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Effect of cryo-rolling with annealing treatment on microstructure and mechanical properties of AZ31B alloy

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ABSTRACT

Magnesium alloy is used in many different fields, but it is most common in the marine, and aerospace fields. The alloy is not used very much, though, because its mechanical properties are not good enough because of its coarse grain structure. The primary technique utilized to enhance the strength and hardness of a metal whereas maintaining its ideal ductility is cryo-rolling. This study seeks to investigate the utilization of cryo-rolling and annealing techniques to improve the mechanical and microstructural properties of the AZ31B alloy. During the experiment, cryo-rolling was done in two separate passes: 10 and 15 passes. Vickers hardness testers and tensile tests were used to test the AZ31B alloy after it had been cryo-rolled and annealed. The cryo-rolled sample was 28% harder than the basic material. After 15 passes, the cryo-rolled materials had a finer and more uniform grain structure. Cryo-rolling and then annealing the AZ31B alloy made it more flexible in terms of its mechanical properties.

Keywords: AZ31B alloy, cryo-rolling, annealing, microstructure and mechanical properties, process innovation.

INTRODUCTION

Magnesium and its alloys are the lightest basic materials, weighing 78% fewer than steel and 40% less than aluminum alloys [1, 2]. In the automotive industry, weight reduction is the most important requirement [31]. The feasibility of manufacturing a number of automotive parts, such as wheels, engine blocks, and steering columns, from magnesium alloy has recently been observed. The microstructure and mechanical characteristics of magnesium alloys have been the subject of numerous published studies, with a particular highlighting on the effects of mechanical alloying on fatigue aspects [3]. Magnesium alloys are known for their remarkable strength, efficient damping qualities, advantageous castability, favorable machinability, superior formability, and recyclability [4]. Their qualities deteriorate due to casting flaws, rendering them unsuitable for such applications. To improve the properties of magnesium alloys, a number of techniques to modify their microstructure have been studied [5].

It is known that two common techniques for changing the microstructure and properties of metallic materials are deformation and recrystallization. Recently, cryo-rolling has been acknowledged as a practical technique for enhancing mechanical characteristics [6]. Different from severe plastic deformation methods carried out at ambient or elevated temperatures, cryo-rolling is acknowledged as a feasible method for producing ultra-fine-grained sheets from their sample forms by distorting them at cryogenic temperatures, requiring significantly less strain [7, 8]. Cryogenic therapy is a process that alters material properties by immersing samples in liquid nitrogen. Deformation at cryogenic temperatures limits recovery and grain development, leading to elevated dislocation densities in comparison to room temperature (RT) deformation [10]. Cryo-rolling improves the strength by the creation of ultrafine grains by inhibiting dynamic recovery [11]. In most deformed metals, annealing results in softening because of dislocation annihilation by climb or cross-slip during recovery, or recrystallization [12]. AZ31B is a polycrystalline magnesium alloy consisting of several HCP-structured grains with varying orientations, and its ideal ductility represents that a material may extensively elongate, bend, or deform under tensile stress without fracturing. Nevertheless, the as cryo-rolled alloys demonstrate inadequate ductility at ambient temperature, constraining their industrialized uses. Nonetheless, ductility can be significantly enhanced with annealing treatment without compromising the extreme strength achieved [13, 14].

Yuan et al. [15] have proposed a novel cryo-rolling technique to initiate a martensite microstructure through multi-pass minor reductions followed by annealing, aimed at producing ultrafine-grained low-carbon steel. Results demonstrate that cryo-rolling the initial martensitic microstructure followed by annealing is a viable method for producing ultrafine-grained steel with smaller ferrite grains. Yu et al. [16] treated the UFG CP Ti sheets through cryo-rolling and RT rolling, subsequently subjecting them to annealing. At the same annealing temperatures, the cryo-rolled sheets have finer grains than the RT rolled sheets. This suggests that cryo-rolling and annealing can produce improved mechanical properties. The results demonstrate that the mechanical properties of the annealed samples are subjective by the dislocation density. Li et al. [17] used short annealing and cryo-rolling at different strains to create pure nickel with exceptional strength and advantageous ductility. It shows that different cryo-rolling stresses greatly increased uniform elongation after annealing without sacrificing strength.

