was polished with 1000 grit size SiC paper. The surfaces of steel disk was cleaned before each run. The cylindrical specimens (radius: 8mm) was fixed at a radius of 50 mm from the centre of steel disk (EN32 Grade; Rockwell Hardness: 65 HRC). The sliding speed and distance are 0.7m/s and 2000m, respectively. According to ASTM Standard G99, wear rate was calculated using the following Eq. (1):

$$\text{Wear rate}(\text{mm}^3/\text{m}) = \frac{\text{Weight loss}}{\text{Sinter density} \times \text{Sliding distance}} \quad (1)$$

where, the weight loss is the difference between the initial weight of pin and its weight after wear test. The weight was measured in a microbalance (model: AG245, Germany) with an accuracy of $\pm 0.0001\text{g}$. The sinter density of the compacts was measured using Archimedes technique. The worn

surfaces were characterized using scanning electron microscope (SEM).

3. RESULTS AND DISCUSSION

3. 1. Microstructural Analysis

Fig. 1(a-d) shows the effect of lead content on the microstructure of sintered as-mixed Al-6061 compacts. The bright phase in the SEM photomicrograph represent lead, which is non-uniformly distributed along the equiaxial Al grains. Since, Pb has no-mutual solubility with Al, during sintering lead segregate at liquid/vapor interface, which decreases the melt surface tension [19]. Consequently, wetting angle reduced and assist spreading of melt along the grain boundaries. The dark phase in Fig. 1(a) along the grain boundaries of 6061Al alloy was further characterized using EDS and results indicate presence of Mg, Si and O. This can be

attributed to the processing of prealloyed 6061 Al powder through atomization where supersaturated solid solution of Mg/Si formed and later on sintering, Mg₂Si precipitate along the grain boundaries [20, 21]. Fig. 2(a-d) shows the microstructure of ball-milled 6061Al-Pb compacts with varying lead content. It is interesting to note that microstructures of ball-milled 6061Al are very different than its as-mixed counterpart alloy. The microstructure illustrates that ball-milling results in elongation of Al grain and also imparts more uniform distribution of lead. However, there is accumulation of lead at higher lead content alloys as shown in Fig. 2(d). These signify that ball-milling is very effective for homogeneous distribution of second phase material during sintering.

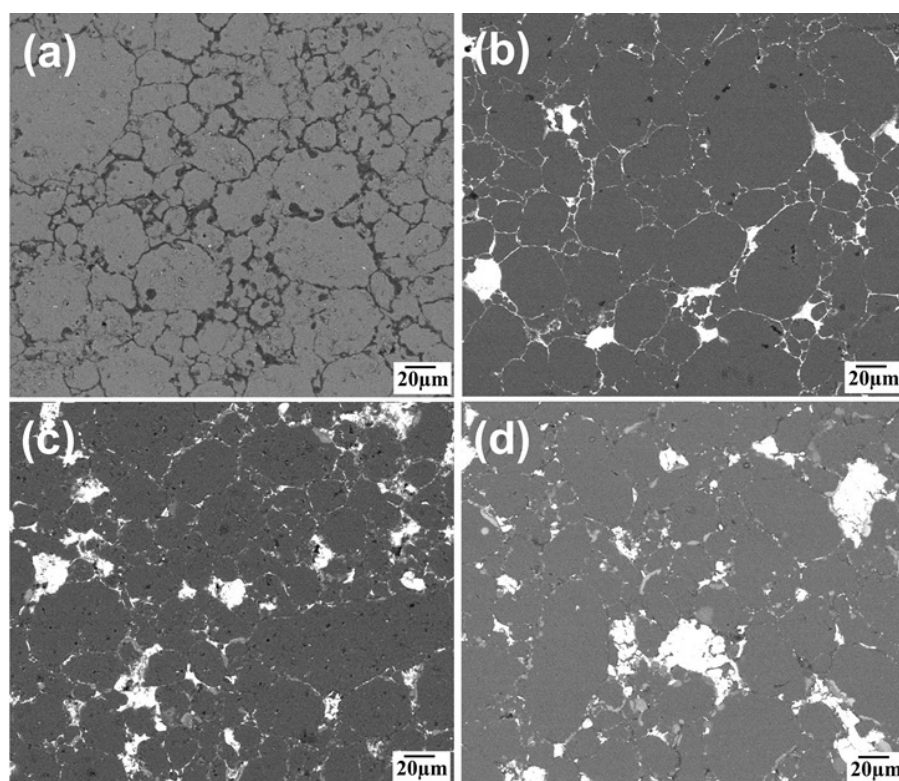


Fig. 1. SEM photomicrographs of as-mixed (a) 6061Al, (b) 6061Al-5Pb, (c) 6061Al-10Pb and (d) 6061Al-15Pb.

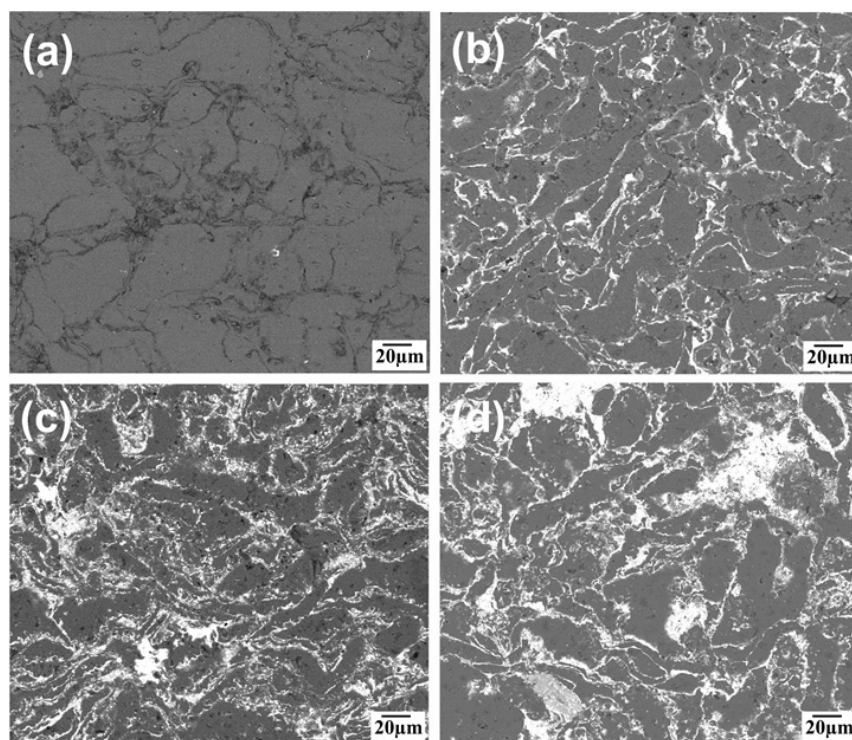


Fig. 2. SEM photomicrographs of ball-milled (a) 6061Al, (b) 6061Al-5Pb, (c) 6061Al-10Pb and (d) 6061Al-15Pb.

3. 2. Hardness

The relationship between load, indentation depths, ball-milling and lead content were shown in Fig. 3(a). The effect of lead content and ball-

milling on indentation depth of 6061Al alloy were illustrated in Fig. 3(b). Depending on the lead content and processing technique to consolidate 6061Al-Pb alloys the indentation depth and the hardness value varies from ~20μm

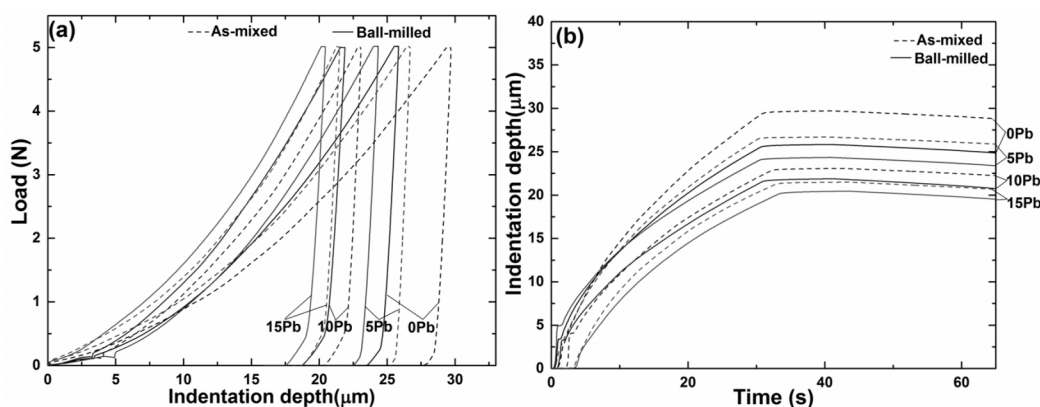


Fig. 3. Effect of lead content and processing condition on the relationship between: (a) load versus indentation depth and (b) indentation depth versus time profile for sintered 6061Al alloy compacts.

