# DEFORMATION RESPONSES OF EXTRUDED ZINC ALLOY

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## *ABSTRACT*

*Severe plastic deformation (SPD) is a technique used to obtain ultra-fine grains (UFGs). However, this technology is expensive and time-consuming. In this study, the conventional deformation technique is used to examine the deformation responses of extruded zinc alloy to achieve fine grain structure by varying die entry angles. Zinc alloy samples were cast into cylindrical billets in sand mould, the samples were machined and tapered at the edge to ease entry into the die for the extrusion process. The die and tools materials were made of mild steel with die entry angles of 15$^o$, 30$^o$, 45$^o$, 60$^o$, 75$^o$, and 90$^o$. Prior to extrusion, the samples were annealed at 400°C because of their hexagonal close-packed (HCP) structure to ease deformation. After annealing, the plastic deformation of alloy samples occurred at 350°C. The extrusion was performed with the aid of a hydraulic press machine that applied pressure on a cylindrical punch and forced the billet through the extrusion die. The results show that the extruded zinc alloy has the highest extrusion stress of 192MPa at 45° die entry angle with fine grains neatly distributed in the matrix and at a 60° die entry angle, the tensile strength and hardness are 177MPa and 95HV respectively with improved ductility. The microstructure of zinc alloy shows equally distributed dendrite-like second-phase intermetallic in the matrix.*

***Keywords: Annealing, Deformation, Dies, Extrusion, Grain Boundary, Intermetallic***

## 1.0 INTRODUCTION

Extrusion is a manufacturing process that is used in producing profiles such as rods, tubes, and other intricate machine components. Extruded sections find almost infinite uses in all forms of complex shapes in the following areas: automotive, structural, oil and gas, military, medicine, chemical, and nuclear plants. Extrusion is widely applied in non-ferrous fields, especially with alloys of aluminum, copper, magnesium, zinc, etc. Zinc alloy as one of the non-ferrous metals requires manufacturing processes that can achieve fine grain structure useful in medical and structural applications where bio-degradable grain structures are required [1,2,3]. A study on the effect of equal channel angular pressing (ECAP) with four passes on the microstructural and mechanical properties of zinc and zinc alloys with the addition of Ag, Cu, and Mn (0.5wt%) at room temperature reported that the crystallographic structure and grain size are major factors affecting twining morphology in grain refinement [1]. Gokhale *et al*. [2] explored the microstructure and texture evolution of extruded pure zinc under severe plastic deformation (SPD). The study was carried out using indentation scratch at room temperature and a temperature of 150°C. A large grain size reduction from 86$\mu$m to (0.6- 2) $\mu$m was observed with a basal texture. It has also been affirmed that casting and solidification techniques are good tools in determining the outcome of the microstructural and mechanical properties of cast products. In line with this, an alloy of alumina silicate (Al-9wt. %Si) was studied and the result showed that there is a close relationship between secondary dendrite arm spacing and mechanical properties [3]. The mechanical properties and microstructure were improved when the extrusion was varied. With the increase in extrusion speed from 0.4mm/s to 2.4mm/s and a corresponding increase in elongation from ~3.9% to 12.2% showed complete recrystallisation with an ultrafine grain size of ~0.85$\mu$m at lower extrusion speed. However, an increase in extrusion speed produces a well-distributed grain structure having a higher recrystallisation rate [4]. The use of inverse optimisation in the deformation of pure zinc having a poly-crystal structure would produce ultra-fine grains (UFGs) at nano sizes and also give good micro-mechanical properties [5]. In the extrusion of annealed zinc–aluminum alloys where aluminum occurs as slightly coarse grain fibres resulting in good creep properties, high tensile strength, and high ductility to form zinc alumina. A strain rate of 0.034/s and a reduction in percentage elongation from 55% -35% were obtained [6]. The modeling and experimental results of secondary arm spacing revealed the relationship between mechanical properties, microstructure, and solidification parameters of Zn-Al alloy castings.

