

Comparison of Single-Pass Differential Speed Rolling (DSR) and Conventional Rolling (CR) on the Microstructure and Mechanical Properties of Mg5Zn

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ABSTRACT

Background

The primary hot rolling method implemented is differential speed rolling (DSR) in which the material is rolled and grains are strained, producing fine dynamic recrystallization (DRX) grains that lead to improved material strength and ductility.

Objective

The material introduced and under investigation in this paper is an Mg-based alloy, Mg5Zn (wt. %), whose microstructure is enhanced through a combination of heat treatments with proper temperature and holding time and subsequent plastic deformation through hot rolling to evaluate the effect on mechanical properties

Methods

The method involves preheating the material to various temperatures in a range from 250 °C to 350 °C and rolling to various thickness reductions to analyze the effect of single-pass differential speed rolling (DSR) and conventional rolling (CR) on the DRX process and its influence on mechanical properties.

Results

The effect of single-pass differential speed rolling (DSR) and conventional rolling (CR) on the DRX process shows that the process produces increasing amounts of finer DRX grains at higher rolling reductions, thereby improving the strength and ductility of the material.

Conclusions

This investigation demonstrated that single-pass DSR can improve the mechanical properties and formability of Mg5Zn more effectively than CR in terms of grain refinement analyzed through OM, SEM, and EBSD resulting in enhanced tensile strength and ductility [1].

KEYWORDS: Magnesium Alloys, Homogenization, Zinc, Differential Speed Rolling, Mechanical Properties, Microstructure

INTRODUCTION

Mg-based alloys have great potential in lightweighting applications, but there are some limitations due to the Hexagonal Close Packed (HCP) crystal structure and a limited number of slip systems along which plastic yielding/ deformation can occur [2, 3]. Wrought Mg alloys demonstrate poor formability at room temperature. However, the formability and mechanical properties of Mg-based alloys can be greatly enhanced through grain refinement and plastic deformation processing such as extrusion and hot rolling [4-5].

Two effective ways to modify the microstructure and enhance mechanical properties include thermomechanical deformation and alloying. One primary processing technique includes hot rolling which can be accomplished by either symmetrical rolling or asymmetrical rolling. Hot rolling has demonstrated its ability to improve strength and ductility by refining the grain size [6-8]. Mg-alloy sheets that are processed with symmetrical or conventional rolling (CR) result in the basal axis of the grains normal to the sheet surface. This type of texture limits the ductility as seen in Mg-based alloys. One of the more effective processing techniques in modifying the texture for the purpose of improving ductility is asymmetrical rolling or differential speed rolling (DSR) [10, 11]. DSR is a technique that has been shown to produce fine-grained microstructures with better ductility as well as a lower ductile-to-brittle transition temperature [9]. Grain refinement improves the alloy's formability at low temperatures. Improvement in ductility is due to the activation of deformation mechanisms other than those in the basal slip and twinning [12,13]. This behavior is crucial in the low-temperature processing of Mg-based alloy sheets, rods, wires, and tubes with ultra-fine grains.

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One of the important aspects to consider in rolling is the thickness reduction per pass (TRPP). The TRPP can influence the final microstructure and texture because coupled with rolling speed, the combination governs the strain and strain rate before recovery and recrystallization of the deformed structure during rolling and intermediate annealing. The larger thickness reduction leads to a more homogenous and fine-grained structure than a lower reduction [14]. Studies have shown that a higher thickness reduction per pass larger than 30% was required to activate the DRX at relatively low rolling temperatures [14, 15]. Based on the literature, the effect of total thickness reduction on the final microstructure and texture can be explained and these studies help understand the effects of twinning, texture, average grain size after rolling, and alloy content on grain refinement can help the understanding of DRX [16-18].