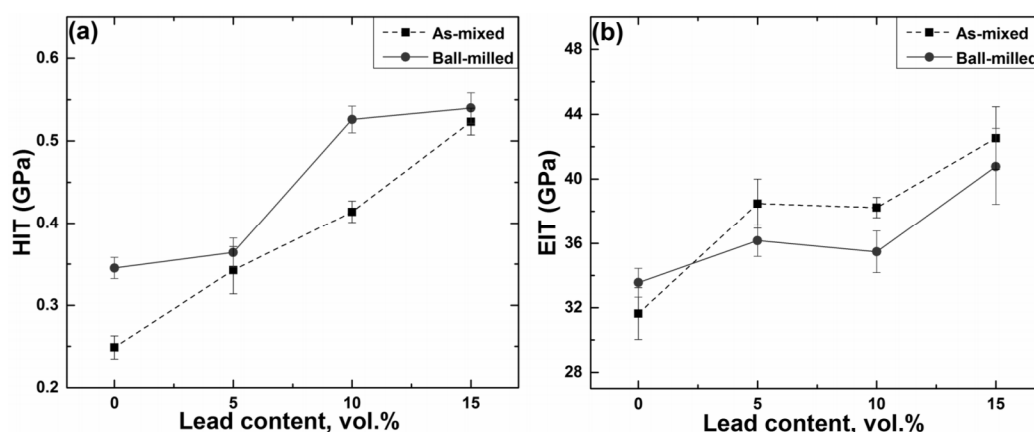


Fig. 4. Effect of varying lead content on the (a) Hardness and (b) Young modulus of sintered 6061Al alloys.

to $\sim 30\mu\text{m}$ and from ~ 0.25 GPa to ~ 0.55 GPa, respectively. The measured instrumented hardness value as a function of as-mixed and ball-milled 6061Al-Pb alloys is presented in Fig. 4(a). This figure illustrated that the hardness of as-mixed 6061Al alloys increases with increase in lead content. Sheng et al. have reported that gliding dislocation shear the lead particle and constraint plastic deformation to improve hardness of Al-Pb alloy [22]. Similar behavior was observed for ball-milled 6061Al-Pb alloys. However, ball-milled 6061Al-Pb alloy have higher hardness value as compare to their counter as-mixed alloys. This behavior attributed to structure refinement, unique microstructure and uniform distribution of Pb along the Al grain [23]. Fig. 4(b) shows the effect of lead content and processing route on young's modulus of as-sintered 6061Al alloy. The change in young's modulus shows similar trend as hardness with respect to increase in lead content. However, with lead addition the ball-milled 6061Al alloy has lower young's modulus value as compared to their counter as-mixed 6061Al alloy. The reason could be the presence of thick Pb layer along the elongated Al grain as shown in Fig. 2, which significantly influences the measurement of young's modulus.

3. 3. Effect of Load and Lead Content on Coefficient of Friction

Fig. 5 shows the variation of coefficient of friction (COF) as a function of sliding distance for both as-mixed and ball-milled 6061Al alloy with varying amount of lead content at loads 20N and 40N. It is worth to note that both as-mixed as well as ball-milled alloys showed decrease in COF with increase in applied load. Few researchers correlated these behavior with Dahl effect where asperities undergo elastic deformation and act like spring to reduce the friction [24, 25]. Fig. 6 represents the effect of varying Pb content on the average COF value for compacts that were subjected to pin-on-disc test at loads 20N and 40N. It was observed that average COF value decreases with increase in lead content at 20N load. The lead particulate dispersed between the specimen and sliding disc to reduce the coefficient of friction. Furthermore, at higher load the plotted graph showed steeper decrease up to 5 vol. % Pb content and thereafter represent negligible effect on coefficient of friction. This behavior attributes to the combine effect of higher load and lead content. Since, increase in lead content increases the area fraction of weak aluminium lead interface while higher load increases the contact area between sliding surface and specimen, which weakens the worn surface strength and leads to delamination.

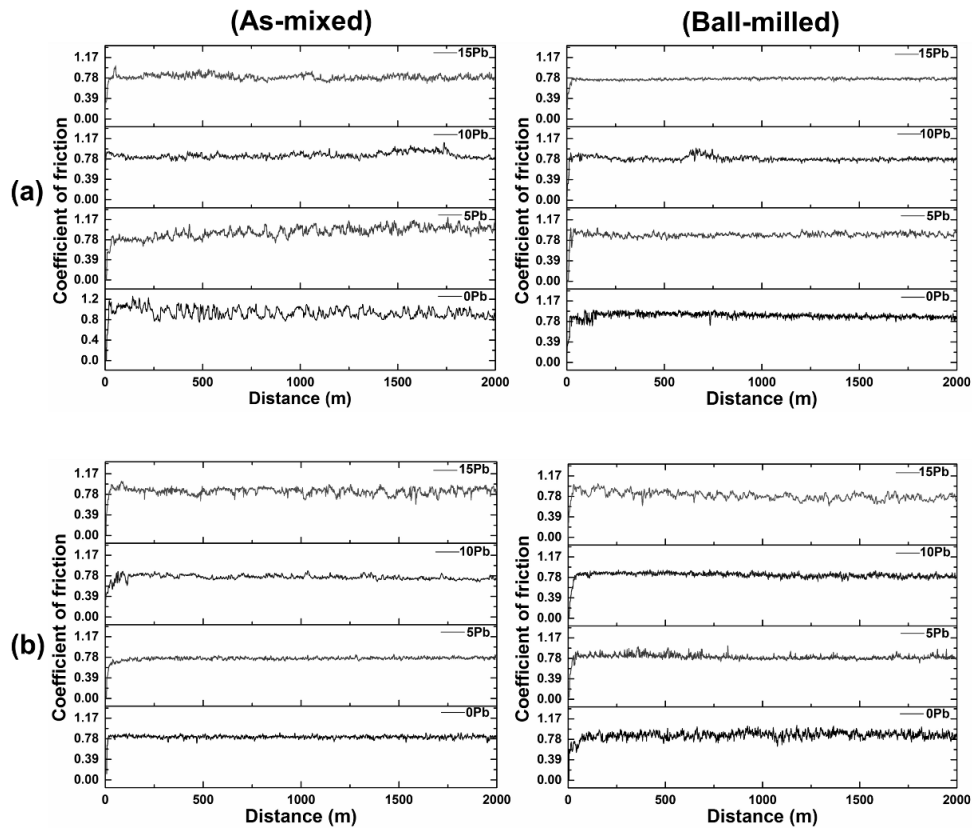


Fig. 5. The variation of coefficient of friction with sliding distance of as-mixed (left) and ball-milled (right) 6061Al-Pb compacts subjected to pin-on-disc wear test at (a) 20N and (b) 40N load, respectively.

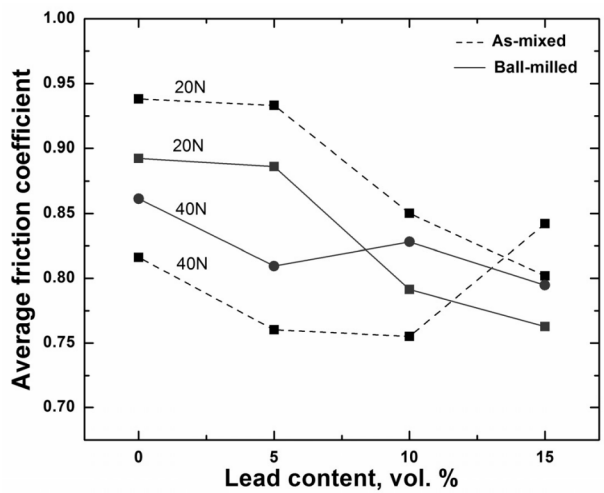


Fig. 6. Effect of lead content and processing condition (as-mixed and ball-milled) on the coefficient of friction for sintered 6061Al-Pb compacts tested at 20N and 40N.

In contrast, the as-mixed 6061Al-15Pb alloy at 40N load showed greater COF value than base alloy. As shown in Fig. 1(d) the microstructure of as-mixed 6061Al-15Pb showed bulk accumulation of Pb that easily delaminate and causes severe erosion.

3. 4. Effect of Lead Content and Applied Load on Volume Loss

The variation of volume loss with respect to lead content under various applied loads for both as-mixed and ball-milled 6061Al alloys is graphically demonstrated in Fig. 7(a) and 7(b), respectively. The addition of lead has improved the wear resistance in both as-mixed and ball-milled material. The reason can be attributed to the formation of mechanical mixed layer between the sliding disc and specimen. Since, mechanical mixed layer is composed of Fe, Pb and oxide, which provide prevention against wear [3]. However, higher lead content reduces resistance to wear properties. The increase in lead content promote the growth of lead/matrix interface, which facilitate crack and voids nucleation due to lower tensile strength of lead/matrix interface. The graphical plot illustrated that volume loss of as-mixed materials was lowest at 5 vol. % Pb. As shown in the microstructure of as-mixed 6061Al-Pb alloy, lead accumulation started beyond 5 vol. % Pb, which adversely effected the wear

resistance properties. In contrast, ball-milled materials exhibit lowest volume loss at 10 vol. % Pb due to uniform distribution of Pb around the grain boundaries. From the above results, it can be concluded that ball-milled materials have superior wear resistance as compared to their counter as-mixed materials. Since, high energy ball-milling has enhanced the strength of sintered compact and distributed second phase particle uniformly. Also it is important to note that volume loss depends on number of variables such as microstructure, hardness and grain size [26].