The secondary arm spacing was discovered as a major factor to stimulate growth rate and solidification time of casting [7].

The microstructural pattern of Al- 9 wt. % Si and Zn-27wt. % Al alloy casting resulted in increasing dendrite arm spacing and decreasing ultimate tensile strength (UTS); the corrosion resistance of the as-cast also increases with increasing dendrite arm spacing. This analysis shows that microstructural pattern affects the mechanical properties of alloys [8]. Piela *et al*. [9] investigated the microstructural and mechanical properties of cast and hot extruded zinc under plastic deformation by conventional extrusion. The mechanical properties of zinc improved with low temperatures having grain refinement that promotes the formation of composite microstructure with hard matrix, forming a soft interconnected structure at the boundary phase. The microstructural manufacturing of Zn-0.8Mg-0.2Sr (wt. %) alloy recorded a minimum grain size of 0.4-0.6$\mu$m and a 2.5$\mu$m average grain size. The microstructure and grain size structure were found suitable for medical applications [10]. Sztwiertnia *et al*. [11] examined a cyclic change of the deformation path that increases the plasticity of the material and stops the formation and spreading of cracks. This resulted in zinc wires with high mechanical properties and heterogeneous grains structures elongated in the boundary lines. The addition of an alloying element to increase its grain refinement, degree of recrystallisation, and decrease bio-degradation rate was achieved by varying the extrusion speed during the extrusion process. The growth of grains reduces at higher extrusion speed while at lower extrusion speed the grain growth increases as well as the recrystallis ation areas, which improves the mechanical properties of the alloy to a higher degree [12].

In this present study, the effect of varying die entry angles on microstructure and mechanical properties of extruded zinc alloy using mild steel dies in place of conventional tool steel (titanium carbide and diamond) dies were studied.

## 2.0 EXPERIMENTAL METHOD

The chemical compositions of zinc ingots in Table 1 were derived from spark test analysis. The zinc ingots were placed in a crucible cup for melting using a crucible furnace at 419.5°C. Moreover, the molten metal was poured into the sand mould and gradually allowed to solidify for a few minutes after which the billets were retrieved by the opening of the mould. This was repeated to produce seven cylindrical rods of 400 mm length and 30 mm diameter respectively. The cast samples of zinc were cut approximately to the same size but tapered in front for proper fittings into the die orifice. The cast samples were cleaned and machined into shape as shown in Figure 1 for the extrusion process.

*Table 1: Chemical composition of zinc alloy (Extract of Spark test analysis)*

| Elemental Composition | Percentage Composition (%) |
|---|---|
| Zn | 83.14 |
| Cu | 0.46 |
| Fe | 0.008 |
| Mn | 0.011 |
| Pb | 10.57 |
| Cd | 0.006 |
| Sn | 0.003 |
| Al | 5.802 |

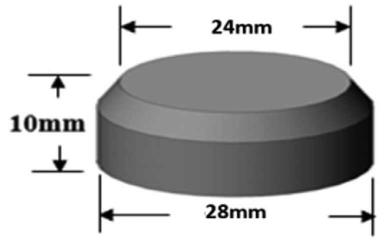


*Figure 1: Isometric view of the zinc specimen to be extruded*

### 2.1 Die and Form Tool Materials

Mild steel dies were machined to form a circular end with entry angles of 15°, 30°, 45°, 60°, 75° and 90° as shown in Figure 2. The chemical composition of the mild steel material is shown in Table 2. The mild steel dies were heated and held for 3hrs at 850°C and were subsequently cooled in the furnace to increase the strength of the material. The mild steel form tool and the ram were heated to 850°C and also held for 3hrs with gradual quenching in water. This process was undertaken to increase the strength and hardness of the tools to prevent wear and deformation during extrusion. Figures 2 and 3 show the details of the form tool and ram.