Generally, grain refinement of Mg alloys has been easily fulfilled through hot rolling such as DSR, a promising method for the continuous fabrication of large-dimension Mg sheets. In DSR, shear deformation is purposely introduced by inverse-direction friction of two rollers rotating at different speeds [19-21]. A phenomenon has been observed that DSR-processed material produces a finer grain size and hence higher ductility than conventional CR-processed sheets under the same process parameters for similar Mg alloys such as AZ31 [22-24]. Results of the effect of DSR on Mg alloys suggest that microstructure and texture are greatly affected by rolling temperature [24], speed ratio [25], thickness reduction per pass [26] and designated rolling paths [22, 24]. Of these factors, the rolling temperature is the most essential one because of the strong activation reliance of slip and twinning on deformation temperature [22]. According to the work of Chapuis and Driver [28], the critical resolved shear stress of non-basal slips and contraction twinning (CTW) decrease rapidly with increasing temperature, which greatly influences the DRX behavior. Also, Watanabe *et al.* [24] indicated that the grain size of DSR processed AZ31 decreases with reducing the rolling temperature from 300 °C to 200 °C. More recently, Cho *et al.* [22] and Kaseem *et al.* [23] stated that a similar evolution tendency of grain size is detected via changing the temperature, while the failure elongation is improved with a higher rolling temperature under which more active DRX behaviors occur.

The formation of DRX grains will be studied further in this study to demonstrate its key importance and relevance to the desired mechanical properties for Mg-Zn alloys. DRX is a type of recrystallization process, found within the fields of metallurgy and geology. In DRX, the nucleation and growth of new grains occur during deformation rather than afterward as part of separate heat treatment. The reduction of grain size increases the risk of grain boundary sliding at elevated temperatures, while also decreasing dislocation mobility within the material. The new grains are less strained, causing a decrease in the hardening of the material. DRX promotes new grains with

different orientations, which can prevent crack propagation. Rather than causing the material to fracture, strain can initiate the growth of new grains, consuming atoms from neighboring pre-existing grains. After DRX, the ductility of the material increases [17].

The aim of this paper is to evaluate the contrast between single-pass DSR and CR on the resulting grain refinement and resulting mechanical properties of homogenized Mg5Zn alloy through successive thickness reduction with optical microscopy (OM), scanning electron microscopy (SEM) as well as electron backscattering diffraction (EBSD). The presence of finer and equiaxed DRX grains is expected to result in an improvement of mechanical properties such as increased tensile strength, ductility, and even hardness [1].

MATERIALS AND METHODS

The as-cast Mg5Zn alloy investigated in this study was produced in the laboratory at North Carolina A & T State University. The nominal composition of the alloy is 95 wt.% Mg and 5 wt.% Zn. Specimens with dimensions of 110 mm length, 14 mm width and 4.8 mm thickness were prepared for DSR and CR. The DSR was performed on a customized rolling machine made by International Rolling Mill Inc. RI, USA. To improve the uniformity of the microstructure, the as-cast material was homogenized with a two-step heat treatment at 330 °C for 12 hours and then 385 °C for 4 hours after which it was quenched in water. The finished material demonstrated complete homogenization at 4 hours as seen in previous work [18].

Samples were preheated at 250 °C and 350 °C for approximately 10 minutes and then rolled at reduction rates of 10%, 20%, 30%, 40%, and 50%, with a speed ratio of 2:1 for DSR and 1:1 for CR while the temperature of the rolls being maintained at 300 °C. The final thickness of the material is shown in Table 1 for each of the given reduction rates.

Table 1: Thickness reduction rate and associated thickness after single-pass rolling.

Sample Set	Reduction Rate (%)	Final Thickness (mm)
1	10	4.32 ± 0.05
2	20	3.84 ± 0.04
3	30	3.36 ± 0.07
4	40	2.88 ± 0.03
5	50	2.40 ± 0.03

Three tensile samples were then prepared from each of the rolled specimens with wire-EDM. Three tensile specimens were also cut from the solutionized (T4) Mg5Zn material. The tensile

specimen was 45.0 mm in total length, 5 mm in width and the gauge length was 15.0 mm with a 3.0 mm cross-section, according to ASTM standards. The tensile specimens from the single-pass rolled material (DSR and CR) were cut along the rolling direction. The same orientation was used for all specimens to evaluate the influence of rolling on microstructure and mechanical properties (specifically strength or ductility). The mechanical properties of the material were evaluated by performing tensile testing. Samples were tested on the Instron Model 5566 machine with a strain rate of $\sim 10^{-3} \text{ s}^{-1}$ and tensile data was gathered to include the yield strength, ultimate tensile strength, and percent elongation for each of the tensile specimens.

