Effect of Zr addition on the mechanical characteristics and wear resistance of Al grain refined by Ti after extrusion

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Abstract. Aluminum and its alloys are normally grain refined by Ti or Ti+B to transfer their columnar structure during solidification into equiaxed one which improves their mechanical behavior and surface quality. In this paper, the effect of addition of Zr on the metallurgical, and mechanical aspects, hardness, ductility and wear resistance of commercially pure aluminum grain refined by Ti after extrusion is investigated. Zr was added at a level of 0.1% which corresponds to the peretectic limit at the Al-Zr phase diagram. The experimental work was carried out on the specimens after direct extrusion. It was found that addition of Ti resulted in decrease of Al grain size, whereas addition of Zr alone or in the presence of Ti, resulted in reduction of Al grain size. This led to increase of Al hardness. The effect of the addition of Ti or Zr alone resulted almost in the same enhancement of Al mechanical characteristics. As for the strain hardening index,n, increase was obtained when Zr was added alone or in the presence of Ti. Hence pronounced improvement of its formability. Regarding the effect of Zr addition on the wear resistance of aluminum; it was found that at small loads and speeds addition of Ti or Zr or both together resulted in deterioration of its wear resistance whereas at higher loads and speeds resulted in pronounced improvement of its wear resistance. Finally, the available Archard model and the other available models which consider only the mass loss failed to describe the wear mechanism of Al and its micro-alloys because they do not consider the mushrooming effect at the worn end.

1. Introduction

Aluminum and its alloys are widely used materials in aircraft and automobile industries due to their attractive properties particularly their high strength-to-weight ratio, electrical and thermal conductivities in addition to their corrosion resistance, [1]. Unfortunately they solidify in columnar structure which tends to deteriorate their mechanical strength and surface quality. Therefore, they are normally alloyed or micro alloyed with other elements; e.g. aluminum–silicon (Al–Si) alloys find their applications in the automotive industry due to their high specific strength and wear resistance. The combination of high thermal conductivity and low coefficient of thermal expansion make them suitable for the internal combustion (IC) engine components such as cylinder block and piston. Sabatino and Arnberg [2] demonstrated that the addition of fluidity of the Al-Si alloys giving the good cast ability. Moreover, improvement in the tensile strength [3] and tribological performance [4] of Al–Si alloys with increase in the silicon content of the alloys was also observed. Morphology of the

eutectic silicon in the hypoeutectic Al–Si alloy was effectively modified by chemical route typically with the impurity level addition of elements such as sodium 1.1[5], strontium [6], phosphorus [7] and scandium [8] by a process known as impurity induced twinning mechanism. However, in hyper-eutectic Al–Si alloys, with the presence of large primary silicon crystals, the modification and refinement by the aforementioned method is not effective. On the contrary, size and morphology of the silicon phase in Al–Si alloys was effectively refined by rapid solidification techniques such as spray forming and powder metallurgy. For instance, Cuietal [9] demonstrated that the primary silicon phase in Al–Si alloys can be effectively refined by spray forming. However, the presence of hard silicon phase which exists in the form of large grains, morethan100 microns inconventionally cast Al–Si alloyseutecticand primary silicon crystals in hypoeutectic and hypereutectic Al–Si alloys respectively is not recommended as it adverselyaffectstheir mechanical properties. Further information is given in references [10,11]. Another method of grain refining the structure of these alloys is by the addition of some grain refiners to their melt prior to solidification, mainly by addition of either titanium or titanium-boron. Several binary Al-Ti and Ternary Al-Ti-B master alloys are produced for this purpose and are now commercially available. Although the literature on the grain refinement in general and aluminum and its alloys in particular is voluminous and reviewed in reference [12]. The mechanism of grain refinement is still a controversial matter despite the different suggested reported models. Summary of these models is reported in [12-13]. Wear is an important phenomenon in machine elements and manufacturing processes, defined as the cumulative mass loss in the machine elements, cutting tools, punches and dies which leads to in change in their dimensions and causing their failure. The mechanism of wear is very complex due to the number of variables involved. Some of these variables is related to the material characteristics e.g. microstructure, mechanical strength, hardness and surface finish. Other variables include process parameters e.g. applied load, velocity, interface temperature, lubricant, corrosion of the surface and existence of foreign particles. The available literature on wear reveals that most of the published work is concerned with the effect of the above mentioned parameters on wear mechanism of aluminum and little work is published regarding the effect of addition of one or more grain refiners on its wear resistance both in the cast and after extrusion conditions.