Cui et al. [18] considered the impression of many annealing treatments on the mechanical characteristics and microstructures of cryogenically rolled AA 8011. The ultimate microstructure of cryogenic rolled material is fibrous and homogeneous. Gairola et al. [19] examined the effects of cryo-rolling and subsequent annealing on the fracture and tensile characteristics of Al alloy. Cryo-rolled materials exhibit superior fracture toughness metrics relative to solution-treated samples owing to an extended crack initiation period. He et al. [20] analyzed the mechanical responses of cryogenically rolled 2195 alloy following heat treatment. The ductility and strength of the heat-treated specimen increased relative to the as-rolled sample. Li et al. [21] examined the influence of cryogenic rolling of pure copper sheets. The substantial accumulation of dislocations and mechanical twins improves the cryo-rolled copper samples.

Several studies have looked into the enhancement of the mechanical properties of aluminium, nickel, steel, and titanium sheets by the cryo-rolling method; nevertheless, the potential for enlightening the mechanical and microstructural properties of magnesium via cryo-rolling remains unexamined. This study concentrates on the development of a refined microstructure in AZ31B using cryo-rolling treatment and examines the impact of cryo-rolling and subsequent annealing on the tensile and hardness characteristics of the AZ31B alloy.

EXPERIMENTAL PROCEDURE

Magnesium alloys possess low density and have excellent machinability. The primary material for this study was a 2.5 mm thick AZ31B alloy sheet. The elemental analysis is displayed in Table 1. Liquid nitrogen is stored in a cryogenic container to prevent evaporation upon contact with air. Dipping the material into the cryogenic container and retrieving it proved challenging; hence, a thermocol box was utilized for this process.

The current investigation employed two independent rolling conditions: 10 passes and 15 passes. Before deformation, the AZ31B alloy sheets were submerged in liquid nitrogen at 197 °C for 30 minutes to achieve consistent cryogenic cooling across the sheet thickness. Subsequent to cryogenic soaking, the samples were transferred to a two-high rolling mill for deformation. The rolling technique utilized rolls with a diameter of 80 mm at a constant speed of 1 m/min, ensuring uniform loading conditions across all trials. Figure 1 illustrates the experimental process flow.

Table 1. Elemental analysis of AZ31B alloy

Constituent	Al	Mn	Cu	Fe	Mg	Zn	Si	Ca	Ni
Percentage	3.02	0.20	0.05	0.005	95.7	0.916	0.10	0.004	0.005

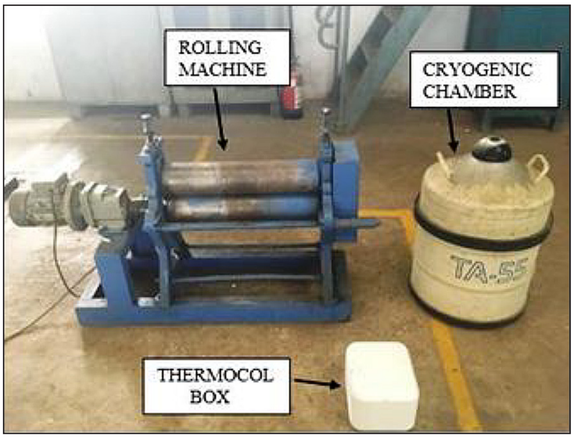


Figure 1. Cryo-rolling experimental setup

Following the cryo-rolling procedure, the samples underwent a heat treatment process of annealing. Heat treatment denotes the procedure of elevating a metal's temperature followed by its cooling to ambient conditions to enhance its characteristics [22]. It is the procedure of heating and maintaining a temperature at a controlled rate, subsequently followed by cooling at a gradual pace. The rolled samples subjected to 10 and 15 passes were annealed in a vacuum chamber at 160 °C for 1 hour. Figure 2 shows the experimental images of AZ31B magnesium alloy under different processing conditions.

Hardness assesses the resistance of solid materials to permanent deformation under applied compressive forces. Using a diamond indenter that is fashioned like a right pyramid with a square base and an angle of 136 degrees between opposing faces, the test material is indented under a weight of 5 kg in order to perform the Vickers test. The complete load is typically exerted for 10 seconds. Tensile test specimens were fabricated in compliance with ASTM B557M-02a requirements, which is shown in Figure 3. The specimens possessed a gauge length of 25 mm, a gauge width of 6 mm, and a thickness reflective of the final rolled state. Tensile tests were conducted at ambient temperature with an Instron universal testing machine, applying uniaxial tensile force until failure occurred. Three specimens were evaluated under each processing setting, and the reported values reflect the average of these measures to assure consistency. An image of the tensile test specimens prior to and after testing under cryo-rolled and cryo-rolled with heat-treated conditions are shown in Figure 4.