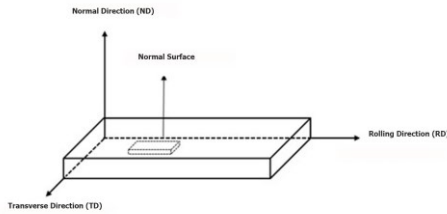


Figure 1. Samples cut for microstructural analysis prepared to the normal surface direction (ND).

The specimens for microstructural analysis were sectioned to the normal direction (ND) as seen in Figure 1. Once the sample sets were collected, they were prepared for metallography by standard mechanical grinding with SiC papers and by the final polishing with $0.05 \mu\text{m}$ Al_2O_3 suspension, then by etching using 10% nitric acid and the grain boundaries were highlighted by picric acid solution. The samples were then cleaned with isopropanol ultrasonically before being dried to prepare for OM on the Zeiss Microscope, Imager AX10. The microstructural features of the 6 processing conditions were evaluated by SEM on the Hitachi SU8000. The grain size was measured by the mean linear intercept method from the OM images. EBSD was performed using the Oxford Instrument (Symmetry) to obtain the grain orientation maps to analyze the amount and grain size of DRX grains using acquisition parameters of 15kV and $20 \mu\text{A}$.

RESULTS AND DISCUSSION

3.1. Microstructure

3.1.1. Microstructure evolution of the materials

Figure 2 shows the inverse pole figure (IPF) maps of the rolled materials for different thickness reduction rates from 10 to 50% at 350°C measured at a magnification of 50x. The fine DRX

grains can be seen throughout the Mg matrices from 10 % to 20 % reductions in Figure 2 (a to e for DSR) and (f to j for CR) and a more continuous amount of fine DRXed grains for the 30 %, 40 % and 50 % rolled specimens.

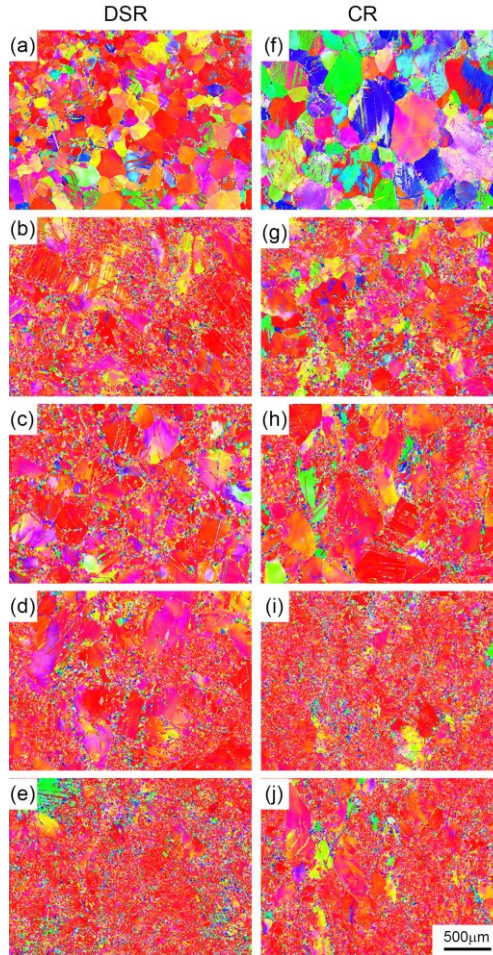


Figure 2. EBSD IPF maps of the microstructure of the DSR processed specimens Mg5Zn (preheated to 350°C) with different reduction rates: a) 10%, b) 20%, c) 30%, d) 40%, and e) 50% reduction and of CR rolled specimens with different reduction rates: f) 10%, g) 20%, h) 30%, i) 40%, and j) 50%. All graphs were taken at a magnification of 50x.

