

Article

Microstructural and XRD Investigations on Zn After Plastic Deformation

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Abstract

This work presents a microstructural analysis and X-ray diffraction (XRD) investigation of the plastic deformation in commercially pure, single-phase hexagonal close-packed (hcp) Zn subjected to rolling and tensile tests up to failure. Samples were examined by optical microscope and XRD; hardness was assessed by Vickers microhardness. High-resolution diffraction profiles with $K\alpha_1/K\alpha_2$ deconvolution were used to identify deformation-induced texture and to estimate the dislocation density. Results show that rolling (40% thickness reduction) and tensile test change texture and cause peak shifts and broadening, with corresponding microstructural changes. Microhardness changes from 28–45 HV (annealed) to 30–50 HV after deformation. After rolling, the texture (002) is the most intense reflection and (004) increases without significant angular shifts. Tensile tests induce low-angle shifts of (101) and (004), as well as selective texture changes (appearance of (103) and (110)). The (101) full width at half maximum increases from $\beta(2\theta) = 0.115^\circ$ (annealed) to 0.160° (rolled) and 0.140° (after tensile test), yielding dislocation densities from $2.73 \times 10^6 \text{ cm}^{-2}$ (annealed) to $3.03 \times 10^{11} \text{ cm}^{-2}$ (rolled) and $3.38 \times 10^{10} \text{ cm}^{-2}$ (after tensile test). Finally, this study quantifies the XRD parameters (full width at half maximum, angular shifts and dislocation density). Plastic deformation of pure Zn leads to significant microstructural changes, including grain refinement, the generation of dislocations, and the formation of new crystallographic orientations, which are then observable in XRD patterns as peak broadening, shifts, and texture development. The severity of these effects depends on the level of deformation.

Keywords: plastic deformation; X-ray diffraction; microstructural investigation; average grain size; dislocation density; peak shift



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1. Introduction

Metals and alloys usually undergo plastic deformation through dislocation slip or twinning. These mechanisms are alternative/competing and are strongly controlled by the stacking fault energy γ_{SF} . In high γ_{SF} metals ($>60 \text{ mJ m}^{-2}$), such as Al [1] and Ni [2], dislocations do not dissociate into two partials; therefore, deformation occurs exclusively by dislocations sliding, cross-slip is possible, and the final structure presents cells and subgrains. In metals with low γ_{SF} ($<25 \text{ mJ m}^{-2}$), such as Ag [3] and AISI 304 stainless steel [4], dislocations are dissociated, cross-slip cannot occur, and plastic deformation produces co-planar dislocation structures with the presence of numerous twins and stacking faults. Metals with intermediate stacking fault energy ($25 \text{ mJ m}^{-2} < \gamma_{SF} < 60 \text{ mJ m}^{-2}$) show mixed structures that vary depending on the slip system involved and the applied strain rate [5].

Zn and its alloys have been extensively researched for their low cost, availability, and overall properties, making them suitable for various industries and applications. Zinc alloys are the most recent innovation in biodegradable metals, providing a sustainable solution. They have a moderate corrosion rate compared to other biodegradable metals such as magnesium and iron-based alloys. However, zinc alloy's mechanical characteristics are often insufficient for load-bearing implant applications. Severe plastic deformation (SPD) can improve the mechanical properties of zinc-based alloys. SPD can affect not only mechanical properties of Zn alloys, but also corrosion and biological behavior. Recent studies have examined how different SPD procedures affect the characteristics of zinc and its alloys [6]. Zinc degrades moderately in biological fluids, and the zinc released during the degradation process is deemed safe for the human body. However, these materials have significant limitations in terms of mechanical properties for medical applications. Adding alloying materials, as well as grain refining by thermomechanical processing, are identified as viable solutions to this problem.

In recent years, severe plastic deformation (SPD) methods have been proposed for processing Zn-based bioalloys to achieve acceptable mechanical properties while maintaining the required biocorrosion behavior [7]. Yu et al. analyzed the microstructure evolution of polycrystalline Zn during tensile testing at room temperature [8]. With strain lower than 15%, EBSD data indicated that dislocation sliding was the primary deformation mechanism, followed by dynamic recrystallization (DRX) and grain growth. Pure zinc's high strength and plasticity can be achieved through grain refinement and DRX. These findings suggest a promising approach for the production of high-strength Zn components. Solas et al. [9] presented a model to simulate the deformation and recrystallization of hexagonal metals, using Zn as a case study. The goal is to predict changes in microstructure, such as grain size and crystallographic texture, which occur during plastic forming and subsequent annealing treatments. The study concludes that for Zn, static recrystallization is controlled by a mechanism where nuclei form in the most highly deformed grains and then grow by consuming the less-deformed neighboring grains. This behavior is typical for materials with high plastic anisotropy, highlighting the model's ability to link deformation processes to recrystallization outcomes and providing a powerful tool for predicting texture and microstructure evolution.