In this paper, the effect of addition of Zr on the mechanical characteristics and wear resistance of commercially pure aluminum grain refined by Ti after direct extrusion is investigated.

1.1 Theoretical considerations

Most of the existing wear theories and mechanisms attempt to explain the wear process on a microscopic level, and relate the magnitude of wear to material properties and to the nature of the local involved parameters. The first mechanism relates the wear rate to the number of inert-atomic encounters between the opposing surfaces [14]. However, this mechanism has not been generally accepted. Later, attempts have been made to establish a new wear theory based the atomic level of wear, but this idea is still far from simple to be accepted, because the wear debris is generated simultaneously with the onset of motion, and the debris consists of aggregated particles of materials. The old reported theory is the most widely accepted theory which is used to predict the volume of the worn material from the following equation:

$$V = KPL/3H \qquad (1)$$

Where V is the worn volume, P is the applied load, L is the sliding distance, and H is the material hardness and K is a constant, which was interpreted as the probability that a particular asperity contact could produce a wear particle which may be considered as a wear rate, [15]. Archad's theory although it gives indication in some cases of adhesive wear, it fails in other cases, particularly in determining the wear of aluminum and other soft materials like high conductivity copper, where in addition to the accumulated mass loss, there exists some plastic deformation at the worn end causing a mushroom like shape. This phenomenon was repeatedly reported in the literature,[12-13]. In the last two references

the authors suggested a model which accounts for plastic deformation at the worn end and the shearing of the particles which accounts for the mass loss or the volume of the removed material.

2. Materials equipment and experimental procedures

2.1 Materials

Commercially pure aluminum of 99.8 % purity of the chemical composition shown in Table 1, was used throughout this work A binary Al-2.92 wt.% Ti and a binary Al-1.17 wt.% Zr master alloys were laboratory prepared and used to obtain 0.15 wt.% Ti and 0.1 wt.% Zr in the different alloys. Master and grain refined alloys were prepared in graphite crucibles and graphite rods were used for stirring the molten bath in all experiments.

Table 1. Chemical composition (wt. %) of commercially pure aluminium

Element	Fe	Si	Cu	Mg	Ti	V	Zn	Mn	Na	Al
Wt. %	0.09	0.05	0.005	0.004	0.004	0.008	0.005	0.001	0.005	Bal.

2.2 Equipment

The extrusion tests were carried out at room temperature, in cold condition, using the extrusion die shown in Figure 1, it consists essentially of a container made of steel D2 having an external diameter of 100 mm, internal diameter of 14 mm and 120 mm height. The punch is 13.96 mm diameter and 80 mm length made of D2 steel. The female die is circular of 10 mm internal diameter and 80 mm external diameter. Extrusion tests were carried out using the extrusion die shown in Figure 1.

