

Research Article

Microstructure Evolution and Fatigue Behaviour of AA2099 Alloy Processed through Hybrid Multi-Axial Forging and Cryo-Rolling

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Received 21 November 2025

Revised 27 December 2025

Accepted 30 December 2025

Abstract:

The effects of post-deformation annealing and hybrid severe plastic deformation (SPD)—multi-axial forging (MAF) followed by cryorolling (CR)—on the microstructural development and low-cycle fatigue (LCF) behavior of AA2099 Al-Cu-Li alloy were examined. After six MAF cycles (cumulative strain of 3.6), solution-treated specimens underwent cryo-rolling at -196°C , leading to an overall thickness reduction of about 90%. Post-deformation heat treatment was then carried out at 150°C and 200°C . As a result of this processing route, the material developed an ultrafine-grained microstructure with an average grain size close to $0.42\text{ }\mu\text{m}$, along with a high density of dislocations and a microstructure dominated by low-angle grain boundaries. Grain size was increased to $0.84\text{ }\mu\text{m}$ after post-annealing at 150°C , which also improved texture intensity ($\sim 3.7\text{ m.r.d.}$) and the high-angle grain boundary fraction. Because of the best precipitation strengthening, the MAF+CR+ 150°C condition demonstrated the best LCF performance, with the biggest hysteresis loops, maximum energy dissipation, and a peak cycle stress of about $\pm 270\text{ MPa}$. For MAF+CR+ 150°C , fracture analysis showed primarily ductile failure with fine equiaxed dimples, whereas MAF+CR+ 200°C displayed coarser features as a result of overaging. Progressive strain recovery with temperature was verified using Kernel Average Misorientation analysis. In ultrafine-grained Al-Cu-Li alloys intended for aerospace applications, this hybrid processing method successfully modifies microstructure to produce improved LCF resistance.

Keywords: AA2099 aluminium alloy, Hybrid multi-axial forging (MAF)–cryo-rolling (CR), Mechanical anisotropy, Low-cycle fatigue, EBSD analysis

1. Introduction

By permitting substantial grain refinement and property enhancement by extreme deformation of the plastic, the invention of SPD (Severe Plastic Deformation) techniques in the 1950s transformed metal production [1]. Equal Channel Angular Pressing (ECAP) [2,3], Multi-Axial Forging (MAF) [4,5], Accumulative Roll Bonding (ARB) [6], High-Pressure Torsion (HPT) [7,8], and cryorolling [9] are all included in SPD. