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Cryo-Rolled AA5052 Alloy: Insights into Mechanical Properties, Formability, and Microstructure

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Abstract: Industries operating in extreme conditions demand materials with exceptional strength, fatigue resistance, corrosion resistance, and formability. While AA5052 alloy is widely used in such industries due to its high fatigue strength and corrosion resistance, its strength frequently falls short of stringent standards. For AA5052 alloy, this study explores the combined use of solutionizing and cryo-rolling, followed by annealing, to improve strength. Although several alloys have been reported to undergo solution treatment before cryo-rolling, this study focuses on how post-processing via annealing can lessen the formability constraints usually connected to conventional cryo-rolling. The study sheds light on the ways that solutionizing, cryo-rolling, and annealing interact to affect the alloy's mechanical characteristics. Microstructure analysis shows that solutionizing improves the grain structure by reducing dynamic recovery, promoting dislocation density, and facilitating precipitate formation. Sheets subjected to solutionizing + cryo-rolling and partially annealed at 250 °C produce optimal results. Interestingly, formability is decreased when cryo-rolling alone is used instead of cold rolling, whereas formability is successfully increased when solutionizing is used. Comparing solutionized + cryo-rolled sheets that are partially annealed at 250 °C to cold-rolled sheets that are annealed at the same temperature, the former show notable quantitative improvements: a notable 17% increase in ultimate strength, a 10% boost in yield strength, and a noteworthy 13% enhancement in microhardness. Formability has improved with the solutionized + cryo-rolled specimens by annealing. This proposed approach led to noticeable gains in formability, hardness, and strength, which would significantly improve material performance for industrial applications.

Keywords: solutionizing; cryo-rolling; cold rolling; AA5052 alloy; partial annealing

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1. Introduction

The growing demand in industrial sectors such as automotive, marine, and aerospace for materials with exceptional mechanical properties, corrosion resistance, and formability has prompted extensive research into advancing aluminum alloys. AA5052 stands out among these alloys due to its high fatigue strength, excellent corrosion resistance, and formability [1]. However, the current AA5052 strength levels may not always meet the elevated requirements of these industries, limiting its widespread application.

To fill this void, scientists have investigated advanced processing techniques, with a particular focus on cryo-rolling (CYR). CYR involves exposing the material to extremely low temperatures during the rolling process, typically −196 °C, resulting in ultra-fine grain boundaries and a significant increase in material strength [2]. Despite the promising results of cryogenic rolling, finding the right balance of strength and formability remains a challenge. This has resulted in the use of a post-processing technique known as partial annealing, which aims to restore formability without sacrificing the increased strength obtained through CYR [3].

A dual-treatment approach involving Soln + CYR (solutionizing + cryo-rolling) processes has successfully exhibited significant improvements in mechanical properties as well as formability. In terms of yield strength, ultimate strength, and elongation, Soln + CYR specimens consistently outperform CYR samples [4]. Anas et al. have also studied the effect of various solutionizing temperatures ranging between 480 °C and 540 °C and for these specimens, the tensile strength and yield strength ranged between 292 MPa and 303 MPa and 245 MPa to 255 MPa, respectively. Stress–strain curves show increased plasticity and deformation capacity in Soln + CYR specimens, which support these enhancements. Limiting dome height (LDH) tests and forming limit curve (FLC) analysis demonstrate the formability of these specimens, which is critical for applications requiring intricate shaping [5,6].

This integrated approach aligns with the findings of Changela et al. [3], Feyissa et al. [5,6], Yogesha et al. [7], Kumar et al. [8], Chandra Sekhar et al. [9], Wang et al. [10], Satish et al. [11], Zuiko et al. [12], Shanmugasundaram et al. [13], Mishra et al. [14], and Panigrahi et al. [15], who have individually explored various aspects of cryogenic rolling and annealing processes on different aluminum alloys. The mechanical behavior and strengthening mechanisms of ultrafine-grained precipitation-hardened aluminum alloys have also been studied [16,17]. Cryogenic deformation has also been investigated as a means of improving the microstructure and mechanical properties of an Al-Cu-Mn alloy [18]. Babu et al. [19] investigated the impact of roller speed, diameter, and percentage reduction on several parameters including average rolling force, average rolling torque, maximum compressive residual stress, and load-carrying capacity. The influence of hydroforming processes on the formability of CYR AA5083 sheets was investigated by Raj et al. [20] and Raut et al. [21]. Dasgupta et al. describe the microstructural changes in a Ti-5Ta-1.8Nb alloy subjected to severe plastic deformation by CYR, including significant grain refinement, grain fragmentation, and the role of substitutional atoms [22]. Table 1 presents a concise summary of the literature review.

Table 1. Literature summary table.

Theme	Source	Relevance to Research Questions	Contribution and Insight	Identified Gaps
The Impact of CYR on Mechanical Properties	Changela et al. [3], Anas et al. [4], Feyissa et al. [5,6], Kumar et al. [8], Wang et al. [10], Zuiko et al. [12], Shanmugasundaram et al. [13], Mishra et al. [14]	RQ1 and RQ3	CYR improves mechanical properties such as hardness, tensile strength, and yield strength by reducing grain size and increasing dislocation density. CYR samples outperformed CR samples in terms of grain refinement.	There have been few studies on AA5052, particularly its microstructural evolution and effect on properties.
Partial Annealing and Formability Studies	Feyissa et al. [5,6], Chandra Sekhar et al. [9], Zuiko et al. [12], Sadeghi et al. [23]	RQ2 and RQ3	Following CYR, partial annealing improves formability and restores ductility. This process balances strength and formability in a variety of alloys, including AA5052, particularly at certain annealing temperatures.	A more in-depth investigation into the effects of various annealing temperatures on AA5052 is required.
Formability Testing and Comparison	Babu et al. [19], Raj et al. [20], Raut et al. [21], Achuthankutty et al. [24]	RQ4	Formability was investigated using hydroforming and related methods. CYR samples demonstrated better formability than CR ones, which is relevant for Erichsen cup testing.	Limited direct comparisons of formability curves between CYR and CR AA5052 samples.