1	Punch
2	Punch Holder
3	Die Holder
4	Dummy
5	Die
6	Backer
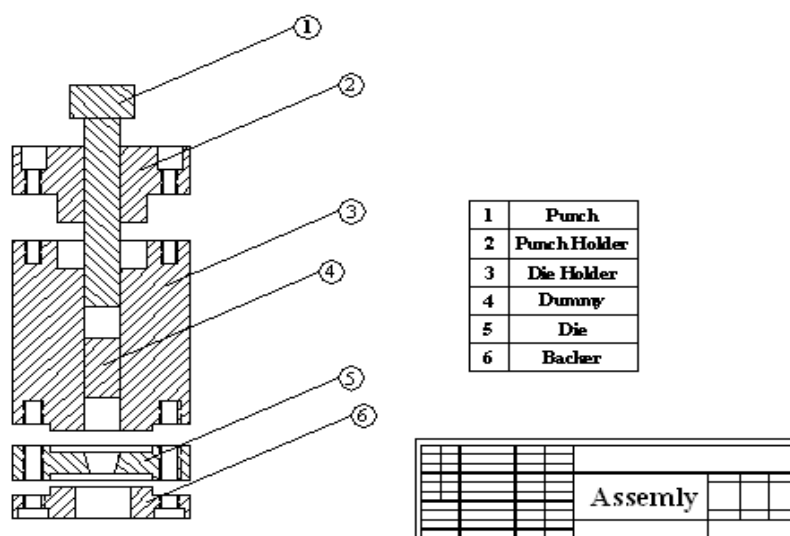
Figure 1. Extrusion die assembly

2.3 Experimental procedures

### 2.3.1 Preparing the binary master alloys and the micro-alloys

The experimental procedures started by preparing the two binary master alloys Al-Ti and Al-Zr. In each case titanium or zirconium was added into molten aluminum at 1100 C and stirred under a cry lite flux. The temperature was kept constant for half an hour before casting the alloy in a plate form of thickness less than 8 mm by spreading the melt over a thick cast iron plate. These master alloys were used in preparing the different Al micro-alloys, namely: Al-Ti, Al-Zr and Al-Ti-Zr. Standard tensile specimens were machined from Al and its prepared micro-alloys. Standard tensile tests were carried

out on the Instron Universal Testing machine of 100 KN capacity at cross head speed of 5 mm/min. and the autographic record was obtained for each specimen from which themechanical behavior was determined.The wear tests were carried outusing the standard wear testing apparatus, (pin on rotating test) at different loads namely: 5, 10, 15, and 20 N and at different linear speeds, namely: 0.276, 0.801, and 3.01 m/sec. All the above mentioned tests were carried out on specimens both in the cast and after extrusion conditions.

3. Results and discussion
3.1 Effect of Ti and Zr on the microstructure of aluminum after extrusion
Figure 2 shows the effect of the extrusion process on the grain size of Al and its three micro-alloys which indicates that it resulted in pronounced decrease in grain size, for example it resulted in a decrease of 254.54%, 41%, and 51.3% in the case of Al-Ti, Al-Zr and Al-Ti-Zr micro-alloys respectively. The effect of Ti or Zr addition to Al either alone or together on its grain size after extrusion is explicitly shown in the photomicrographs of Figure 3.
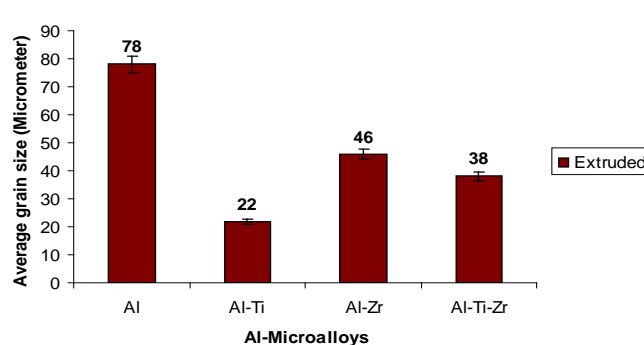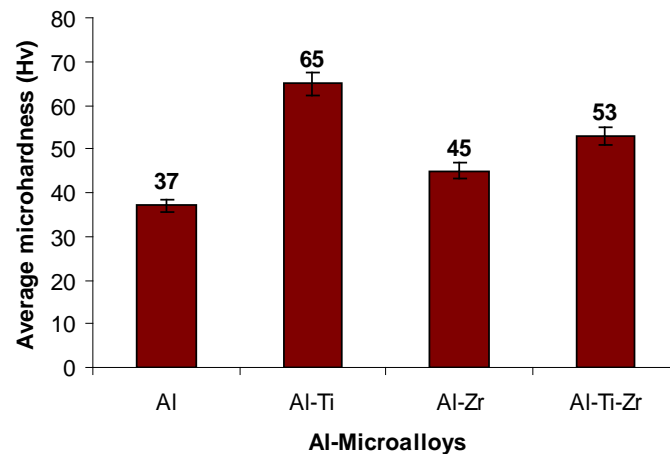