RESULT AND DISCUSSION

Microstructure analysis

Figure 5 illustrates the microstructure of the AZ31B alloy in its unrolled state. The elongated grains have a bright and dark scattering, indicating a significant accumulation of dislocations within the grains. The deformation degree of the Mg alloy fails to meet the requirements for whole recrystallization during manufacture. In certain regions, the deformation magnitude is beneath the essential threshold, preventing dynamic recrystallization, hence leading to the presence of elongated and coarsely equiaxed grains, which show signs of insufficient dynamic recrystallization [24].

Figures 6a and 6b present the optical micrographs of the cryo-rolled materials subjected to 10 and 15 passes, respectively. The microstructure indicates a significant disparity in grain size, characterized by a homogeneous and fine mixture, within the rolled plate. The grains of the cryo-rolled sample with 15 passes was marginally less than that of the sample with 10 passes, attributable to the greater compressive strain exerted on the rolled samples during processing.

The microstructures of the cryo-rolled and annealed samples are depicted in Figures 7a and 7b. The grain size of the annealed cryo-rolled specimensis coarser related to the cryo-rolled samples. The findings indicated that the grain size remained same substantially, suggesting that the energy supplied by annealing was insufficient to facilitate quick grain boundary movement at the specified temperature and duration, despite the occurrence of dislocation recovery. Nevertheless, the grain was evidently coarsened post-annealing. The grain size of cryo-rolled specimens is less than that of cryo-rolled specimens subjected to subsequent annealing. The grain structure is more uniform and finer was succeeded in the cryo-rolled materials after