3. 5. Effect of Milling and Applied Load on Wear Rate

To understand the effect of milling on tribological properties of as-mixed and ball-milled 6061Al-Pb alloys, a comparative analysis has been made for each composition with increasing load and shown in Fig. 8(a-d). The figure illustrated that wear rate increases with increase in load for both type of 6061Al-Pb alloy. In addition, at lower load the difference in wear rate between as-mixed and ball-milled based alloys was negligible. However, at higher load as-mixed 6061Al-Pb alloys have regime of severe wear whereas ball-milled 6061Al-Pb alloys have mild wear. This signifies that ball-milling suppress the transition to severe wear regime and impeded the transition load to higher

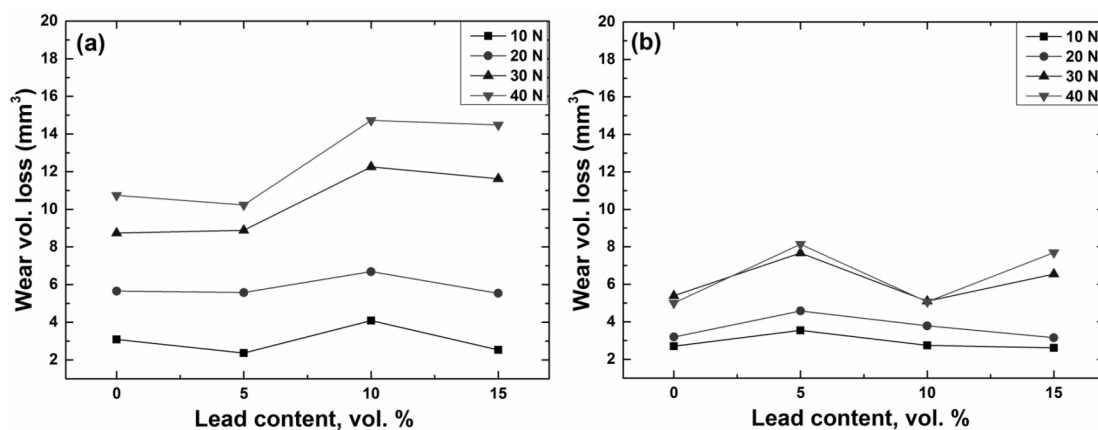


Fig. 7. Effect of varying lead content on the wear volume loss of 6061Al-Pb alloys prepared by (a) mixing and (b) ball-milling. The as-sintered compacts were subjected to wear test at various loads (10-40N).