Figure 2. Effect of zirconium addition on the grain size of Aland Al-Ti after extrusion

a) Pure Al (78 µm)

b) Al-Ti (22 µm)

c) Al-Zr (46 µm)

d) Al-Ti-Zr (38 µm)
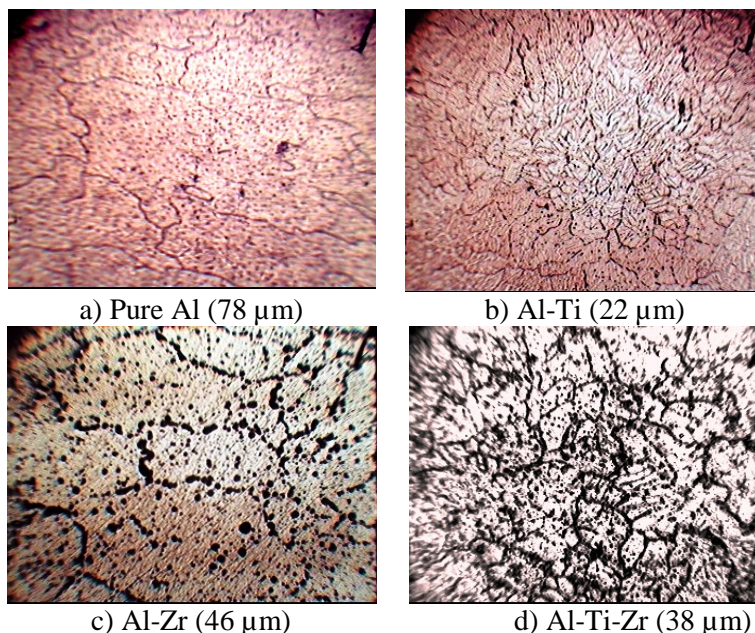
Figure 3. Photomicrographs showing the general microstructure of Al and its micro alloys after extrusion at x250

*3.2 Effect of Ti and Zr addition on the hardness of aluminum after extrusion*
The histogram of Figure 4 shows the Vickers's micro-hardness of the Al and its micro-alloys Al-Ti, Al-Zr and Al-Ti-Zr after extrusion from which it can be seen that the extrusion process resulted in increase of the micro-alloy's hardness by the addition of Ti or Zr either alone or together by the following percentages: 75.68, 21.6 and 43.24 respectively. This is attributed to the grain refinement of the structure and the intermetallic compounds which exists in the main matrix.

Figure 4. Effect of zirconium addition on the average
micro-hardness of Al, Al grain refined by Ti after extrusion

3.3 Effect of Ti and Zr on the Mechanical Characteristics of Aluminum after Extrusion
Figure 5 shows the effect of Zr addition on the mechanical behavior of Al and Al grain refined by Ti. It can be seen from this figure that addition of Ti or Zr either alone or together to Al resulted in enhancement of its mechanical strength. The best improvement was achieved when they were added both together. Furthermore, it can be seen from Table 2 the effect on its mechanical characteristics, where an increase of 34.34 % was achieved in case of adding Ti or Zr alone and 7.37 % when added together in the flow stress at 20 % strain. Similarly, the strength coefficient was increased by 76.2 % when Ti or Zr was added alone and by 19.7% when added together. Regarding the effect on the strain hardening index ,n, it was highly increased by 154.5 % when Ti or Zr were added alone and 63.63 % when added together. This will result in enhancement of its formability; hence it reduces the number of stages required for forming at large strains.
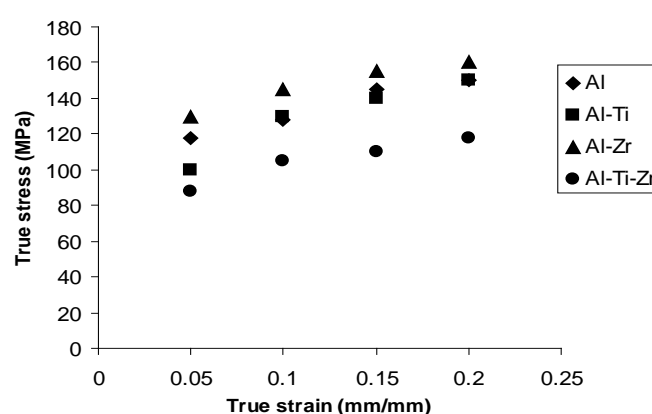