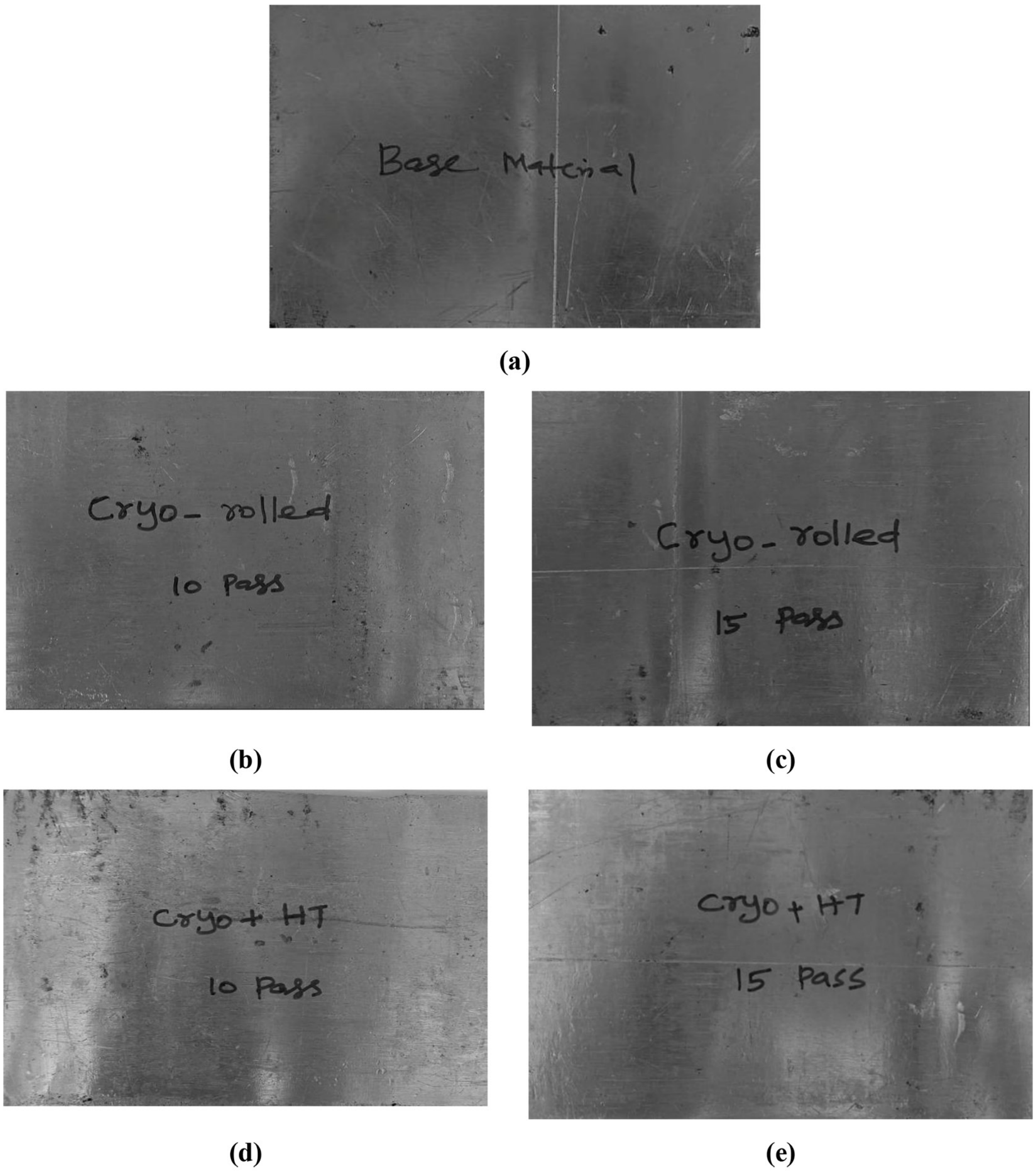


Figure 2. Experimental images of AZ31B alloy under different processing conditions: (a) base metal, (b-c) cryo-rolled, (d-e) cryo-rolled with heat-treated

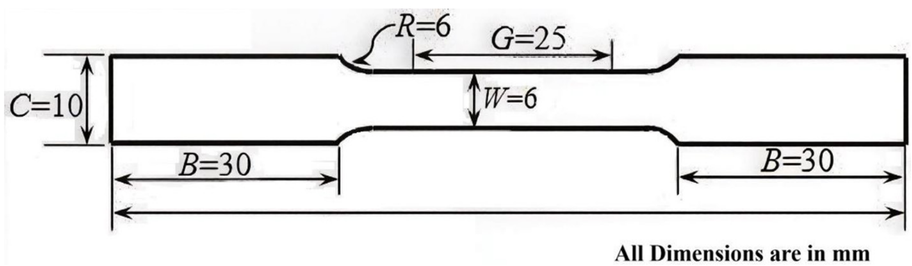
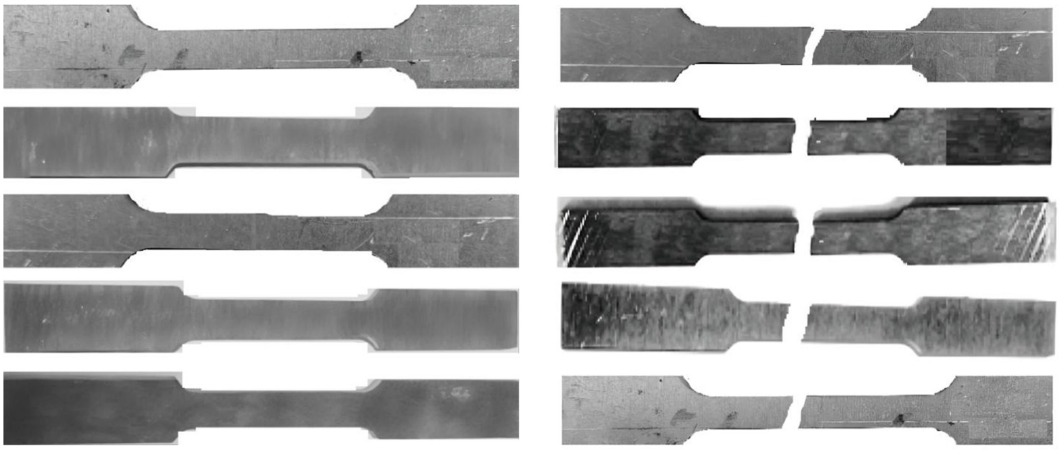


Figure 3. Tensile specimen as per ASTM B557-02a: G – gage length, W – width, R – radius of fillet, B – length of grip section, C – width of grip.

15 passes. According to previous research by Chen et al. [25], the grain size of cryo-rolled specimens is smaller than that of cryo-rolled specimens subjected to subsequent annealing, because annealing facilitates static recrystallization and grain growth, resulting in a coarser microstructure than the highly refined structure created by cryorolling.



(a) (b)