As the demand for resilient materials surges, the outcomes of this research hold practical significance for crafting durable yet lightweight components. This is particularly crucial in sectors like aerospace, automotive, and defense, where advanced materials play a pivotal role. The study seeks to enhance the existing framework of aluminum alloy AA5052 by introducing a dual-treatment method (Soln + CYR) and incorporating a post-processing technique called partial annealing (with annealing temperatures set at 150 °C, 200 °C, 250 °C, and 300 °C). The focus is on highlighting the distinctive advantages of this approach in enhancing both mechanical properties and formability.

1.1. Objectives of the Study

This research is primarily dedicated to examining the effectiveness of a dual-treatment strategy that integrates Soln + CYR processes. Additionally, it incorporates a post-processing technique known as partial annealing, with annealing temperatures varying. The aim is to enhance the mechanical properties and formability of the AA5052 aluminum alloy. Specific objectives include:

1.1.1. Evaluation of Mechanical Properties

- Assess the impact of the dual treatment process and partial annealing temperatures on key mechanical properties, including yield strength, ultimate strength, and formability.
- Utilize LDH tests and forming limit curve (FLC) analysis to quantify and compare the formability of the different treatment processes.

1.1.2. Comparison with Conventional Processes

- Contrast the performance of the dual-treatment approach with conventional CYR processes, emphasizing the limitations addressed by the integrated method.
- Provide a comprehensive understanding of the advantages offered by the proposed Soln + CYR technique.

1.1.3. Microstructure Analysis

- Explore the microstructural changes induced by Soln + CYR, with a focus on precipitate formation.

1.2. Research Questions with Literature Summary

RQ1: How does Soln + CYR affect the mechanical properties of AA5052 alloy?
RQ2: How does partial annealing influence the formability of the Soln + CYR AA5052 alloy?
RQ3: How do the microstructures differ in CR, CYR, Soln + CYR samples?
RQ4: Compare formability curves arising from Erichsen cup testing.

This study pioneers a dual-treatment method combining solutionizing and cryogenic rolling (Soln + CYR), coupled with partial annealing at various temperatures. While prior research has explored CYR's benefits on mechanical properties, our work stands out by demonstrating an optimized balance of strength and formability in AA5052 alloy. This is further validated through advanced formability analyses such as LDH and FLC, which have been rarely applied in tandem for this alloy.

2. Materials and Methods Methodology

2.1. Sample Preparation

AA5052-H32 sheets with a thickness of 2 mm were obtained for the experimental study. To ensure accurate dimensions, the sheets were precisely cut into 50 × 80 mm samples using Wire-Electrical Discharge Machining (EDM). The as-received material exhibited a yield strength of 166 MPa, an ultimate tensile strength (UTS) of 237 MPa, and a percentage elongation of 11.1%.

2.2. Division into Batches

The samples were divided into three distinct batches to undergo different processing techniques, each starting with an initial thickness of 2 mm. Batch 1 was designated for CR, where the material was processed in a two-high mill roller, reducing the thickness to

1.5 mm (a 25% reduction) over 5 passes. Batch 2 samples were assigned for CYR, where they were first subjected to cryogenic treatment before rolling. Batch 3 samples followed a combined process of solutionizing, followed by CYR.

2.3. Cold-Rolling

Batch 1 samples were CR using a two-high mill roller. This process involved applying uniform pressure and reduction in thickness to achieve the desired thickness of 1.5 mm.

2.4. Cryo-Rolling

Cryogenic treatment was applied to Batch 2 samples by immersing them in liquid nitrogen (−196 °C) contained within a polystyrene box. Following a 30 min submersion, the samples were CYR to achieve the desired thickness of 1.5 mm.

2.5. Solutionizing + Cryo-Rolling

In the case of Batch 3, the samples were first solutionized at a temperature of 510 °C for 1 h to relieve internal stress. Following that, the material was quenched in water to increase the hardness. Following that, cryogenic treatment was performed by immersing the samples in liquid nitrogen for 30 min, followed by CYR to achieve the desired thickness of 1.5 mm. The schematic diagram of Soln + CYR is depicted in Figure 1.

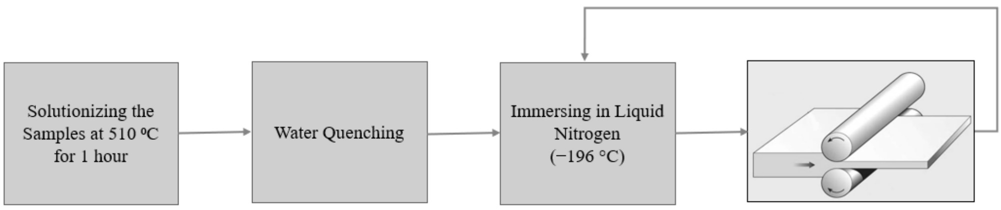


Figure 1. Soln + CYR Process.

By dividing the samples into distinct batches and subjecting them to specific treatments, the study aims to understand the effects of different processing techniques on the mechanical properties and microstructure of the AA5052. Figure 2 illustrates the comprehensive methodology employed in this research.

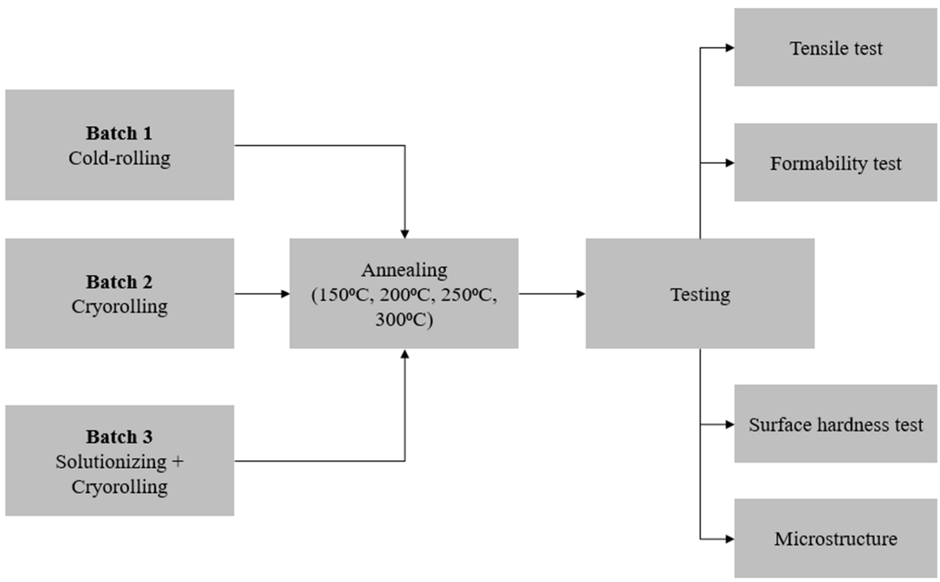


Figure 2. Methodology employed in this research.

2.6. Annealing Process

After the rolling process, all the workpieces obtained, whether from CR or CYR, or Soln + CYR (as depicted in Figure 2), underwent an annealing process at various temperatures. The purpose of the annealing process was to relieve any residual stresses induced during the rolling and to promote microstructural changes that could influence the material's mechanical properties. Different batches of workpieces were annealed at specific temperatures, ranging from 150 °C to 300 °C, for 90 min, followed by furnace cooling. This annealing step allowed for the optimization of the material's properties and formability, making it suitable for further testing and analysis.

2.7. Tensile Testing

Tensile tests were performed on specimens from all three batches. For consistent and reliable measurements, the samples were prepared following the ASTM E8/E8M standard [25] (shown in Figure 3). Tensile testing was used to compare the upper yield stress and plastic behavior of materials that had been annealed at various temperatures. The results of these tests will be discussed in the following section, which will provide valuable insights into the mechanical properties. Uniaxial tensile tests were conducted at an initial strain rate of 1 min^{-1} using a Tinius Olsen machine, operating with a constant crosshead speed.

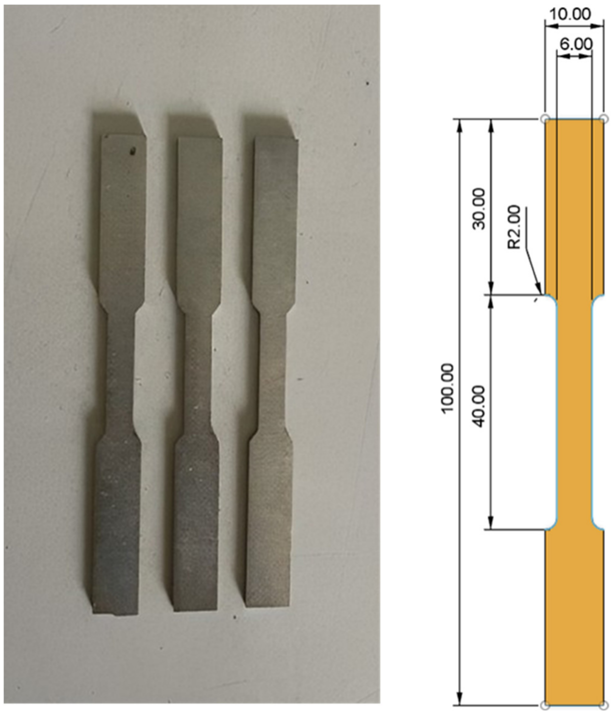


Figure 3. Samples prepared as per ASTM E8/E8M for tensile test.

2.8. Erichsen Cupping Test

An Erichsen cupping test was conducted on a CTM-II apparatus using annealed samples at various temperatures to evaluate the Limiting Dome Height (LDH) and assess the formability of the materials. In this test, the specimen was stamped with a rubber stamp with a 5 mm diameter circle. The stamped specimen was then placed in the Erichsen cup tester, which used a 20 mm indenter to apply pressure to the material. The indenter's pressure caused a bulge in the specimen, which eventually formed into a cup shape. The procedure was repeated until the specimen was on the verge of cracking at its weakest point along the circumference, where the indenter was inserted. The stamped circles on the samples were precisely 5 mm in diameter. This test aimed to assess the material's formability and its ability to withstand deformation without failure.

The height of the indented cup was measured using the LDH method. This method involves utilizing a set of dials integrated with the Erichsen cup testing apparatus. Furthermore, stamped circles within the cupped region were carefully observed using a profile projector, which was used to accurately measure their diameters. The major and minor strains of the circles were then measured. These strain values were then plotted on a graph to reveal information about the material's formability and ability to withstand deformation without failure during the Erichsen cupping test. Along with precise measurements and graphical analysis, the LDH method contributes to a comprehensive evaluation of the material's behavior under various annealing conditions. Figure 4 shows the Erichsen cupping test samples.