Figure 5. True stress- True strain curves of Al and its three micro-alloys after extrusion

Table 2. Mechanical characteristics of Al and its different micro-alloys after extrusion

Micro alloys	Flow stress (MPa)at strain=20%	Strain hardening index (n)	Strength coefficient (K) MPa	General equation of mechanical behavior
Al	99	0.11	118.6	$\sigma=118.6\varepsilon^{0.11}$
Al-Ti	133	0.28	209	$\sigma=209\varepsilon^{0.28}$
Al-Zr	133.4	0.28	209.3	$\sigma=209.3\varepsilon^{0.28}$
Al-Ti-Zr	106.3	0.18	142	$\sigma=142\,\varepsilon^{0.18}$

4.5 Effect of Zirconium addition on the wear resistance of aluminum and aluminum grain refined by titanium after extrusion

It can be seen from Figure 6 that the relationship between the accumulated mass loss and time for Al and its micro-alloys Al-Ti, Al-Zr, and Al-Ti-Zr at loads of 10 and 20 N and speed of 0.27m/s is almost linear.The commercially pure aluminum indicated less wear resistance than its micro-alloys, i.e. addition of Ti or Zr either alone or together to Al resulted in improvement of its wear resistance. Furthermore doubling the load to (20 N) at the same speed (0.27m/s) did not change the trend but the refiners became more effective, as illustrated by Figure 9 and Tables 3 and 4. It is worth noting that the wear rate, K, in the last column of Table 3 as calculated from Archad theoretical model is misleading because it indicates less value for Al than the K of its micro-alloys. However, the values of the reduction in height and the final area of the worn end which are the indicators of the mushrooming effect reveals that only addition of titanium resulted in reducing Al wear resistance. Therefore it is concluded that investigating the wear resistance to wear of soft materials like those in this paper should consider both the mass loss and the mushrooming effect at the worn end.
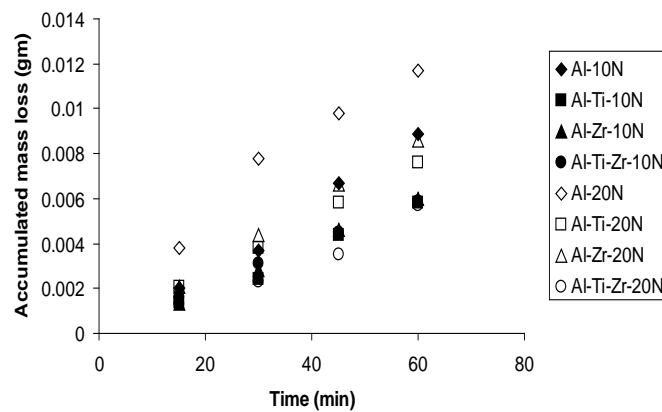
Figure 6. Effect of zirconium addition of Al and Al grain refined by Ti afterextrusion at (S = 0.27m/sec, loads = 10, 20 N)
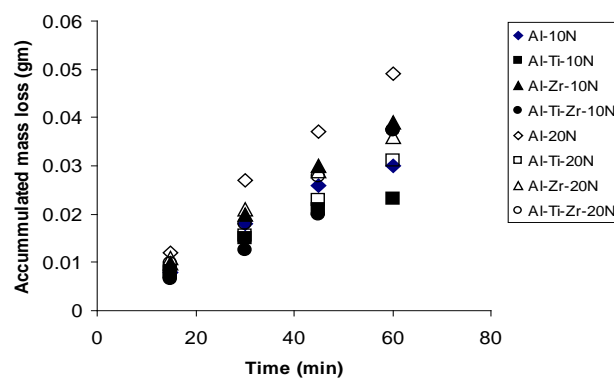
Table 3. Mass loss and dimensional changes after one hr. wear for Zr addition to Al and Al grain refined by Ti after extrusion. (Load = 10 N, S = 0.27 m/sec, A_o = 38.48 mm^2)