Piela et al. [10] compared the microstructure and mechanical properties of pure Zn subjected to two different plastic deformation methods: conventional extrusion and an extrusion method with a forward–backward rotating die, known as the KoBo method. The research used two types of initial Zn material: a cast ingot and a commercially hot-extruded rod. The KoBo extrusion method significantly increases the strength and ductility of Zn compared to conventional extrusion. For the ingot material, KoBo extrusion increased the yield strength by approximately 67% and the tensile strength by 37%. Furthermore, the tensile elongation for the ingot batch was 133% higher with KoBo compared to the conventional method. For the hot-extruded batch, the increment in yield and tensile strength were about 12% and 14%, respectively. For both initial materials, the grains in the KoBo-extruded samples were considerably smaller than those in the conventionally extruded ones. The process also creates a more homogenous grain size across the final product's cross-section.

The influence of cold-rolling on microstructure evolution of Zn-3Cu-1Mg-0.3Nd alloy was explored by Liu et al. [11] at varying reduction levels. The study elucidates the strengthening and toughening mechanisms in zinc alloys through cold-rolling and the addition of the Nd element, particularly in terms of microstructural control and crack passivation.

The aim of this work is to study the behavior of commercial pure Zn, a high-density metal with intermediate stacking fault energy ($\gamma_{SF} = 35 \text{ mJ m}^{-2}$), deformed under two

different conditions: rolling and tensile test up to failure. Zn was chosen because it is a single-phase, hexagonal, close-packed metal. Localized plastic deformation was characterized on the same specimens by combining optical microscopy, Vickers microhardness, and X-ray diffraction. To achieve this goal, Vickers microhardness tests, X-ray diffraction measurements, and optical metallography were performed close to the failure for tensile test specimen and on the surface of rolled Zn samples to obtain a precise correlation between microstructural variations, particularly grain size, dislocation density, and texture. The work provides a careful and detailed set of experimental observations on deformation induced microstructural evolution useful not only in commercially pure Zn but also in hcp metals in general.

2. Materials and Methods

Commercially pure zinc (Zn 99.99%, ingot form) was used. Prior to machining, material was annealed at 200 °C for 30 min and water-quenched. This thermal treatment allows the alloy to relieve internal stresses, reduce defects, and increase ductility and softness. Dog-bone tensile specimens with reduced section $1.5 \times 10 \times 40$ mm and plates for rolling with 4 mm initial thickness were prepared from the same batch. These were subjected to metallographic preparation and chemical etching (77% H₂O, 1% Na₂SO₄, 16% CrO₃, and 6% HNO₃) to analyze their structures, such as grains and twins.

Optical microscopy observations were conducted before and after rolling and tensile test close to the failure zone. Tensile test was performed on chemically etched specimens until failure. The tests were conducted using an MTS Insight 50 kN machine under static conditions (0.5 mm/min crosshead speed). The other specimens were subjected to rolling up to a 40% reduction in thickness.

Vickers microhardness measurements (25 g load, 10 s) were performed along the main axes of the samples before and after tensile test and rolling. X-ray diffraction measurements were carried out on the sample annealed, after tensile test and after rolling. The precision profiles of the most intense peaks were recorded using Mo-K α radiation ($\lambda = 0.071$ nm) with step-by-step scans (2Θ step of 0.005° and counting time of 20 s per step). In the case of the tensile test specimen, the beam was focused on the local region to be examined.

After identifying the contributions of K α_1 and K α_2 (see example in Figure 1), the position, relative intensity, and half-width β of all diffraction peaks were determined. For deconvolution, the Rachinger method [12] was adopted, considering the following three constraints: (i) the area ratio K α_1 :K $\alpha_2 = 2:1$; (ii) the Full Width at Half Maximum (FWHM) for both K α components must be the same; (iii) an angular offset of K α_2 toward higher 2θ by $\Delta(2\theta)_{K\alpha}$, which is fixed by Bragg's law via Equation (1) from the wavelengths $\lambda_{K\alpha1}$ and $\lambda_{K\alpha2}$.