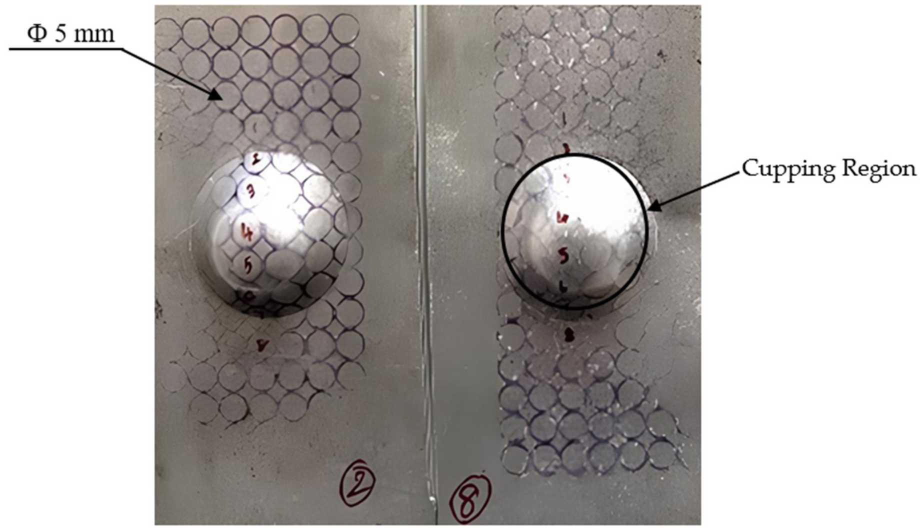


Figure 4. Erichsen cupping test samples.

2.9. Hardness Test

The Vickers hardness test method was used to assess surface hardness using a microhardness tester. The hardness value is determined by applying a specific load to the surface of the specimen with a diamond indenter and then measuring the indentation depth. The study sought to assess the effect of various thermal treatments on the surface hardness of the material. To accomplish this, specimens from all batches were annealed at various temperatures, including CR, cryogenic rolling, and Soln + CYR samples. The study aimed to gain valuable insights into the mechanical behavior of the aluminum alloy under different rolling and annealing conditions by analyzing the hardness values obtained from these tests.

2.10. Microstructural Examination Procedure

The specimens in the microstructure study were meticulously prepared to observe their internal characteristics. Initially, the samples were cut into 16 $\times$ 16 mm pieces and polished with emery paper ranging from 200 grit to 1200 grit. This step was essential for removing surface scratches and achieving a smooth, mirror-like finish. Following that, the diamond paste was used to further polish the specimen's face, enhancing its lustrous appearance, and making it easier to observe under an optical microscope.

X-ray Diffraction (XRD) analysis was conducted to examine the phase composition in the processed samples. The measurements were performed using a Rigaku Ultima IV XRD system operated at 40 kV, configured for powder, thin film, and small-angle X-ray scattering (SAXS) applications. The scans covered a 2θ range of 20° to 90° with a step size of 0.02° and a scan speed of 2°/min. Samples were prepared by mechanically polishing their surfaces to ensure uniformity. The diffraction patterns obtained were analyzed against standard files to identify phases.

3. Results and Discussions

3.1. Mechanical Properties of Rolled Sheets

The Soln + CYR specimens consistently outperform the CR and CYR groups in terms of yield strength, ultimate tensile strength, and percentage elongation across all annealing temperatures, as shown in Table 2. This enhanced performance is attributed to the synergistic effects of the Soln + CYR processes.

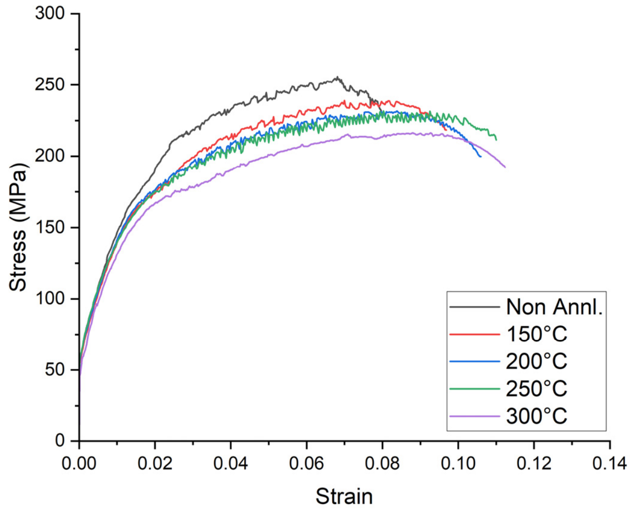
Table 2. Tensile testing results of various specimens.

Annl. Temp °C	CR			CYR			Soln + CYR		
	YS (MPa)	UTS (MPa)	Elng. %	YS (MPa)	UTS (MPa)	Elng. %	YS (MPa)	UTS (MPa)	Elng. %
Non Annl.	200	255	8	210	261	5.5	230	291	9.1
150	180	239	9.7	182	245	9.2	199	276	12.7
200	169	231	10.8	180	238	9.6	184	263	13.0
250	166	229	11.0	178	235	10.0	183	268	13.5
300	150	216	11.9	157	219	10.8	176	244	13.7

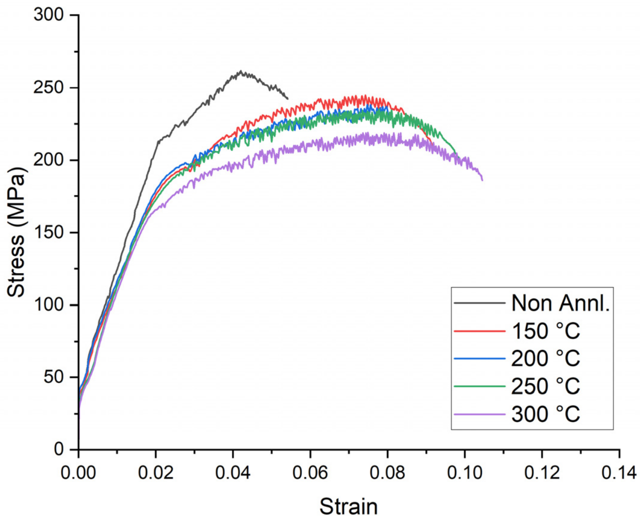
As displayed in Table 2, both Yield Strength (YS) and Ultimate Tensile Strength (UTS) decrease across all processing methods as the annealing temperature increases. For instance, in the CR samples, YS drops from 180.1 MPa at 150 °C to 149.6 MPa at 300 °C, while UTS declines from 238.8 MPa to 216.2 MPa. Similar reductions are observed in CYR samples, with YS decreasing from 182 MPa to 157 MPa and UTS from 245 MPa to 219 MPa over the same temperature range. The Soln + CYR samples exhibit relatively higher strength retention compared to CYR samples alone, with YS decreasing from 199 MPa to 176 MPa and UTS from 276 MPa to 244 MPa as the temperature rises from 150 °C to 300 °C. Although the Soln + CYR samples show a slightly larger absolute reduction in UTS (31 MPa), their mechanical properties demonstrate better stability at elevated temperatures, likely due to the combined effects of solution treatment and CYR. For the non-annealed specimens, the initial YS and UTS values are 200 MPa and 255 MPa for CR, 210 MPa and 261 MPa for CYR, and 230 MPa and 291 MPa for Soln + CYR, respectively. Among these, Soln + CYR shows the best combination of strength and ductility with an elongation of 9.1%. Additionally, elongation generally increases with temperature, demonstrating improved ductility, with the Soln + CYR samples achieving the highest elongation values, increasing from 12.7% at 150 °C to 13.7% at 300 °C. This indicates that the combined process provides an optimal balance of strength and ductility compared to the other processing methods.

Solutionizing at 510 °C for one hour effectively relieves internal stress and promotes a uniform microstructure in the material. The reduction in induced stresses during annealing and grain structure refinement after CYR contributed to enhanced mechanical properties. The refined grain structure improves yield strength through grain boundary strengthening (Hall–Petch effect), while the alleviation of residual stresses during solution treatment allows for improved elongation by enabling uniform plastic deformation. Solution treatment prior to CYR dissolves solute particles and homogenizes the microstructure, reducing microstructural inconsistencies and promoting more uniform deformation during the subsequent CYR process. This process, conducted by submerging samples in liquid nitrogen, further refines the grain structure, producing ultra-fine grains and enhancing strain hardening. In contrast, CR or CYR samples starting from the as-received state retain pre-existing microstructural inhomogeneities, which limit their yield strength despite the higher accumulated strain. Consequently, the combination of solution treatment and CYR results in superior mechanical properties, as evidenced by the higher yield and ultimate strengths of the Soln + CYR samples compared to CR or CYR samples alone [26].

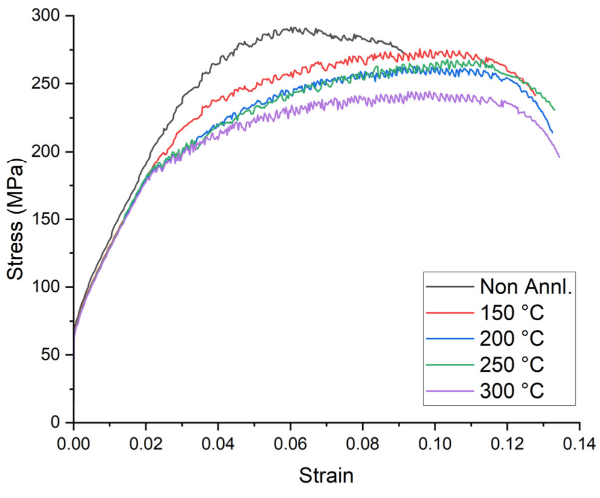
The stress–strain plots depicted in Figure 5a–c complement the findings from the mechanical property comparison. The stress–strain curves of the Soln + CYR specimens exhibit a more significant plastic region, indicating their ability to undergo greater deformation before failure. This improved plastic behavior aligns with the higher % elongation values observed in the Soln + CYR group.



(a)



(b)



(c)

Figure 5. (**a**) Stress v/s strain graph of CR specimens. (**b**) Stress v/s strain graph of CYR specimens. (**c**) Stress v/s strain graph of Soln + CYR specimens.

The LDH test was conducted to evaluate the formability and stretchability of the specimens after undergoing various thermal treatments. The LDH values, representing the height of the indented cup, were measured for the CR, CYR, and Soln + CYR specimens at different annealing temperatures.

The LDH values for each specimen group at different annealing temperatures are shown in Figure 6. The plot reveals that the Soln + CYR specimens consistently displayed the highest LDH values across most annealing temperatures. At 150 °C, the Soln + CYR specimens exhibited an LDH value of 7.05 mm, while the CR and CYR specimens had LDH values of 6.56 mm and 5.78 mm, respectively.