Name	Mass loss (g)	D_f (mm)	Δh (mm)	A_f (mm^2)	K Wear rate
Al	0.0089	7.80	0.18	47.76	0.03764
Al-Ti	0.0058	7.90	0.10	48.99	0.04309
Al-Zr	0.006	7.35	0.16	42.41	0.03086
Al-Ti-Zr	0.0059	7.35	0.19	42.41	0.03574

Table 4. Mass loss and dimensional changes after one hr. wear for Zr addition to Al and Al grain refined by Ti after extrusion.(load =20 N, S = 0.27 m/sec, A_o = 38.48 mm^2)

Name	Mass loss (g)	D_f (mm)	Δh (mm)	A_f (mm^2)	K Wear rate
Al	0.049	7.90	0.15	48.99	0.1036
Al-Ti	0.0306	8.05	0.57	50.87	0.1136
Al-Zr	0.0364	7.50	0.58	44.16	0.0936
Al-Ti-Zr	0.0373	7.80	0.60	47.76	0.1130

Increasing the speed to 3m/s and at 10 and 20 N, the mass loss versus time for Al and its micro-alloys is shown in Figure 7, and the mushrooming parameters are explicitly shown in Tables 6 and 7, from which it is seen that the trend remained as before, except the addition of Ti has also resulted in enhancement of the wear resistance and all of them became more effective.

Figure 7. Effect of zirconium addition of Al and Al grain refined by Ti after extrusion at (S = 3m/sec, loads = 10, 20 N)

Table 6. Mass loss and dimensional changes after one hr. wear for Zr addition to Al and Al grain refined by Ti after extrusion. (Load = 10 N, S =3.01 m/sec, A_o = 38.48 mm^2)

Name	Mass loss (g)	D_f (mm)	Δh (mm)	A_f (mm^2)	K Wear rate
Al	0.0244	8.35	0.55	57.39	0.0093
Al-Ti	0.0429	8.20	0.56	52.78	0.0286
Al-Zr	0.0194	8.35	0.50	57.39	0.6525
Al-Ti-Zr	0.0213	8.95	0.51	62.88	0.0116

Table 7. Mass loss and Dimensional Changes after one hr. Wear for Zr addition to Al and Al Grain Refined by Ti after Extrusion.(load =20 N, S = 3.01 m/sec, A_o = 38.48 mm^2)

Name	Mass loss (g)	D_f (mm)	Δh (mm)	A_f (mm^2)	K Wear rate
Al	0.0117	11.5	0.22	103.82	0.0022
Al-Ti	0.0076	10.0	0.20	78.50	0.0025
Al-Zr	0.0086	9.55	0.20	71.59	0.0020
Al-Ti-Zr	0.0057	8.90	0.14	62.18	0.0015

4. Conclusions

The following points are concluded:

i.   Addition of either Ti or Zr alone or together to Al resulted in grain refining of its structure. The best refinement is when Ti is added alone.

ii.   Addition of Ti or Zr alone or together to Al caused improvement of its mechanical strength represented by increase of its flow stress, strength coefficient, ultimate tensile strength, its ductility and its strain hardening index. The last two items cause improvement of formability. This in turn will reduce the stages in the forming processes when large strains are involved. Also its hardness was increased. This is a great achievement because the increase in hardness is normally associated with decrease of ductility and formability.

iii.   The Archad wear model cannot be used to explain the wear of Al and its micro-alloys due to the mushrooming effect at the worn end. This will lead to change of the part dimensions, which in some cases it can be more damaging than the mass loss.

5. Acknowledgment

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