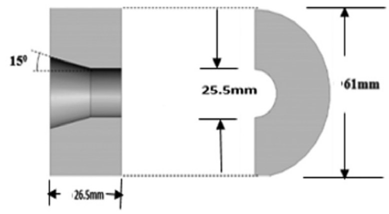


*Figure 2: Front view and End view of the die at 15° entry angle*

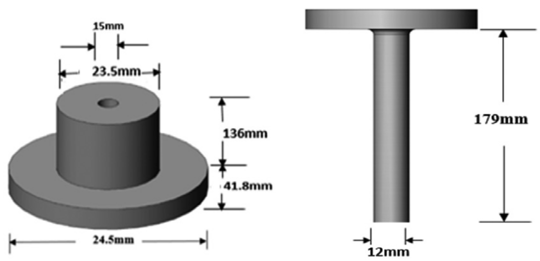


*Figure 3: Isometric view of form tool and the schematic diagram of the ram*

*Table 2: Mild Steel Spectrometer analysis*

| Elemental Composition | Percentage Composition (%) |
|---|---|
| C | 0.1195 |
| Si | 0.2887 |
| S | 0.0097 |
| P | 0.0099 |
| Mn | 0.503 |
| Ni | 0.0207 |
| Cr | 0.0430 |
| Mo | 0.0052 |
| V | 0.0065 |
| Cu | 0.0312 |
| Fe | 98.9 |

### 2.2 Extrusion Process

The direct extrusion of the cast samples of zinc alloy was performed using an England Avery Denison machine with identity: (EN76065 7113DCJ), and capacity: 600kN which was adapted for extrusion at ambient temperature to provide a compressive load on the ram. The die was fitted into the form tool and the samples of zinc alloy to be extruded were thereafter inserted through the upper cylindrical part of the form tool. The load (kN) applied on the ram was read alongside the strain gauge attached to the ram of the Avery Denison machine to measure the strain rate. Here, the time taken for the indicator on the strain gauge to complete a revolution was recorded with a corresponding load. Each complete revolution represents a 1mm elongation. The form tool setup was completed by inserting the die into the tool face after which the base was fastened to the container. The sample in Figure 1 was placed into a container through the centre hole and then covered with the ram, thereby completing the extrusion set-up. A compressive load from the punch was applied to the ram by gradually turning down the knob of the extrusion machine, and the ram in turn forced out the samples through the orifice at an extrusion pressure recorded for each sample. The extruded sample showed a reduction in thickness and an increase in length; which was performed by using six samples of zinc alloy at die entry angles of 15°, 30°, 45°, 60°, 75°, and 90° respectively. The extrusion pressures as well as the corresponding strain values were recorded for further analysis as illustrated in Figure 4.

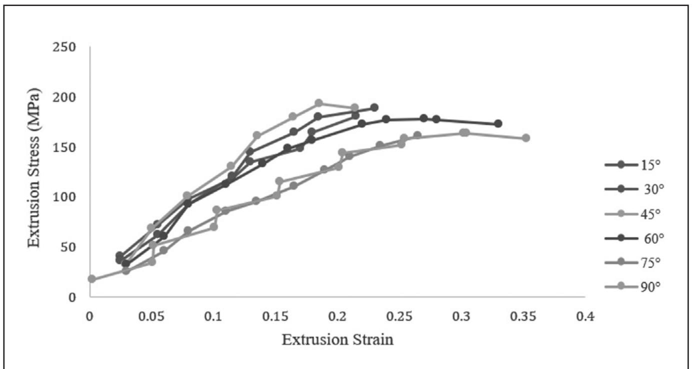


*Figure 4: Extrusion stress against Extrusion strain for extruded zinc at varying die entry angles*

### 2.3 Microstructural Examination

The extruded samples of zinc were first rough ground on a bench vice by filing them to an appreciable smoothness and consequently for smooth grinding using 220$\mu$ and 600$\mu$ emery papers. The smoothened surfaces of these samples were polished to remove scratches obtained during the grinding process. Samples were held on the surface of a polishing machine containing aluminum powder and kept moist by continuous application of waterman. Etching was performed for the 20s using 5g of sodium hydroxide (NaOH) dissolved in 100 mL of water. The etched samples were finally examined on a Celtic metallurgical microscope, model: (S/N: 07035552) at a magnification of X200. The micrographs obtained from Celtic metallurgical microscope were analysed using imageJ software to determine the grain size diameter and count frequency while the origin software was used to analyse the results.