Figure 4. Tensile specimen (a) Before (b) after

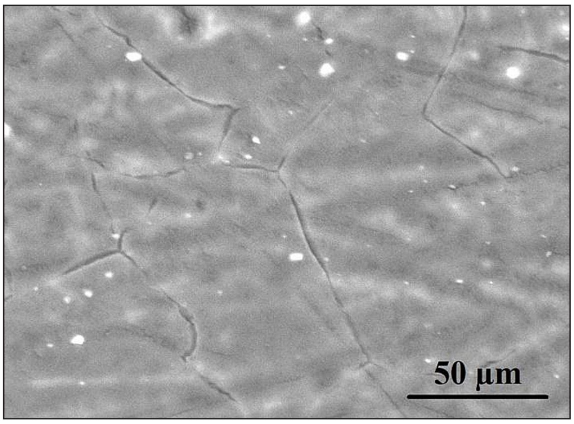
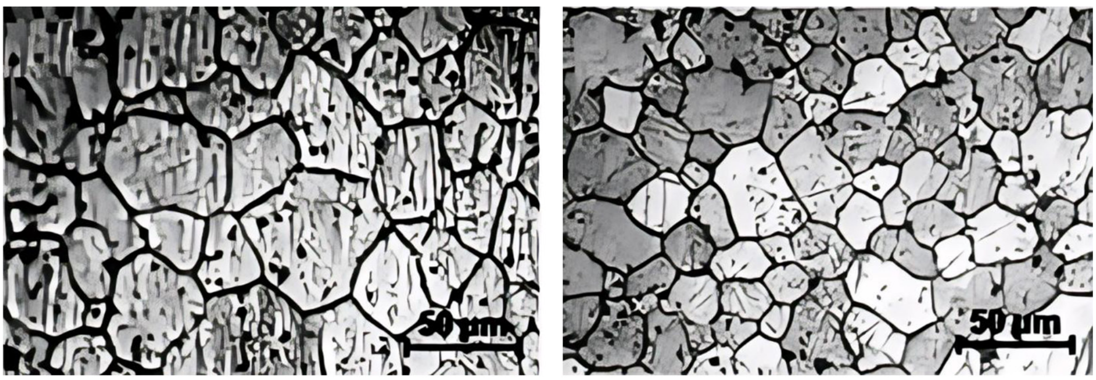


Figure 5. Microstructure of base metal

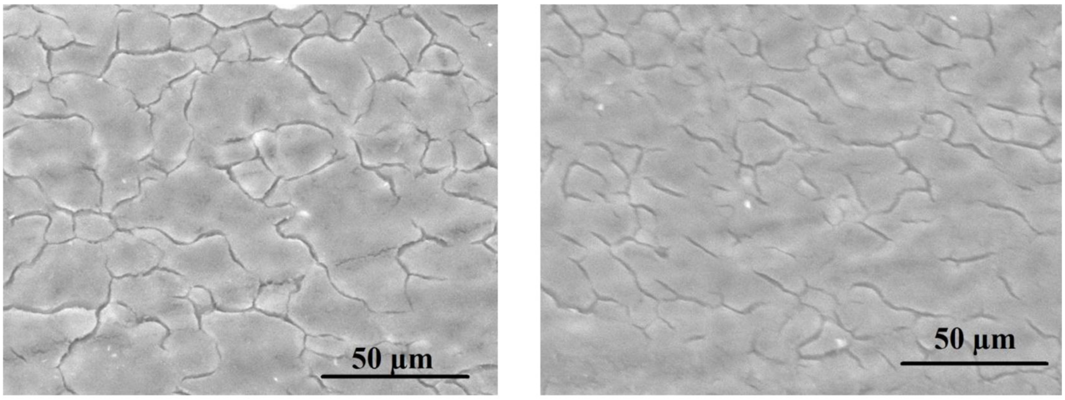
Hardness test

The hardness and average hardness of the cryo-rolled AZ31B alloy, both with and without annealing, were shown in Table 2 and Figure 8 as they were received. The average hardness value of the obtained material was determined to be 68.36 HV. The average hardness values of 84.36 HV and 87.46 HV were achieved after cryo-rolling with 10 and 15 passes, respectively. The base material demonstrates minimal hardness owing to its coarse grain structure and reduced dislocation density. Cryo-rolling significantly enhances hardness after 10 and 15 passes, mostly due to intense plastic deformation at cryogenic temperatures that inhibits dynamic recovery, resulting in grain refinement and a substantial concentration of dislocations. The hardness attains its peak after 15 passes, signifying that further deformation promotes strain hardening and microstructural refinement. Following subsequent heat treatment, the hardness of the cryo-rolled samples diminishes for both 10 and 15 passes. This decrease is linked to recovery and partial recrystallization during annealing, which decreases dislocation



(a) (b)

Figure 6. Cryo-rolled Material (a) 10 pass (b) 15 pass



(a) (b)

Figure 7. Cryo-rolled with heat treated material (a) 10 pass (b) 15 pass

density and alleviates internal stresses. Nonetheless, the annealed samples exhibit greater hardness than the base material, indicating that the finer grain structure resulting from cryo-rolling is not entirely diminished and persists in enhancing strength. The hardness value of the cryo-rolled sample rose by 28% relative to that of the base material. The rise in hardness is mostly due to the formation and buildup of dislocation networks. Cryogenic temperatures cause deformation that inhibits the recovery of dislocations, resulting in a continuous increase in dislocation density with cryo-rolling strain [26].