After K α_1 /K α_2 deconvolution, the doublet geometry is constrained by Bragg's law: the angular offset between components is fixed by the wavelength ratio and the Bragg angle, as detailed in Equation (1):

$$\Delta(2\theta)_{K\alpha} \approx 2 \tan \theta_{\alpha1} \left(\frac{\Delta\lambda}{\lambda_{\alpha1}} \right) \quad (1)$$

where $\Delta\lambda = \lambda(k\alpha_2) - \lambda(k\alpha_1)$.

For each X-ray diffraction peak, the total line broadening β_T is essentially due to two contributions: the size of the coherently diffracting domains (β_D), i.e., the grain size, and the micro-strains (β_ϵ). Thus, β_T can be written as follows:

$$\beta_T = \beta_D + \beta_\epsilon = \frac{K\lambda}{d \cos \vartheta} + 2\epsilon \tan \vartheta \quad (2)$$

where d is the domain size, ε is the average micro-strain, ϑ is the Bragg angle, λ is the wavelength, and K is a constant ($=0.89$). By studying the different precision peaks, the grain size (D) and the micro deformations (ε) are obtained in the following way:

$$\underbrace{\frac{\beta_T \cos \vartheta}{\lambda}}_y = \underbrace{\frac{1}{\langle D \rangle}}_q + \underbrace{2\varepsilon}_m \underbrace{\frac{\sin \vartheta}{\lambda}}_x \quad (3)$$

If the micro-strains are isotropic, the equation just written does not depend on the reflections, therefore by plotting the values $(\beta_T \cos \vartheta)/\lambda$ of the peaks as a function of $\sin \vartheta/\lambda$, a straight line with an angular coefficient equal to 2ε is obtained, which intercepts the y -axis at zero at the point $1/\langle D \rangle$. Finally, the dislocation density ξ , was calculated using the Williamson–Smallman relationship [13]:

$$\xi = \frac{\Xi \varepsilon^2}{k_0 b^2} \quad (4)$$

where $\Xi = 16$ is a constant, $b = 2.665$ nm is the modulus of the Burgers vector, and $k_0 \cong 1$ is a factor depending on the interaction between the dislocations.

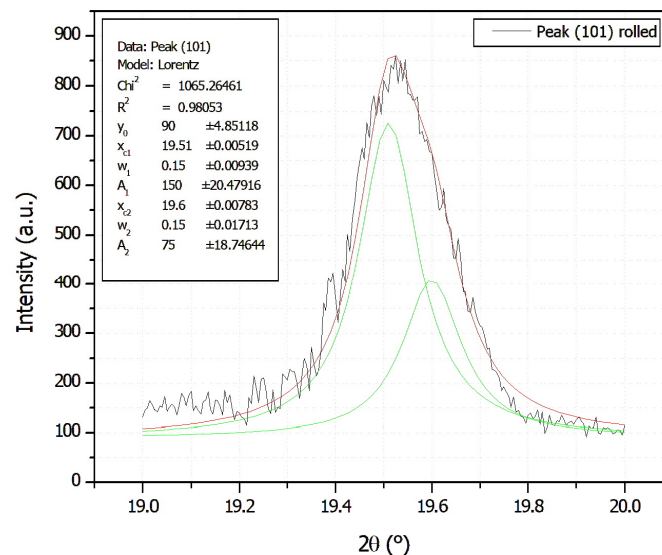


Figure 1. Deconvolution of the (101) peak in rolled sample: raw data (black line), fitted (red line), $K\alpha_1$ (green line—left) and $K\alpha_2$ (green line—right).

3. Results and Discussion

The Zn samples after metallographic preparation and chemical etching present a variable grain size as shown in Figure 2a,b. For the annealed samples, inhomogeneous structures have been evidenced and twins are also present (Figure 2a). Figure 2c,d show samples after tensile test in which grain protrusion, slip lines, and twins are noted. Figure 2e,f show rolled samples in which grain elongation in the rolling direction, twins, and recrystallization can be noted. In the annealed sample, the microhardness values along the sample axis range from a minimum of 28 HV to a maximum of 45 HV. In both the tensile and rolled samples, there is no appreciable increase in microhardness along the sample axis; in fact, the values range from a minimum of 30 to 50 HV. Variability is quantified by reporting mean $\pm$ standard deviation: annealed ($\mu \pm \sigma$) = 36.5 ± 4.5 HV, rolled 40% ($\mu \pm \sigma$) = 40.0 ± 5.0 HV, after tensile tests ($\mu \pm \sigma$) = 39.0 ± 4.5 HV.