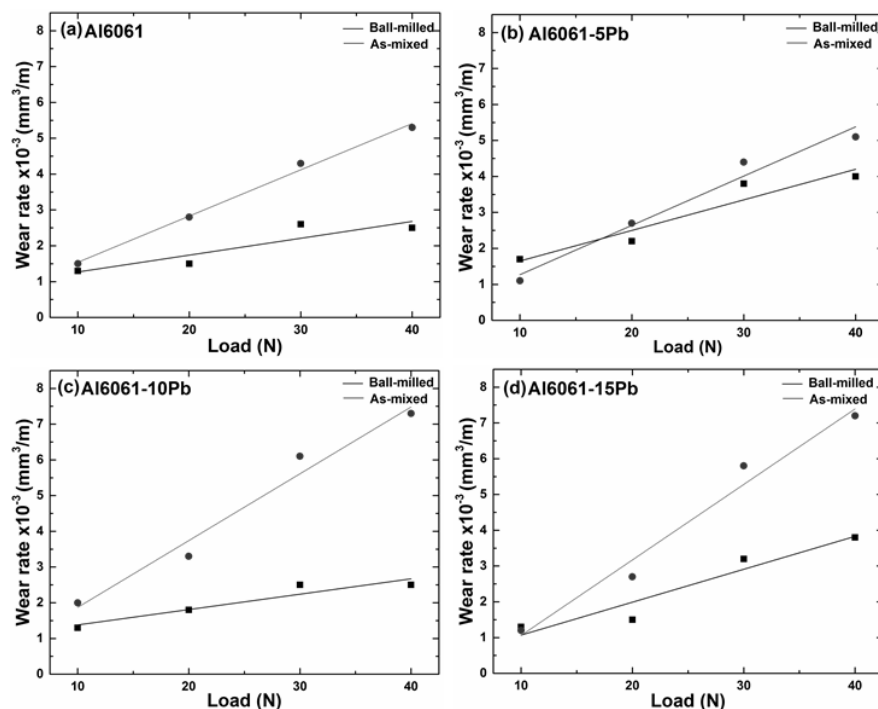


Fig. 8. Effect of varying load on the wear rate of as-mixed and ball-milled (a) 6061Al alloy and 6061Al-Pb compacts containing (b) 5, (c) 10 and (d) 15 vol. % lead.

value. Hence, mechanical alloying technique has improved the wear resistance of 6061Al-Pb alloys at a particular Pb content as compare to their counter as-mixed based alloys. Furthermore, ball-milled 6061Al-Pb alloys have thin elongated grain with uniform distribution of Pb along the Al grain boundaries to strengthen the wear resistance properties. Whereas, 6061Al-Pb alloys have coarser rounded grain and non-uniformly distributed lead, which undermine the strength of worn surfaces.

3. 6. Worn Surface Characterization

The worn surface topography of as-mixed 6061Al alloy with varying amount of Pb content was shown in Fig. 9(a-d). A comparative observation of wear out scars in the photomicrograph exhibited that they become finer with lead addition. However, at higher lead volume fraction the worn surfaces consist of deep ploughing, coarser debris and severe erosion. During wear, direct contact between the

specimen and sliding disc reduces due to formation of mechanical mixed layer. In case of 6061Al-Pb alloy, mechanical mixed layer composed of lead, which imparts lubrication and thus decreases coefficient of friction. Fig. 9(d) clearly depicted that as-mixed 6061Al-15Pb have severe erosion and coarser debris. This attribute to increase in accumulation of lead as shown in Fig. 1(d), which squeezed out of matrix and weakened the strength of worn surfaces. Similarly, the worn surface topography of ball-milled 6061Al with varying amount of Pb content is shown in Fig. 10(a-d). It clearly demonstrated that with increase in Pb content the smeared layer of Pb, oxide and Fe becomes thicker and denser. Fig. 10(b-d) illustrated that an optimum amount of lead content is required to form continuous film of smeared layer.

To better understand the effect of lead addition on wear mechanism the debris formed during wear were analyzed. Wear debris generated on the ball-milled 6061Al and 6061Al-10Pb compact surface were shown in Fig. 11(a) and

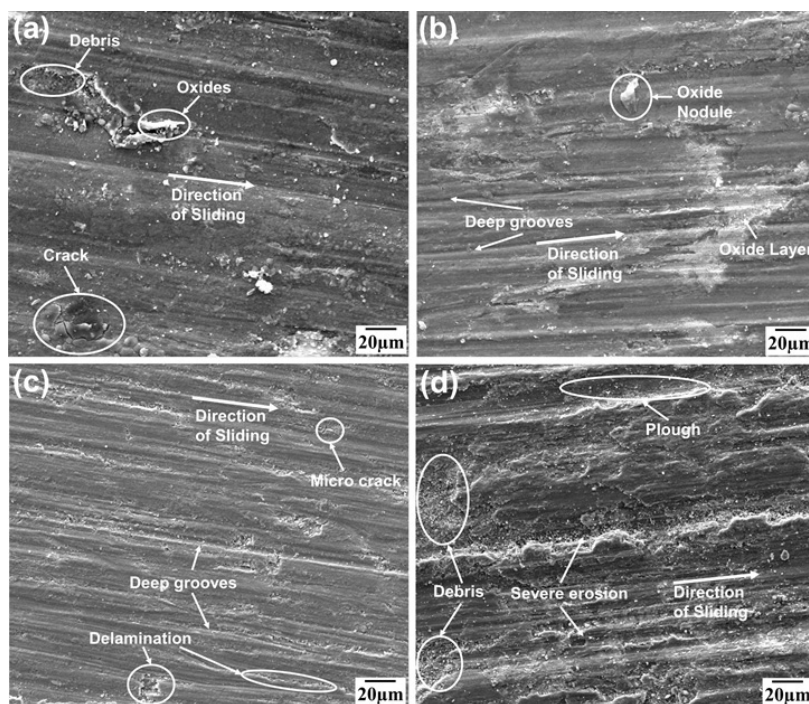


Fig. 9. SEM images of worn surfaces of as-mixed (a) 6061Al alloy and 6061Al-Pb compacts with (b) 5, (c) 10 and (d) 15 vol. % lead. The compacts are sintered at 590°C and subjected to pin-on-disc wear test at 30N load.