### 2.4 Hardness Test

A Vickers micro hardness test was performed on Japan-Matsuzawa, model: MMT-X7A, digital micro hardness tester with an applied load of 100gf for a dwell time of 10s. A microscope was attached to the hardness tester to determine the accuracy and the alignment between the indenter and the specimen geometry. Three readings were taken for each sample and the average values were obtained for analysis in Figure 11.

## 3.0 RESULTS AND DISCUSSIONS

### 3.1 Extrusion Stress of Zinc Alloy Extruded at Different Angles

The extrusion stresses of zinc alloy extruded at different die entry angles are shown in Figure 4 to be 180MPa, 188MPa, 192MPa, 177MPa, 160MPa and 163MPa at 15°, 30°, 45°, 60°, 75° and 90° die entry angles respectively. The tensile strength of zinc alloy increases as the die entry angle increases to a maximum extrusion stress of 192MPa at 45°; further increase in the die angle leads to a gradual reduction in the extrusion stress of zinc alloy at 90°. The following are the strain values obtained at different die entry angles: 0.215, 0.23, 0.186, 0.27, 0.27 and 0.302. The strain value increases with increase in die angle. However, at 45°, the strain value declines, before it continues to a maximum value of 0.3 at angle 90°. This undulating pattern could be due to non-homogeneity of work hardening of the extrudate. The ductility, strength and hardness of 0.27/177MPa/ 95HV and 0.302/~163MPa/ ~73HV were obtained at 60° and 90°die entry angles respectively. The study observed that extrusion at 60°, 75°, 90° die entry angles enhances zinc alloy ductility. The extrusion stress of 192MPa at die angle 45° was the highest with the lowest extrusion strain of 0.186, while die entry angle 90° recorded the lowest extrusion stress of ~163MPa with the highest extrusion strain of 0.302. The least ductile sample was achieved at 45° die entry angle while samples extruded at 60° and 90° die entry angles showed increase in ductility. This is attributed to the reduction of grain size at higher extrusion speed while at lower extrusion speed the grain size increases, which improves the mechanical properties of the alloy [12].

### 3.2 Morphology of Zinc Extruded at Different Die Entry Angle

Micrographs of zinc extruded at 15° die entry angle as shown in Figure 5b contains very fine crystals together with the inter-metallic phase. The dendrite-like features of the inter-metallic are dispersed in the grain boundary region and well distributed in the matrix. Figure 5a shows the grain size distribution which gives a fine grain size of 5$\mu$m and a maximum bin count frequency of 90Hz. At 30° die entry angle, the zinc matrix in Figure 6b contains fine crystals of the second phase as spiral dendrite features. These dendrites features are interwoven and are well distributed in the matrix. The zinc alloy shows a grain size of 20$\mu$m with the maximum frequency of 8Hz in Figure 6a. Also, the zinc matrix at 45° die angle contains dendrite-like features with fine second phase intermetallic that are well distributed in the matrix as shown in Figure 7b. The dendrite-like crystals of the second phase inter-metallic are sandwiched in the matrix.