As can be seen in Figures 3 and 4, the pole figure for the T4 state shows a random orientation. The magnification for all the EBSD IPF maps and pole figures was at 50x for an accurate comparison of the T4 to the rolled specimens. Relative to the DRXed grain size after rolling that was less than 20 μm , the T4 grain size was large and close to 200 μm . Thus, there were a limited number of grains in the T4 specimen relative to the overall number of grains (area fraction) that can thus produce a strong pole intensity with preferred orientation. This is the primary explanation why the texture of the T4 was showing to be higher than the DSR and CR rolled specimens, where there was a large range of larger and smaller DRXed grains within the measured area fraction of the sample

Rolling with increasing thickness reduction results in pole figures showing strong basal poles for the specimens. Another observation is the difference in the pole figure evolutions for the DSR and CR processed materials. Although the materials processed with DSR and CR both show basal textures, those processed with DSR show the basal poles slightly shifted toward the rolling direction (RD) which becomes more prominent for larger reductions, as shown in Figure 3. On the other hand, the pole centers of the CR processed materials remained close to the center as shown in Figure 4. This is consistent with the literature in studies that have shown that a higher thickness reduction per pass that is larger than 30 % was required to activate the DRX at relatively low rolling temperatures [14-16].

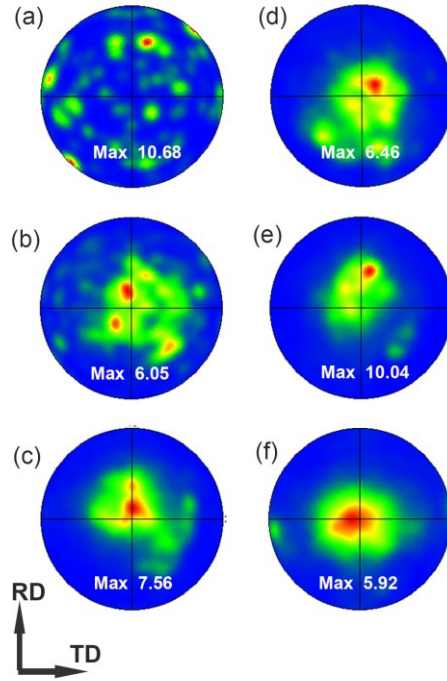


Figure 3. (0001) Pole figures of Mg5Zn showing a) T4 and DSR processed specimens with different reduction rates: b) 10%, c) 20%, d) 30%, e) 40%, and f) 50% reduction, noting how the basal poles are oriented and tilted to the rolling direction (RD).

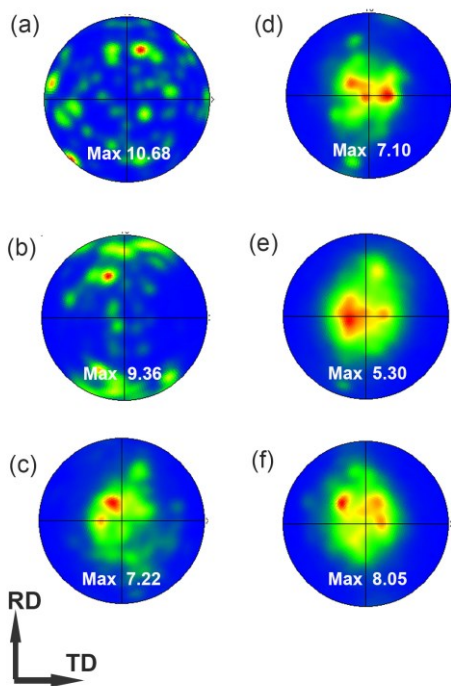


Figure 4. (0001) Pole figures of Mg5Zn showing a) T4 and CR processed specimens with different reduction rates: b) 10%, c) 20%, d) 30%, e) 40%, and f) 50% reduction, noting how the basal poles are not tilted but in closer proximity to the center.