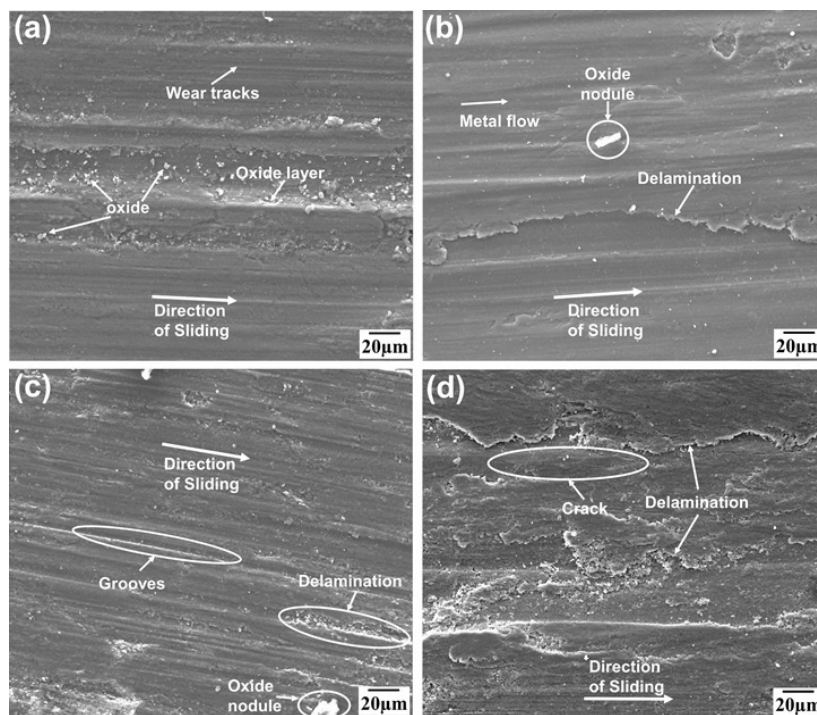


Fig. 10. SEM photomicrograph of worn surfaces of ball-milled (a) 6061Al alloy and 6061Al-Pb compacts with (b) 5, (c) 10 and (d) 15 vol. % lead, all compacts were tested at 30N load.

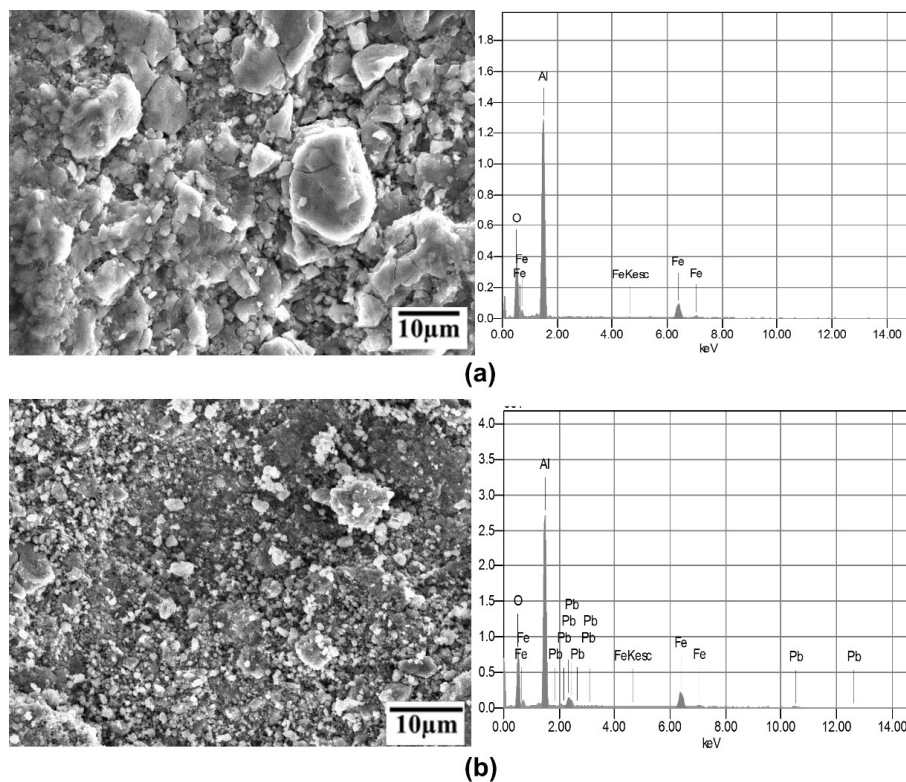


Fig. 11. Morphology of the worn debris and the corresponding EDS spectrum for ball-milled (a) 6061Al and 6061Al-10Pb compacts.