The secondary dendrite spacing is known to be proportional with dendrite structure and a smaller dendrite spacing, will give a finer structural dendrite morphology [2]. A smaller dendrite secondary spacing improves the mechanical strength of non-ferrous alloy [3,7]. The characteristics of cast structures, having smaller dendrite spacing is mainly due to the shorter wavelength of the periodicity of the micro segregation [7]. Figure 7a gives a grain size of 32.5$\mu$m with a frequency of more than 70Hz. The crystals of the zinc matrix in Figure 8b for 60° die angle shows dendritic crystals that are significantly larger in this sample than in Figures 6b and 7b. In Figure 8a, the grain size is 10$\mu$m with a maximum frequency of more than 250Hz. Moreover, when the die angle increases to 75° in Figure 9b the result shows more precipitated crystals in the matrix than the results in Figure 8b. However, there is a reduction in the clustering of the inter-metallic phase as well as increase in the fineness of the second phase inter-metallic. In Figure 9a, the grain size is 45$\mu$m with a maximum frequency of 110Hz. Figure 10b shows the microstructure of extruded zinc at 90°. Here, there is also an increase in the volume of the second phase precipitated in the matrix, which shows that extrusion at higher angle gives fine HCP structure. The zinc alloy and second phase intermetallic are fine at die angle of 90°, when compared to samples in Figure 10b. Figure 10a gives a grain size of 10$\mu$m with a frequency of more than 80Hz.

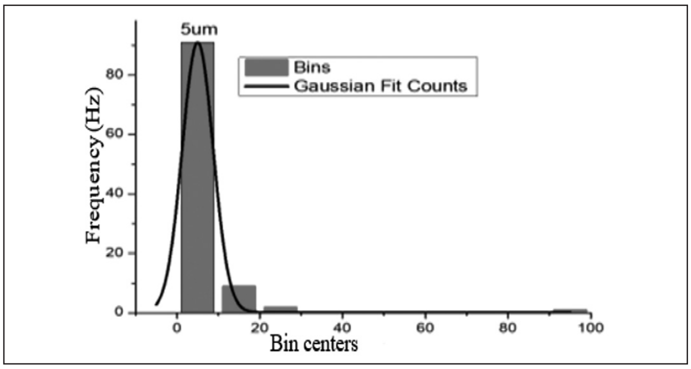


*Figure 5a: Count frequency against bin count at 15° die entry angle*

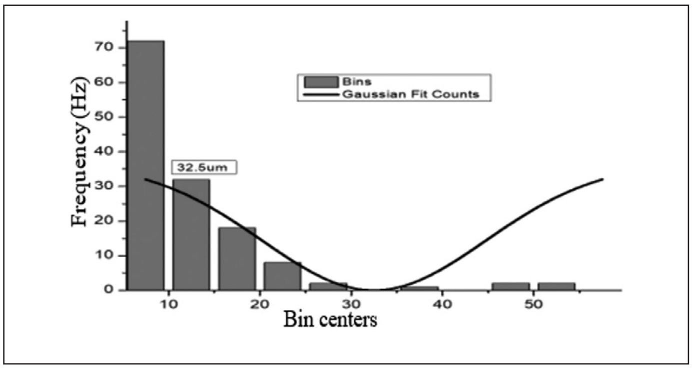


*Figure 7a: Count frequency against bin count at 45° die entry angle*

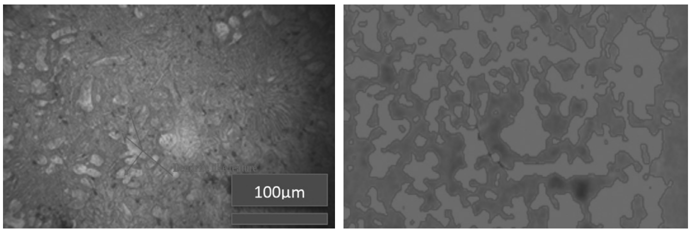


*Figure 5b: Micrographs of zinc extruded at 15° die entry angle*

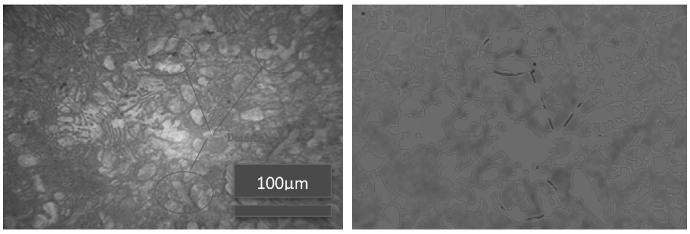


*Figure 7b: Micrographs of zinc extruded at 45° die entry angle*

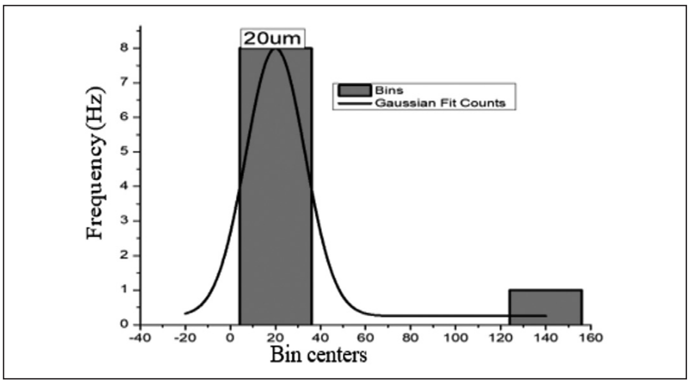


*Figure 6a: Count frequency against bin count at 30° die entry angle*

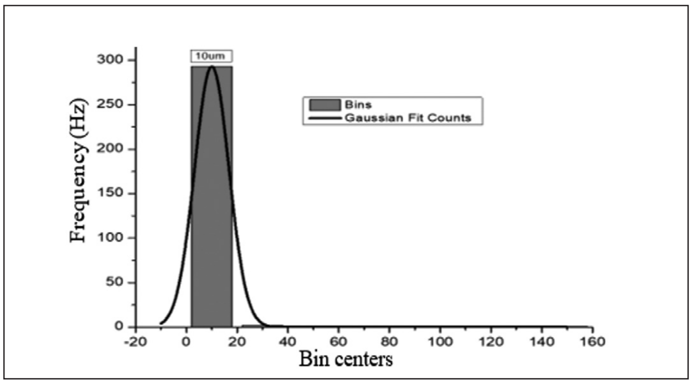


*Figure 8a: Count frequency against bin count at 60° die entry angle*

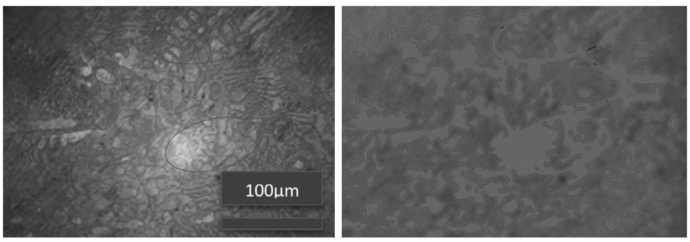


*Figure 6b: Micrographs of zinc extruded at 30° die entry angle*

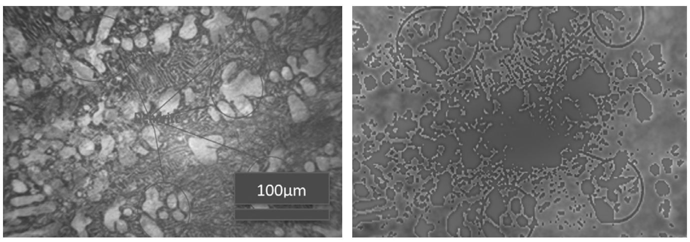


*Figure 8b: Micrographs of zinc extruded at 60° die entry angle*

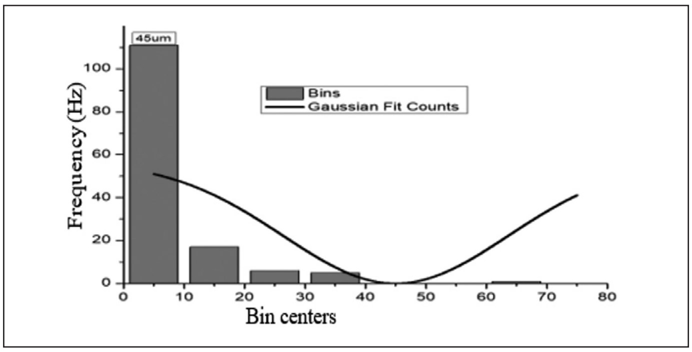


*Figure 9a: Count frequency against bin count at 75° die entry angle*