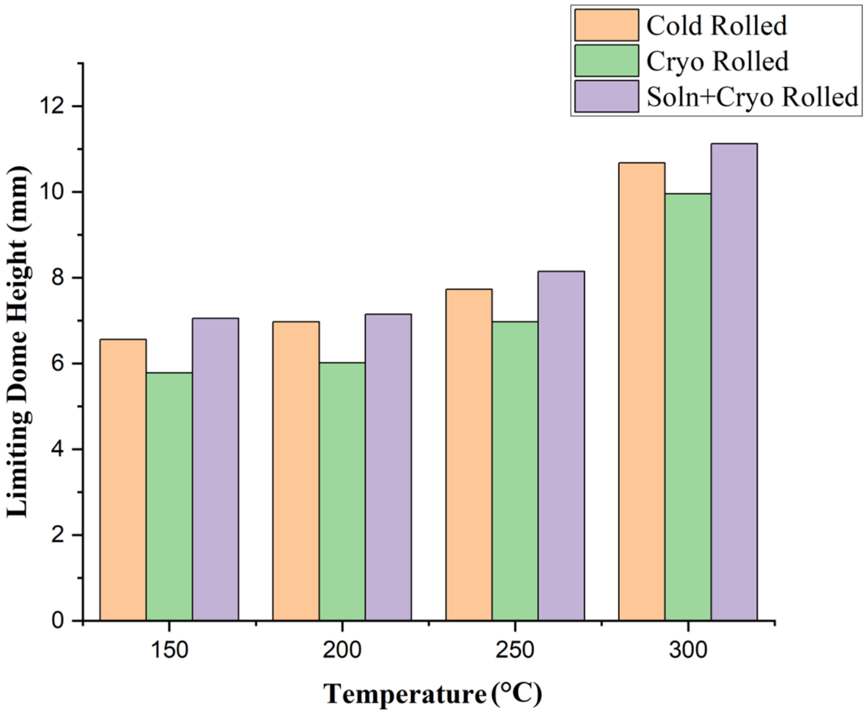


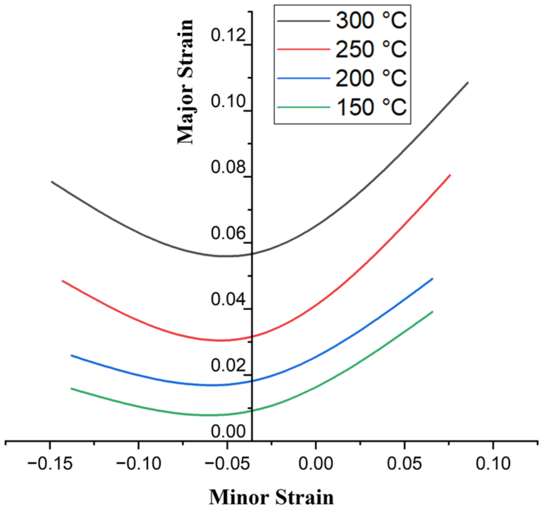
Figure 6. LDH Results for rolled specimens annealed at different temperatures.

Similarly, at 200 °C, 250 °C, and 300 °C the Soln + CYR specimens demonstrated higher LDH values compared to the other groups. Figure 7a–c represent the FLCs for CR, CYR, and Soln + CYR specimens, respectively. These FLCs provide essential insights into the formability of the rolled sheets under various annealing conditions.

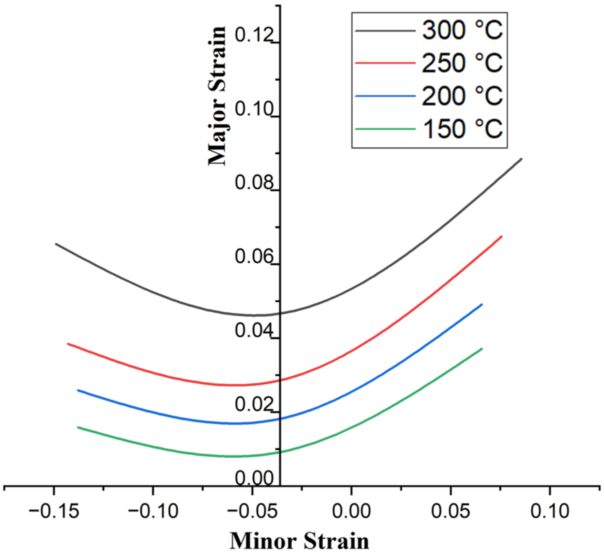
The results clearly indicate that the annealing temperature plays a significant role in influencing the formability of the rolled sheets. Among the three cases, the Soln + CYR specimens exhibit the most favorable formability results.

This trend is consistent with the LDH results, where the LDH values for the Soln + CYR specimens are higher than those of the other two groups. The improved formability observed in both the FLC and LDH results for the Soln + CYR specimens can be attributed to the combined effect of Soln + CYR. Solutionizing helps in relieving internal stress and achieving a more uniform microstructure, while CYR contributes to grain refinement and enhanced mechanical properties.

The Vickers hardness test results consistently emphasize that the Soln + CYR specimens achieve consistently elevated levels of hardness compared to the other groups, specifically the CR and CYR specimens (as depicted in Figure 8). The solutionizing procedure, which is carried out for one hour at 510 °C, and the quenching that follows, significantly increases the hardness of the material.