Figure 5 shows the effect of DSR and CR on pole intensity. The effect appears to demonstrate that the slighter lower pole intensities exist for DSR as compared to CR. The lower pole intensities influence the presence of in-plane anisotropy. It may also be noted in the figure that theory demonstrates that pole intensity is usually lower for DSR than CR. There are several ways in which to analyze a pole figure. The pole intensity produced at a certain sample orientation is directly proportional to the area fraction of grains in this reflective condition. For the reduction at 40% for DSR and CR, the proportion of DRXed grains produced was very close to each other and within the margin of error. Thus, the CR produced could actually have a

lower pole intensity than DSR for the sample area measured for the given sample set.

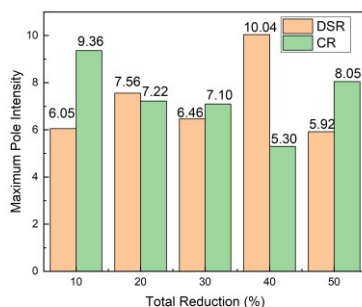


Figure 5. Pole intensity of Mg5Zn comparing DSR and CR rolled specimens from 10 to 50% reduction processed at 350 °C. The effect appears to demonstrate a slightly lower pole intensity for the DSR specimens.

3.1.2 Grain size of the rolled Mg5Zn materials

Upon evaluation, there was an increase in the fraction of the DRXed grains with the increase in the reduction rate, but a few lingering parent grains (unDRXed grains) even remained at 30 %, 40 %, and 50 % reductions. Figure 6 shows the grain size for the Mg5Zn material for DSR/CR rolled material for each reduction rate. The figure shows that the average grain size after DRX at 50% was 7.0 µm for the DSR material and 7.3 µm for the CR material processed at 250 °C. Thus, DSR produced a slightly finer grain size than CR, but not a significant difference that may even be considered within the statistical error. This was also valid for the 350 °C temperature comparing DSR at 7.7 mm and CR at 8.2 mm.

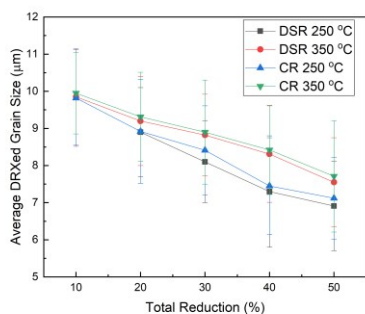


Figure 6. DRX average grain size of the Mg5Zn comparing single-pass DSR and CR rolled specimens.

3.1.3 Tensile testing of the rolled Mg5Zn materials

Table 2 shows data from the tensile testing for the single-pass DSR and CR rolled material. Results indicate that the strength of the material increases up to a 40 % reduction rate and then begins to taper off afterward, which shows that the material reaches its peak strength around a 40 % reduction rate

Table 2. Tensile results for the single-pass DSR and CR Mg5Zn samples.

Sample Condition	UTS (MPa)	YS (MPa)	ε (%)
DSR			
250 °C 20%	235.2 ± 6.4	130.6 ± 2.4	17.1 ± 2.2
250 °C 40%	260.1 ± 5.8	147.2 ± 3.1	28.2 ± 2.1
250 °C 50%	254.3 ± 4.8	150.3 ± 4.4	16.3 ± 3.2
350 °C 10%	208.2 ± 9.2	105.5 ± 3.2	10.2 ± 3.1
350 °C 20%	222.3 ± 9.8	109.3 ± 4.1	19.1 ± 2.9
350 °C 30%	242.2 ± 13.2	125.3 ± 5.2	21.3 ± 2.4
350 °C 40%	255.1 ± 15.2	132.3 ± 4.6	27.4 ± 2.6
350 °C 50%	250.2 ± 10.5	141.4 ± 3.8	17.3 ± 2.1
CR			
250 °C 10%	225.2 ± 5.2	110.2 ± 2.5	6.9 ± 1.1
250 °C 20%	236.1 ± 5.8	126.1 ± 2.9	10.1 ± 1.3
250 °C 30%	255.3 ± 6.2	141.3 ± 3.2	13.9 ± 1.5
250 °C 40%	261.2 ± 5.4	145.1 ± 3.5	16.2 ± 2.1
250 °C 50%	255.3 ± 6.4	148.2 ± 3.1	14.2 ± 1.9
350 °C 10%	212.1 ± 4.5	105.3 ± 2.2	7.1 ± 1.1
350 °C 20%	232.3 ± 4.8	112.2 ± 4.2	12.2 ± 2.2
350 °C 30%	252.2 ± 6.5	125.1 ± 3.4	14.1 ± 2.4
350 °C 40%	257.1 ± 6.8	129.3 ± 4.1	16.5 ± 2.6

350 °C 50%	252.4 ± 5.2	139.2 ± 4.8	14.5 ± 1.8
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The graphs of the tensile strength for the T4 as well as the single-pass DSR and CR at 250 °C and 350 °C material can be seen in Figure 7. In the figure, the 40 % rolled tensile specimen exhibited the highest UTS while the T4 exhibited the lowest UTS. Ductility also increased up to 40 % reduction material and tapered off as reduction increased to 50%. The increase in strength and ductility up to 40 % reduction is attributed to the increase in presence of the small DRXed grains and the slight drop at 50% was within the statistical error. UTS and % elongation for each of the conditions can be seen in Figures 7 and 8. In Figure 6, DSR at 250 °C and 350 °C had UTS similar

to CR but considerably higher elongation (ductility) for the DSR at both temperatures.

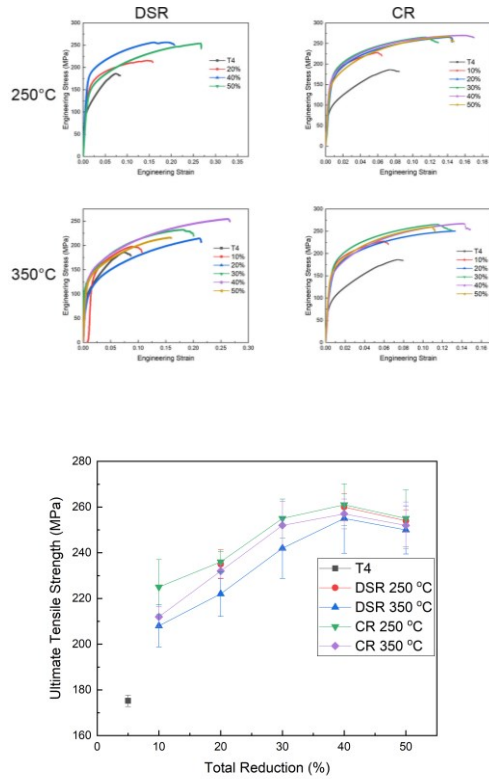


Figure 8. Ultimate tensile strength (UTS) of the DSR and CR specimens processed at 250 °C and 350 °C for reductions from 10 to 50%.

In Figure 8, UTS is highest for both DSR and CR at 250 °C which correlates to the grain size being the smallest at 250 °C. On the other hand, the elongation (ductility) is highest for DSR and CR at 350 °C shown in Figure 8, where the grains are slightly larger. As stated previously, the reduction of grain size increases the risk of grain boundary sliding at elevated temperatures, while also decreasing dislocation mobility within the material. The new grains are less strained, causing a decrease in the hardening of the material. DRX promotes new grains and orientations,

which can prevent crack propagation. After DRX, the ductility of the material increases as can be seen in Figure 9. A similar trend was found in reference 17.