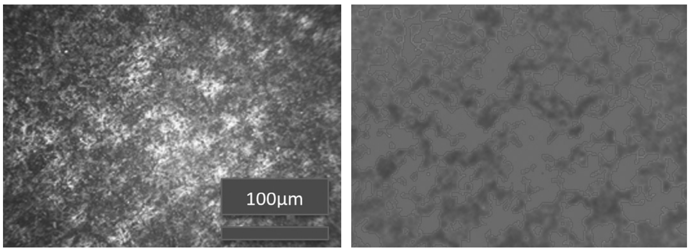


*Figure 9b: Micrographs of zinc extruded at 75° die entry angle*

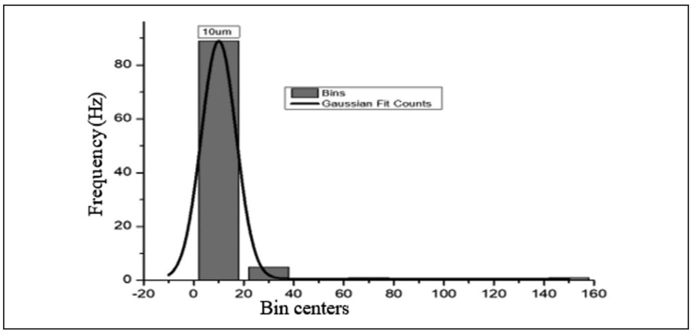


*Figure 10a: Count frequency against bin count at 90° die entry angle*

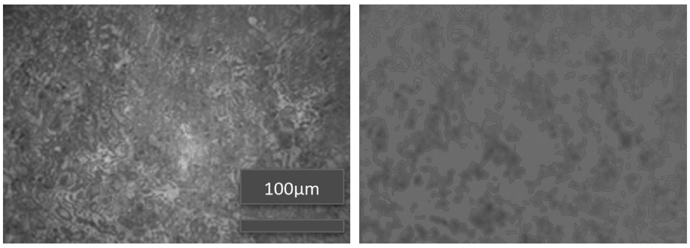


*Figure 10b: Micrographs of zinc extruded at 90° die entry angle*

## 3.3 Hardness Features of Zinc Alloy Extruded at Various Die Angles

Hardness test results of the extruded zinc alloys are shown in Figure 11. At die entry angles of 15°, 30°, 45°, 60°, 75°, and 90° with their corresponding hardness values of 85HV, 85HV, 72HV, 90HV, 95HV, and 92.5HV respectively. The zinc alloy shows progressive increase in hardness from 85HV to 95HV between die entry angles of 15° to 60°, before declining to 72HV at 90°. Die entry angle affects the orientation of grains, which appears to align parallel to direction of dislocation motion, thereby relieving the impediment potency along that direction [13]. This enhances grain crystallisation and growth.