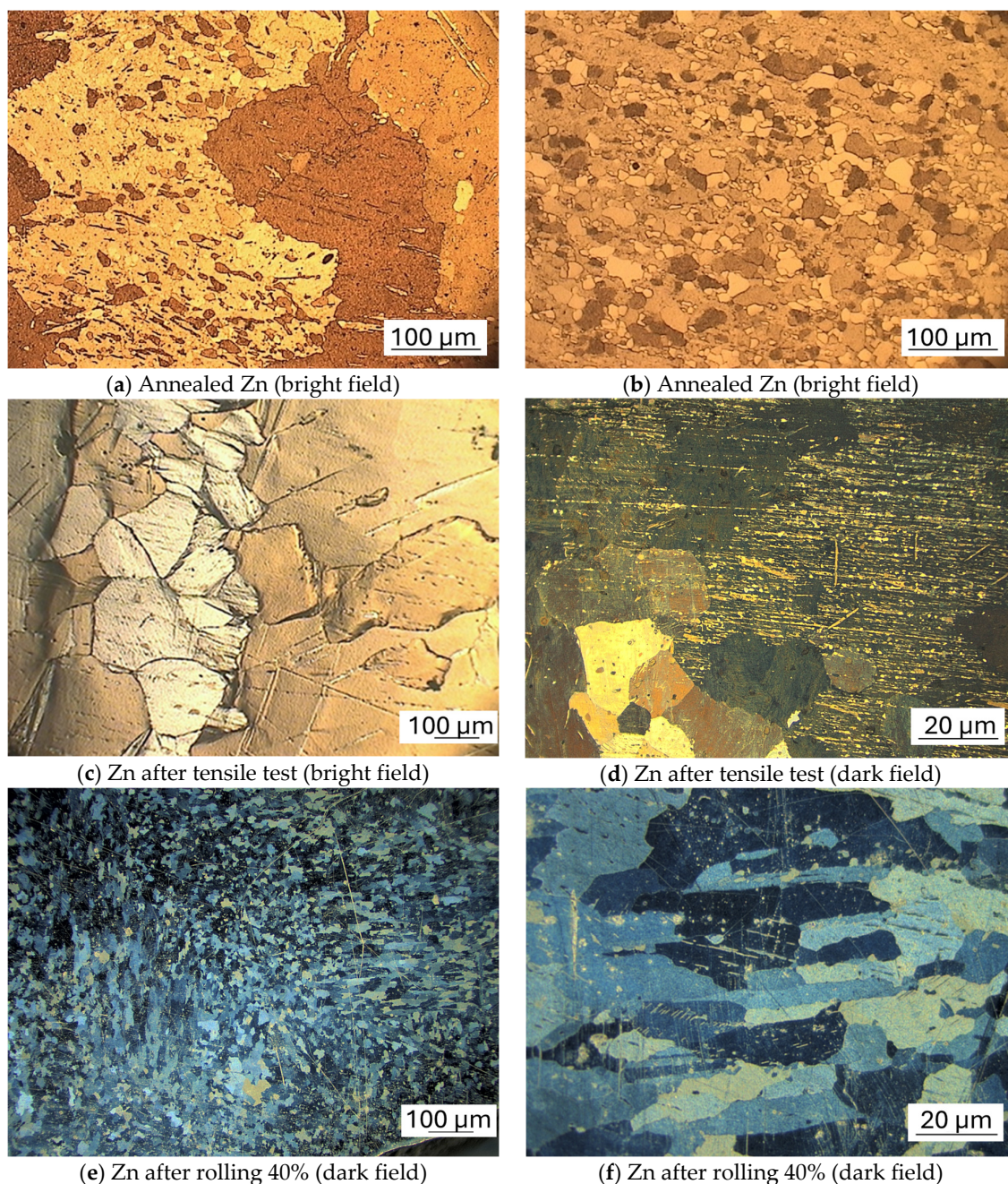


Figure 2. Optical micrographs of Zn: (a,b) annealed (200 °C, 30 min, WQ), (c,d) after tensile test (central region), (e,f) rolled 40% (RD vertical).