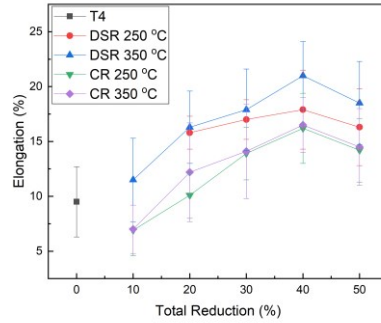


Figure 9. Elongation (%) showing ductility of the DSR and CR specimens processed at 250 °C and 350 °C.

The enhancement of mechanical performance through grain refinement can be analyzed and evaluated through the Hall-Petch relationship. According to the Hall-Petch relationship, $\sigma = \sigma_0 + k \times d^{-1/2}$, the strength of a material increases with a decrease in grain size. We can see the Hall-Petch relationship of the specimens for DSR and CR at 250 °C and 350 °C as seen in Figure 10. The graph exhibits a Hall-Petch relationship in which the strength of the material can be achieved by the reduction of its grain size.

One general approach to strengthen metals can be achieved by changing the material's microstructure to make dislocation motion more difficult, also referred to as grain boundary strengthening. Most metals have a large number of randomly oriented grains, or crystals, separated by grain boundaries. These polycrystalline materials have crystals with differing orientations of their lattice and slip systems forced to conform to the overall strain. A polycrystal is deformed by disassembling it into its constituent grains and allowing each to slip according to Schmidt's Law, thereby introducing statistical dislocations. When the crystals are subsequently reassembled, they no longer "fit" together, where some adjacent grains may have moved apart leaving a gap. The number of dislocations required to put the polycrystal back together is proportional to the strain times the grain size times a "geometrical constant."

The smaller grains produced through grain boundary strengthening result in a higher strength of the fine grained polycrystalline specimen and was first quantified by Hall (1951) and Petch (1953) in the familiar Hall-Petch relationship (see equation in manuscript). The behavior is typical of other types of boundaries such as second-phase particles and mechanical twins. In general, more closely spaced barriers to dislocations produce greater strength.

The grain size dependence of yield stress from Hall-Petch states that there is no requirement for dislocation pile-ups at grain boundaries. Instead, the boundary may act as a source of dislocations, and the capacity to emit dislocations is dependent on the character of the grain boundary [29].

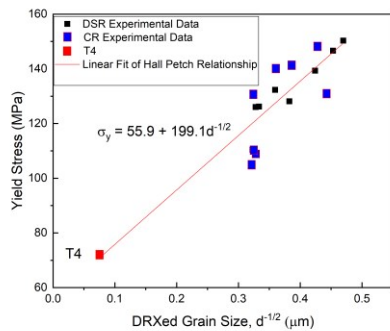


Figure 10. Relationship of yield stress (MPa) and DRXed grain size for the DSR and CR specimens processed at 250 °C and 350 °C for reduction from 10 to 50%. The graph exhibits a Hall-Petch relationship in which the strength of the material can be achieved by the reduction of its grain size.

CONCLUSIONS

The mechanical properties of Mg-based alloys can be enhanced by single-pass DSR and CR by increasing the proportion of DRX grains through dynamic recrystallization. The results of this investigation show an increase in DRX grains for increasing thickness reduction rate and improvement in both the strength and ductility of Mg5Zn achieved at higher thickness reduction rates for the DSR and CR. These results are consistent with literature wherein an increase in the level of DRX grains produces higher strength and ductility. However, the DSR specimens exhibited a slightly smaller grain size at higher reductions than the CR counterparts (but within the margin of error) and a slightly larger area fraction of DRX grains compared to the CR counterparts. This was also consistent with literature stating that DSR produces a finer grain size than CR.

Furthermore, the larger thickness reduction lead to a more homogenous and fine-grained structure in this study as consistent with prior studies where a thickness reduction per pass higher than 30 % activated the DRX at relatively low rolling temperatures.

Further work is being done in this area with the Mg5Zn samples to evaluate the influence of DRXed grains and precipitates on the strength and ductility and overall mechanisms behind dynamic recrystallization.

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