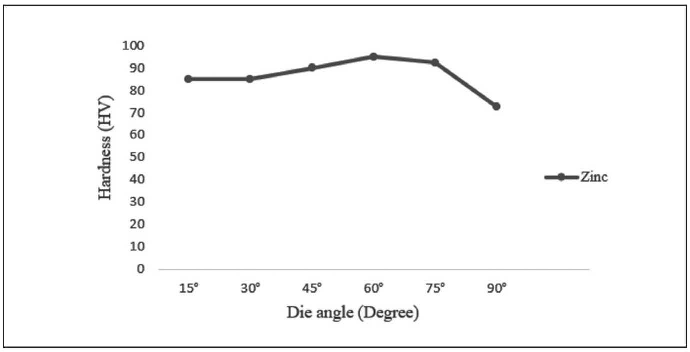


*Figure 11: Hardness against die entry angle*

## 3.4 Effect of Extrusion Angle on Ram Speed

The ram velocity decreases with increase in die entry angle when zinc alloys are extruded from 0.2mm/s at 15° to 0.032mm/s at 90°. Between 45° and 90° entry angles, the ram velocity decreases slowly as the value at 45° and 60° are equal in Figure 12. The ram velocity action is dependent on the hardness and strength of the alloy samples, which in turn is a function of the die entry angle and in all, is dependent on the effect of die entry angles on the microstructure of the extruded alloy. Increase in ram speed hinders the growth of grains and as the speed decreases the grain size and recrystallisation increases as recorded in die angles 45°(0.044mm/s), 60°(0.044mm/s), 75°(0.036mm/s) and 90°(0.032mm/s) in Figure 12[4]. It is known that materials with fine grain have proved to improve mechanical properties like hardness, tensile strength, and wear resistance [14]. The hardness and ductility properties of extruded zinc alloy increases with increase in ram speed. The mechanism for these occurrences is traceable to the presence of immobile and mobile dislocations in the alloy matrix respectively. The fragmentation of grain boundaries as a result of the size reduction enhances the dislocation generation, which promotes specimen elongation as the ultimate tensile strength and hardness values increase [15]. At a high ram speed, there is no sufficient time for grain recrystallisation and grain growth, thus forming smaller grain size, which results into improved properties. The result obtained in this research is in agreement with (Kumar *et al.,* 2018) [16], reported increase in the tensile strength and surfaced finish of the alloy with increase in ram speed in the study on the effect of ram speed in cold extrusion of nano SIC reinforced 6061 aluminum alloy. The improvement in the property of the extruded alloy was ascribed to strain hardening of the material.

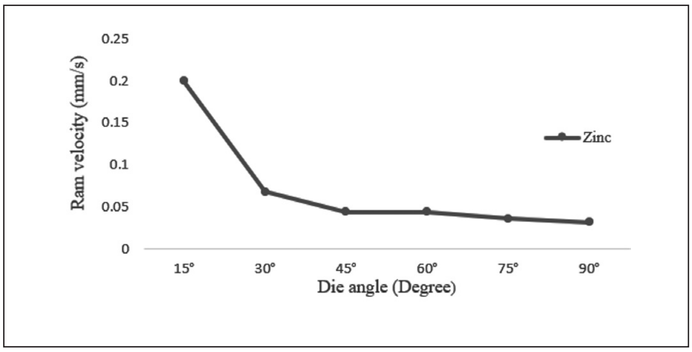


*Figure 12: Ram velocity against die entry angle*

## 4.0 CONCLUSIONS

The experimental study conducted on the deformation responses of extruded zinc alloy considering its microstructural and mechanical properties gave the following characteristics:

- The extrusion of zinc gives improved ductility; excellent strength and hardness at 60°extrusion die angle.
- The extrusion ram velocity of zinc alloy decreases with increase in extrusion die angle.
- The use of extrusion at higher die entry angle could be used to help fineness of hexagonal close pack (HCP) alloys.
- This study has shown that aside the use of severe plastic deformation to obtain fine crystals the use of appropriate die entry angle can be utilised to achieve fine crystals.
- Decrease in ram speed increases the texture of microstructure.
- The extrusion at die entry angle of 45° achieved the least ductility with fine texture in the microstructure. ■

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