After deformation, both the rolled and the tensile-tested samples exhibit pronounced deformation localization with sub-grains and partially recrystallized grains (Figure 2c–f), consistent with the hcp lattice of Zn, which provides limited slip systems at room temperature and promotes heterogeneous plasticity and twinning. The relatively low recrystallization temperature of Zn further facilitates recovery/recrystallization during or shortly after deformation, yielding the mixed microstructure evidenced by optical microscopy.

X-ray diffraction measurements were performed on all the samples, after tensile test and rolled samples, close to the central region (maximum deformation). Figures 3 and 4 show the diffraction spectra of the annealed sample after tensile tests and rolled one, carried out in the central region.

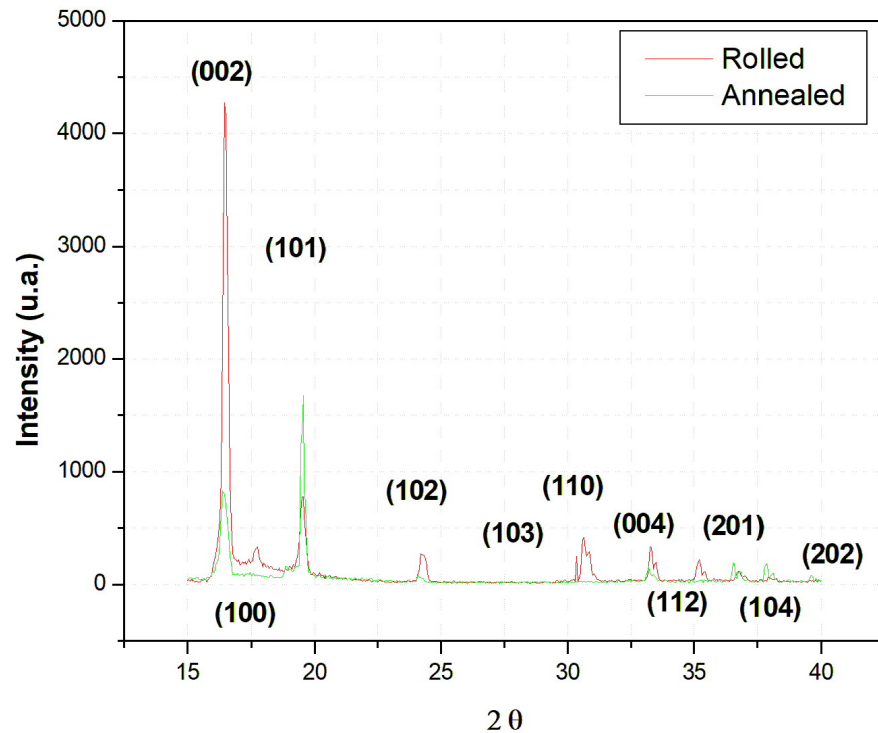


Figure 3. XRD spectra (2θ –intensity) recorded in the central region: annealed vs. rolled 40%. Main reflections are labeled.