(**a**)



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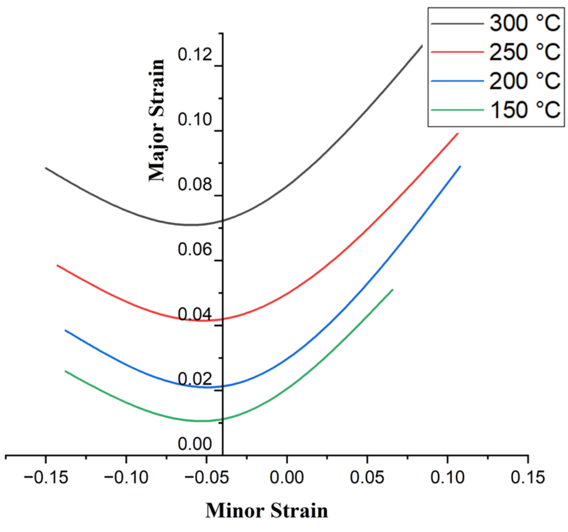

(**c**)

Figure 7. (**a**) Forming limit curve of CR specimens. (**b**) Forming limit curve of CYR specimens. (**c**) Forming limit curve of Soln + CYR specimens.

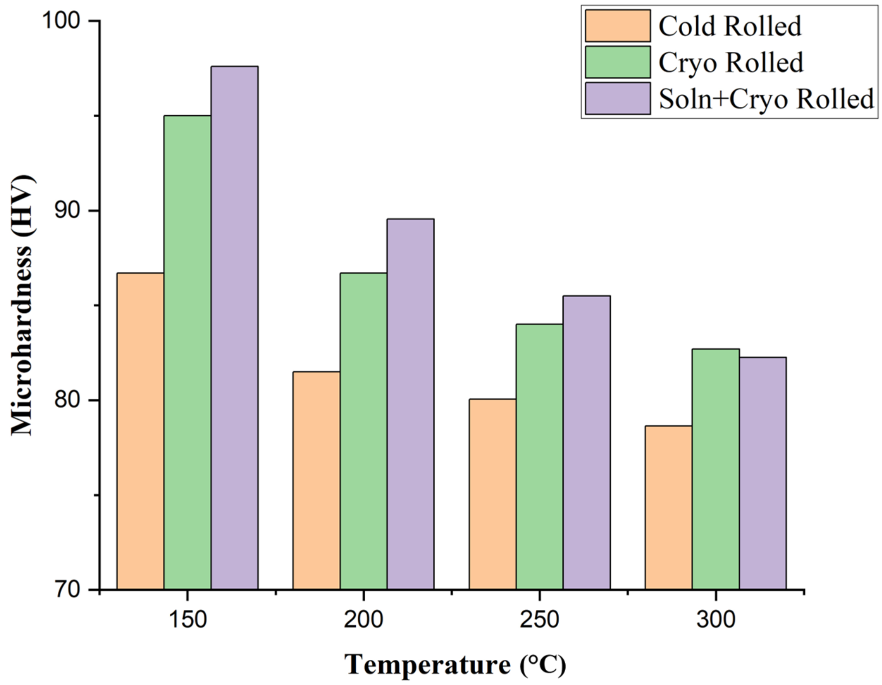

Figure 8. Microhardness Comparison of Rolled Specimens Annealed at Different Temperatures.

Moreover, there is a noticeable downward trend in the Vickers hardness values with an increase in annealing temperature. The purpose of this annealing procedure is to cause microstructural changes and relieve any residual tensions created during the rolling process. These data are consistent with the anticipated decrease in dislocation density that frequently results from increasing the annealing temperature.

At 300 °C, the slightly higher microhardness of CYR compared to Soln + CYR can be attributed to localized surface effects. CYR samples retain a higher density of dislocations near the surface, which are only partially relaxed during annealing. This residual dislocation density and sub-grain structure enhance surface hardness, even though the bulk material exhibits lower YS and UTS compared to Soln + CYR.

3.2. Microstructural Observations

The microstructures seen in CR, CYR, and Soln + CYR specimens following annealing at 150 °C are shown in Figure 9a, Figure 9b, and Figure 9c, respectively. The microstructure of the base material reveals that the primary phase in the AA5052 alloy is α-Al, accompanied by the secondary intermetallic phase β-Mg_2Al_3 [1]. Notably, the microstructure of CYR and Soln + CYR specimens (Figure 9b,c) exhibit a noticeable variation in the β-Mg_2Al_3 intermetallic phase compared to the CR condition. While Figure 9a shows a lower density of β-Mg_2Al_3 precipitates, Figure 9b,c highlight that the size and distribution of these precipitates differ significantly across the conditions. The β-Mg_2Al_3 intermetallic phase influences the mechanical properties of the AA5052 alloy primarily through its role in impeding dislocation motion. The observed microscale precipitates contribute to strengthening via particle hardening mechanisms, with their distribution and size playing a key role in enhancing yield strength and hardness.

Figure 10 confirms the presence of both the primary phase (α-Al) and a secondary intermetallic phase (β-Mg_2Al_3) in the AA5052 base material. Analysis of the XRD patterns reveals the dominant peak, consistent with previous findings by Anas et al. [4] where a similar phase distribution was observed in a Soln + CYR sample.