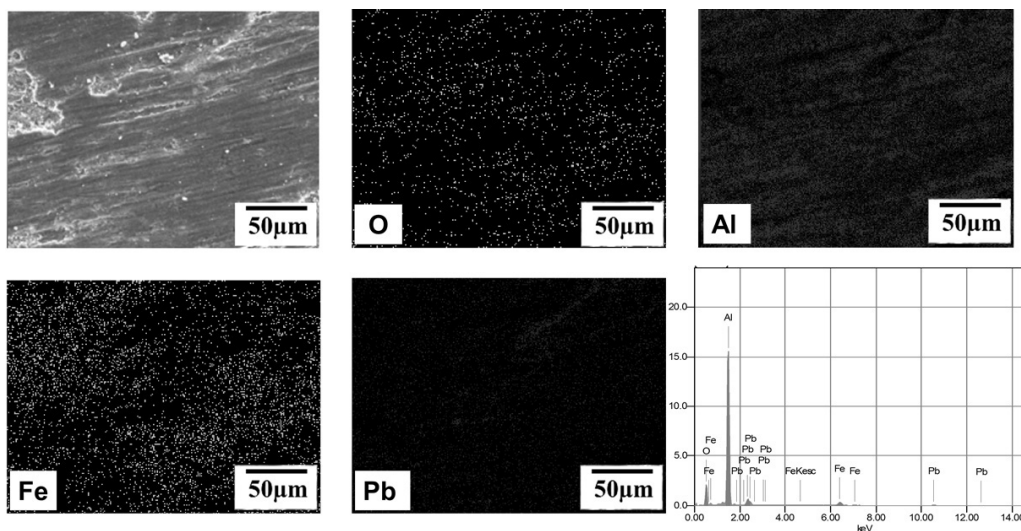


Fig. 12. EDS spectrum and distribution of alloy constituents on the worn surfaces of ball milled 6061Al-Pb alloy subjected to 30N load.

11(b), respectively. As shown in Fig. 11(a) the ball-milled 6061Al alloy have coarser agglomerated debris of few micrometer. On the other hand, with lead addition the debris were loosely agglomerated with fine particle of 2 to 5 μm size. Since, addition of lead reduces the probability of direct contact between disc and specimen. The worn debris generated on ball-milled 6061Al specimen surface under 30N load consists of Fe, oxide particle and metal sheets. During wear test metal sheets were generated through processes such as cracking, delamination and decohesion of adhesion bond [27]. Fig. 12 illustrates the elemental mapping analysis of worn surface of ball-milled 6061Al-5Pb alloy subjected to 30N load. The presence of Fe element on the worn surface indicated occurrence of adhesion phenomenon during wear. Beside that oxygen was also found on the worn surface, which causes due to oxidation of aluminium alloy during wear test [28]. These oxide particles causes abrasive wear against the soft aluminium matrix. The worn surface characterization has determined that wear mechanism for 6061Al-Pb alloys are composed of abrasion, adhesion and delamination. Other investigators were reported similar mechanisms for aluminium based alloy [29].

4. CONCLUSIONS

The influence of processing method, lead content and applied load on volume loss, wear rate and topographical features of worn surfaces were discussed in detail. The microstructure of ball-milled 6061Al-Pb alloys exhibited uniform distribution of lead along the grain boundaries as compare to as-mixed 6061Al-Pb alloys. The hardness and modulus of 6061Al-Pb alloy increases with increase in Pb content. The ball-milled based alloys have shown higher hardness value as compare to as-mixed counterpart alloy at a particular Pb content. In both as-mixed and ball-milled materials the wear rate increases with increase in load. The mechanical alloying of 6061Al-Pb alloy resulted considerable improvement in wear resistance. At higher load ball-milled 6061Al-Pb alloys were able to sustain tribo-layer but as-mixed 6061Al-Pb alloys underwent delamination. The wear rate of

6061Al-Pb alloys decreases with increase in Pb content up to an optimum composition above, which it further increases. The lowest wear rate was observed at 5 and 10 vol. %Pb for as-mixed and ball-milled 6061Al-Pb alloys, respectively.

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