The average hardness of the cryo-rolled samples, subjected to annealing with 10 and 15 passes, was 72.76 and 76.24 Hv, respectively. As further passes were made during the cryo-rolling process, the hardness value started to climb. The hardness of the material resulted from structural transformations triggered by alterations in alloying distribution and grain size. The extent of internal strain reduction is contingent upon the annealing temperature, which subsequently enhances the firming of the cryo-rolled material [27]. Furthermore, the hardness of the annealed AZ31B alloy subjected to cryo-rolling is lower than that of the cryo-rolled samples. Consistent with previous research by Lee et al [28], the hardness of the cryo-rolled AZ31B alloy after subsequent annealing is lower than that of the cryo-rolled samples without annealing.

Tensile test

The change in dimesions of the specimen from its original size is detailed in Table 3. The original dimensions of the AZ31B magnesium alloy were 100 × 50 × 2.5 mm. Following cryo-rolling, thickness was systematically diminished as length and width increased due to plastic deformation. Specimens undergoing 10 and 15 passes demonstrated ultimate thicknesses of roughly 1.65 mm and 1.36 mm, respectively. Heat treatment did not induce any discernible alteration in specimen dimensions; hence, the dimensions of both cryo-rolled and cryo-rolled with heat-treated samples were unchanged. Figure 9 illustrates instances of heat-treated and cryo-rolled specimens, with (a–b) depicting the load–displacement curves and (c–d) presenting the related stress–strain curves.

Figure 10 illustrates the tensile features of the AZ31B alloy under various cryogenic processing

Table 2. Hardness for cryo-rolled AZ31B alloy with and without annealing

Sl. No.	Base material	Cryo-rolled (10 pass)	Cryo-rolled (15 pass)	Cryo-rolled 10 pass + HT	Cryo-rolled 15 pass + HT
1	68.1	84.1	87.1	72.2	77.2
2	67.3	83.2	88.2	73.5	75.6
3	68.8	85.5	87.5	72.7	75.1
4	68.3	84.7	87.2	73.3	76.4
5	69.3	84.3	87.3	72.1	76.9

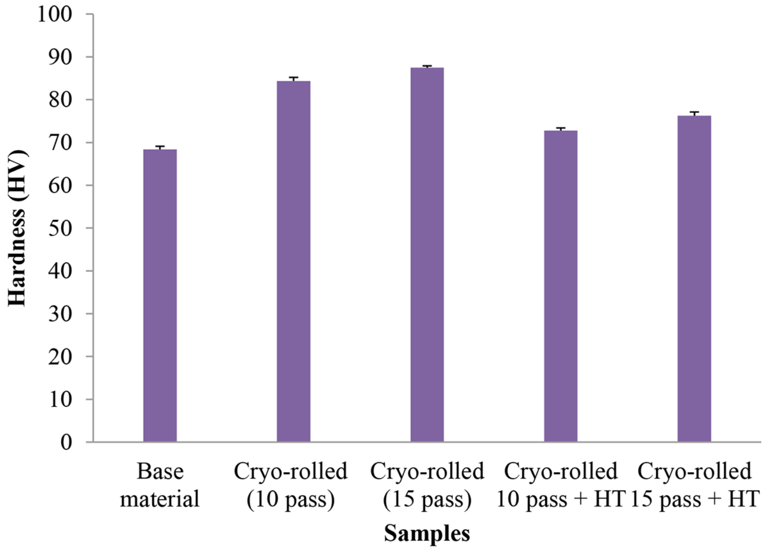


Figure 8. Average hardness for cryo-rolled AZ31B alloy with and without annealing

Table 3. Dimensions of AZ31B alloy before and after cryo-rolling

Condition	Number of passes	Length (mm)	Width (mm)	Thickness (mm)
As-received	–	100	50	2.5
Cryo-rolled	10	108	54	1.65
Cryo-rolled	15	115	57	1.36
Cryo-rolled + Heat-treated	10	108	54	1.65
Cryo-rolled + Heat-treated	15	115	57	1.36