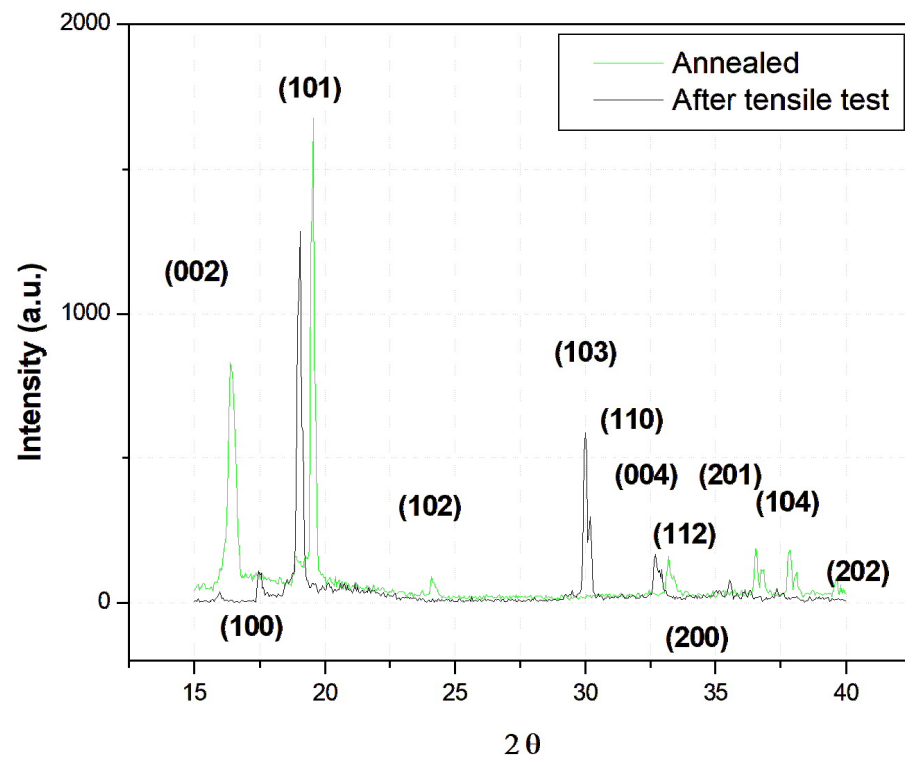


Figure 4. XRD spectra (2θ –intensity) recorded in the central region: annealed vs. after tensile test.

Table 1 shows the positions of the peaks, and Table 2 shows the relative intensities obtained from the spectra in Figures 3 and 4. The data in Table 2 show the variations in the relative intensities of the peaks after the two different types of deformation. In the case of tensile test, a moderate asymmetry of the precision peaks was also detected; this is determined by the presence of twins in the material. In the samples rolled up to 40%,

a significant variation in texture can be observed compared to the annealed sample; in the annealed sample, the most intense line is the (101), while in the 40% rolled sample, it is the (0 0 2) and the (0 0 4) reflections present in the annealed sample that reappear. A shift in the (1 0 1) and (0 0 4) peaks towards smaller angles is observed, consistent with the increase in interplanar distance related to the applied tensile strain. In the case of rolling, no angular displacement of the peaks was detected, compared to the material with random orientation, and in this case, twins were found as well. Quantitative peak positions (2θ , °) and shifts ($\Delta 2\theta$ vs. annealed) are summarized in Table 1. In the sample after tensile test, the common peaks (101) and (004) shift to lower angles by -0.500° , while (002) shifts by -0.450° and (100) shifts by -0.240° . In the rolled sample, shifts are very small and in the opposite direction (higher angles).

Table 1. XRD peak positions. (2θ , °).

(hkl)	Ref (4-831)	Annealed	Rolled 40%	After Tensile Tests
(002)	16.490	16.400	16.450	15.950
(100)	17.678	17.690	17.750	17.450
(101)	19.530	19.550	19.550	19.050
(102)	24.271	24.250	24.250	—
(103)	30.647	—	30.510	30.000
(110)	30.883	—	30.760	30.200
(004)	33.321	33.200	33.230	32.700
(112)	35.200	—	35.400	35.050
(200)	35.802	—	—	35.550
(201)	36.799	36.700	36.800	36.150
(104)	37.972	37.850	37.900	37.250
(202)	39.654	39.600	—	—

Table 2. Relative intensity of XRD peaks.

(hkl)	Ref (4-831)	Annealed	Rolled 40%	After Tensile Tests
(002)	53	50	100	3
(100)	40	7	8	8
(101)	100	100	18	100
(102)	28	5	6	—
(103)	25	—	6	46
(110)	21	—	9	23
(004)	2	9	8	13
(112)	23	—	5	3
(200)	5	—	—	6
(201)	17	11	3	4
(104)	3	11	2	4
(202)	5	5	—	—

In Table 2, texture strengthening can be highlighted by the comparison of the peak intensities. In particular, in the case of the tensile test sample, a variation in texture is evident, with the disappearance of the peaks (1 0 2) and (2 0 2) in comparison with the annealed sample, a significant reduction in intensity of the peaks (0 0 2), (2 0 1), and (1 0 4), and the appearance

of the peaks (1 0 3), (1 1 0), and (200). The peak (101) remains the dominant reflection in samples after tensile test, whereas rolling enhances texture with (002) dominance.