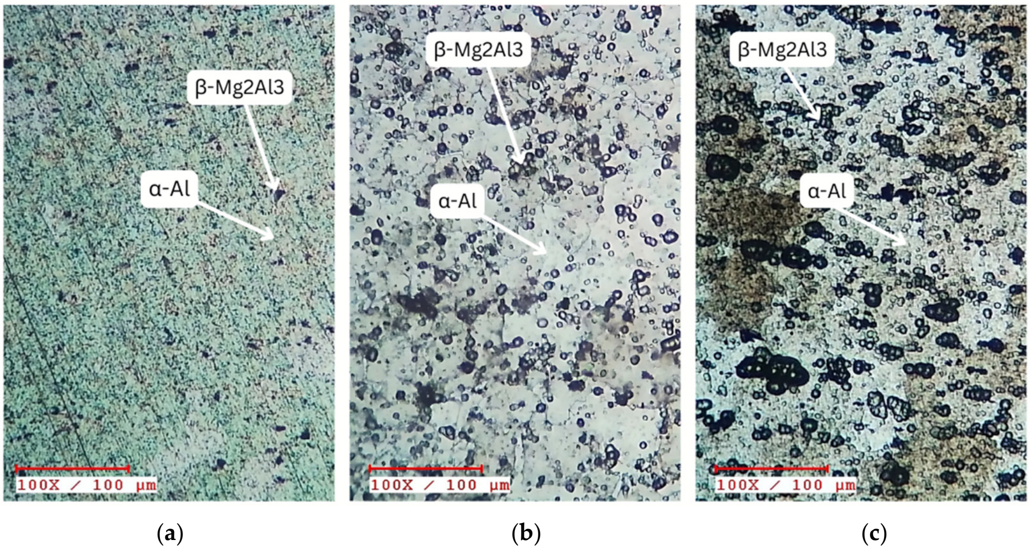

Figure 9. (**a**) Microstructure of CR AA5052 annealed at 150 °C. (**b**) Microstructure of CYR AA5052 annealed at 150 °C. (**c**) Microstructure of Soln + CYR AA5052 annealed at 150 °C.

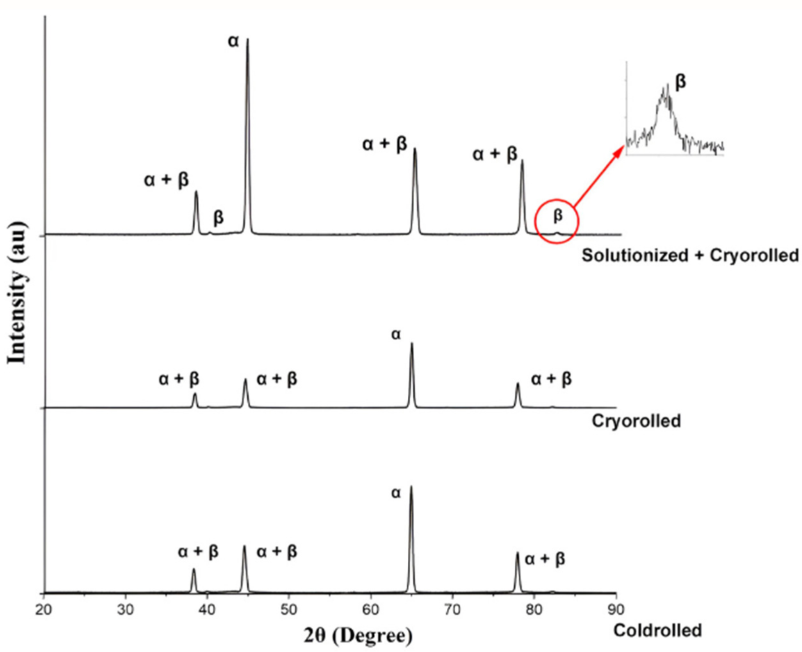

Figure 10. XRD plot for rolled specimens annealed at 150 °C.

4. Conclusions

- Dual Treatment Synergy: This study demonstrates that the mechanical properties and formability of aluminum alloy AA5052 can be effectively enhanced by combining Soln + CYR processes. A comparison of specimens subjected to CYR with those that underwent both CR and Soln + CYR shows a significant reduction in formability.
- Mechanical Performance: Specimens that were Soln + CYR and then annealed at 250 °C had superior mechanical qualities. Soln + CYR specimens annealed at 250 °C exhibit a 10% increase in yield strength, a 17% increase in ultimate strength, a 2% improvement in formability, and a 13% improvement in microhardness when compared to CR sheets annealed at the same temperature.
- Formability: LDH tests and forming limit curve (FLC) studies demonstrated that Soln + CYR specimens undergoing annealing have acceptable formability features,

which are important for applications needing complex shaping. Comparing Soln + CYR specimens to CR specimens at different annealing temperatures, about 5% improvement was seen. For Soln + CYR specimens, an increase in annealing temperature from 150 °C to 300 °C resulted in a roughly 36% improvement in formability.
- Enhanced Plasticity: Stress–strain curves support the mechanical property improvements, revealing increased plasticity and deformation capacity in the Soln + CYR specimens.
- Microstructure Insight: Microstructure study confirms mechanical property enhancements, showing a higher density of Al-Mg precipitates in the α-aluminum solid-solution matrix of Soln + CYR specimens.
- The Soln + CYR process strikes an optimal balance between strength and formability, making it ideal for industries like automotive and aerospace, where both are crucial. By improving strength and ductility through controlled annealing, this process enables the production of lightweight, high-performance components that can undergo complex shaping, offering a promising solution for advanced manufacturing applications.

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Abbreviations

Nomenclature/Abbreviation	Description
YS	Yield Strength
UTS	Ultimate Tensile Strength
Elng. %	Percentage Elongation
Annl. Temp.	Annealing Temperature
CR	Cold Rolling
CYR	Cryo Rolling
Soln	Solutionizing

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