conditions. The UTS of the base metal, cryo-rolled, and cryo-rolled followed by annealing was assessed using an Instron tensile testing machine. The base material exhibits moderate tensile strength and relatively low elongation, attributable to its coarse-grained microstructure and the restricted number of active slip systems in the HCP structure. After ten passes, the cryo-rolled sample's UTS of 260.14 MPa surpasses the base material strength of 242.26 MPa. Following cryo-rolling (10 and 15 passes), the tensile strength markedly rises, peaking at 15 passes. This enhancement results from intense plastic deformation at cryogenic temperatures, which inhibits dynamic recovery and promotes grain refinement. This enhancement is accompanied by a decrease in elongation, particularly after 10 passes, as the elevated dislocation density impedes dislocation movement. A comparable trend is noted in the cryo-rolled subsequent annealing sample with 10 and 15 passes, where the UTS rise from 248.4 to 250.5 MPa. Post-annealing, a significant enhancement in elongation is evident for both 10- and 15-pass samples, although tensile strength experiences only a little reduction. The maximum ultimate tensile strength was achieved in the cryo-rolled samples, attributed to dislocation strengthening resulting from a substantial accumulation of dislocations within the material. The destruction of dynamic recovery consequences in dense dislocations inside the material's microstructures.

Additionally, compared to the coarse-grained material as received, cryo-rolled samples show noticeable microstructural refinement, consistent with the observed deformation features. The intense plastic deformation caused by cryo-rolling results in grain dispersal and the development of finer substructures, hence improving mechanical characteristics. This refinement, caused by significant plastic deformation at cryogenic temperatures, increases the material's strength and hardness while preserving adequate ductility, therefore enhancing its overall mechanical qualities. The results agree with Zhang et al. [29], indicating that cryogenic treatment of the hot-rolled sheets greatly refines the grains, resulting in a more homogenous size. This indicates that the cryogenic treatment of hot-rolled