The analysis of the precision profiles of (1 0 1) peak, the most intense among the common one, allows to evaluate (Table 3) the variations in FWHM and the dislocations density (χ). From the peak-profile analysis (after $K\alpha_1/K\alpha_2$ deconvolution), FWHM values used for Williamson–Hall fits and Williamson–Smallman [13] dislocation density have been estimated. The peaks of the 40% rolled sample were broadened, compared to the annealed specimen, which was greater than that of the samples after tensile test. In particular, the (101) FWHM increases from $\beta(2\theta) = 0.115^\circ$ (annealed) to 0.160° (rolled) and 0.140° (after tensile test), corresponding to $\chi \approx 2.73 \times 10^6 \text{ cm}^{-2}$ (annealed), $3.03 \times 10^{11} \text{ cm}^{-2}$ (rolled), and $3.38 \times 10^{10} \text{ cm}^{-2}$ (after tensile test).

Table 3. FWHM $\beta(2\theta)$ and dislocation density values.

	Plane	FWHM $\beta(2\theta)$	$\xi \text{ (cm}^{-2}\text{)}$
Annealed	(1 0 1)	0.115	2.727×10^6
Rolled 40%	(1 0 1)	0.160	3.031×10^{11}
After tensile test	(1 0 1)	0.140	3.379×10^{10}

In both cases, rolling and tensile test, the phenomenon of recrystallization is evident and overlapped on work hardening. In fact, the dislocation density ξ is given by the sum of several factors, and recrystallization gives an opposite contribution to that of work hardening. So if one prevails over the other, a net decrease occurs. The difference of χ between the rolled and after tensile test samples is ascribable to different contribution of DRX to the defect accumulation. Furthermore, the FWHM reduction may also be due to the different texture variation observed in the spectra of the two deformed samples. Moreover, as confirmed by the metallography, the phenomenon of recrystallization is present in both considered cases of plastic deformation.

Average grain size (from line broadening) spans from sub-micrometer values in recrystallized regions, below optical resolution, to hundreds of microns, consistent with the observed heterogeneous mix of recrystallized and deformed grains. Once again, this suggests that in both deformation cases, there are contrasting contributions deriving from the different sizes of the recrystallized grains formed both during annealing and plastic deformation. Experimental results show that recrystallization has a more pronounced effect on the sample after tensile test compared to the 40% rolled sample. In fact, during tensile tests, recrystallization processes influence the dislocation density even more (3.379×10^{10}), which is approximately one order of magnitude lower than in the 40% rolled sample.

4. Conclusions

This work investigates plastic deformation in single-phase hcp Zn subjected to rolling (40% thickness reduction) and after tensile test by combining X-ray diffraction, Vickers microhardness, and optical metallography, and it provides quantitative links between diffractometric analysis and microstructural metrics.

Plastic deformation involves an increasing dislocation density and residual lattice strain, texture evolution, and twinning, often coupled with dynamic recrystallization.

Quantitatively, rolling enhances the texture, evidenced by $I(002)/I(101) = 100/18 \approx 5.56$ vs. 0.50 in the annealed state, whereas tensile test preserves (101) dominance but the second in intensity changes from (0 0 2) in the annealed sample to (1 0 3). Furthermore, tensile test produces systematic low-angle shifts [$\Delta 2\theta(101) = \Delta 2\theta(004) = -0.500^\circ$; $\Delta 2\theta(002) = -0.450^\circ$] ascribable to the increase in interplanar distance related to the applied tensile strain. The

(101) FWHM increases from 0.115° (annealed) to 0.160° (rolled) and 0.140° (after tensile test), yielding $\xi \approx 2.73 \times 10^6$, 3.03×10^{11} and $3.38 \times 10^{10} \text{ cm}^{-2}$, respectively. A huge distribution of average grain size has been found and the smallest one reaches $\sim 0.1 \mu\text{m}$, below the resolution of the optical microscopy. These results exhibit an apparent non-monotonic behavior: recrystallization partially offsets defect accumulation, particularly after the tensile test.

Recrystallization exerts a stronger influence in the tensile-mapped region (near failure) than in the rolled condition, consistent with the lower ξ , despite similar post-deformation hardness ($\sim 30\text{--}50 \text{ HV}$).

However, it should be noted that tensile test caused the material's failure, and the X-ray analysis was performed close to the failure zone, not in the maximum deformation zone, while rolling stress was analyzed up to a 40% reduction in sample thickness.

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