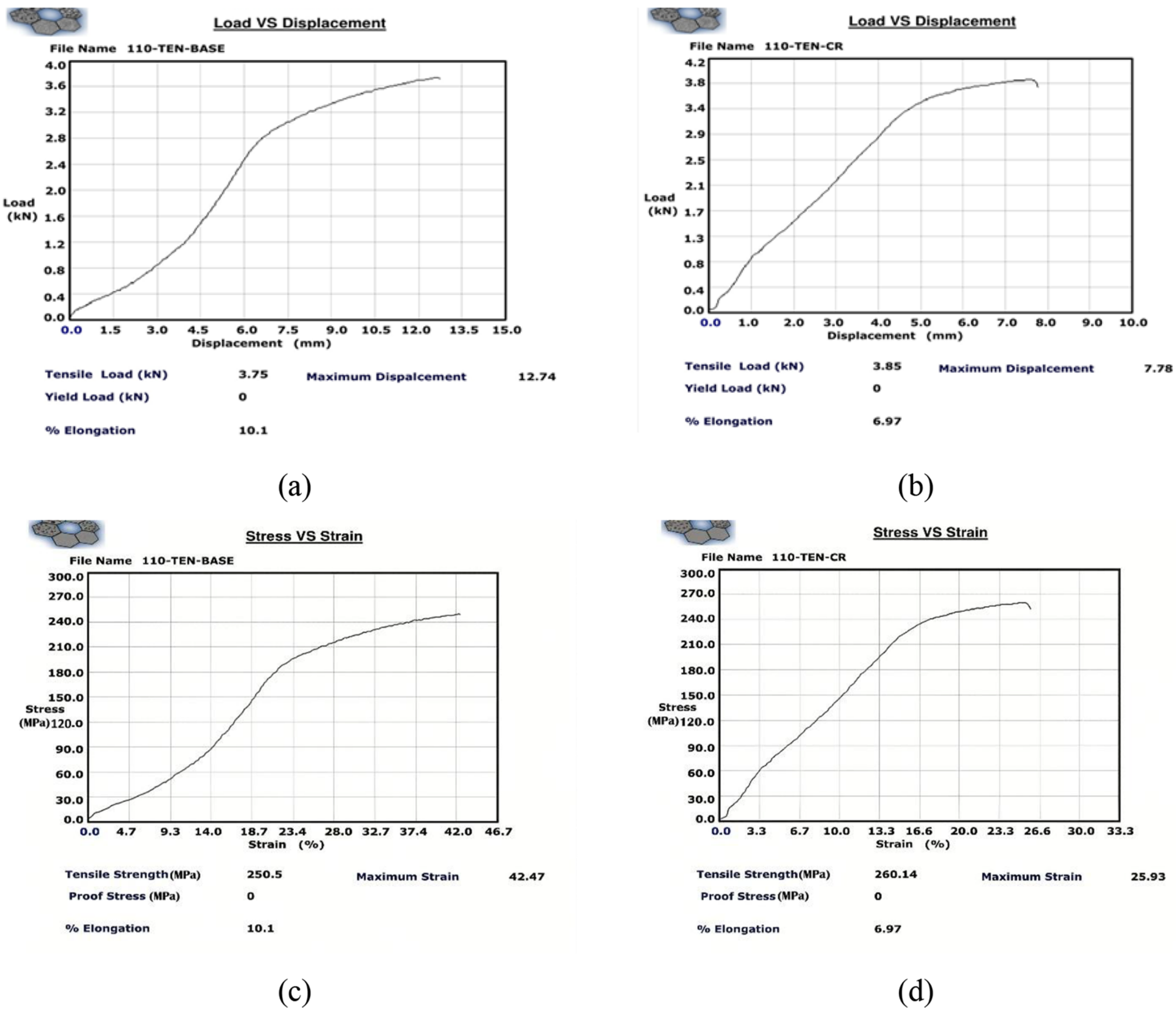


Figure 9. Tensile test of heat-treated and cryo-rolled specimens: (a-b) load–displacement, (c-d) stress–strain

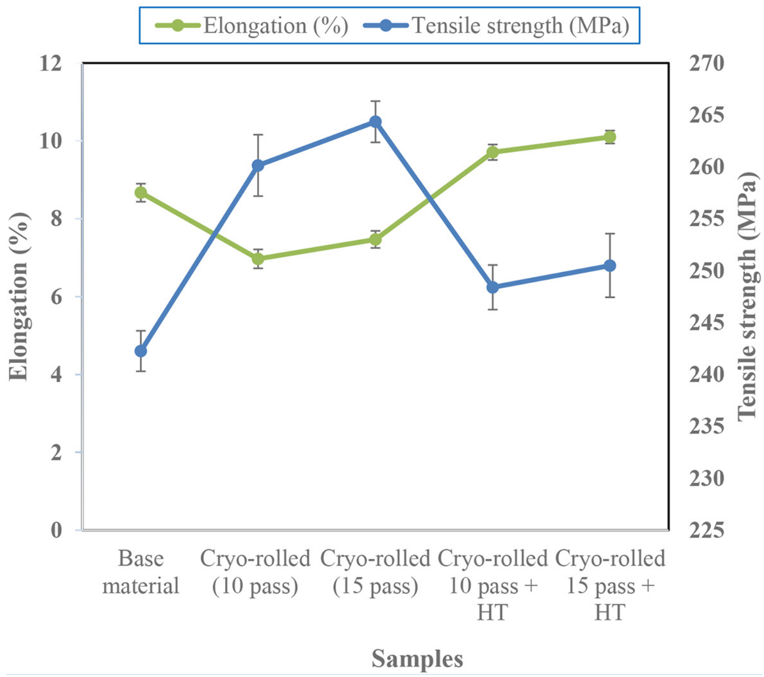


Figure 10. Tensile properties for cryo-rolled AZ31B alloy with and without annealing

sheets can significantly enhance mechanical characteristics. For cryo-rolled materials with 10 and 15 passes, the elongation was 6.97% and 7.47%, respectively. After 10 and 15 passes of the annealing process, the cryo-rolled material had elongations of 9.71% and 10.1%, respectively. According to the findings, the cryo-rolled samples that underwent annealing had more elongation than the samples that did not. Kim et al. [30, 32] observed similar results, reporting that cryo-rolled magnesium alloy specimens subjected to subsequent annealing exhibited significantly higher elongation than those retained in the as-deformed condition [33, 34].

CONCLUSIONS

Cryo-rolling and annealing in two separate passes are an efficient way to treat AZ31B materials. The effects of cryo-rolling and post-annealing treatments on AZ31B materials are methodically investigated in this work. The following are the main conclusions:

The AZ31B's as-received micrographs showed clear equiaxed and elongated grains. The cryo-rolled samples exhibited a homogeneous and fine-grained structure. Due to the increased compressive strain applied to the rolled samples during processing, the grain size of the cryo-rolled sample with 15 passes was comparatively smaller than the 10 passes.

Compared to the basal material, the cryo-rolled samples showed a higher degree of hardness. Furthermore, compared to the cryo-rolled samples, the cryo-rolled samples with annealing showed a lower hardness.

The increased tensile strength of the cryo-rolled samples was ascribed to the effects of grain boundary and refinement mechanisms, indicating that the cryo-rolling process produced a significant work hardening. By subjecting cryo-rolled AZ31B alloys to subsequent post-annealing cryo-rolling, their ductility can be enhanced.

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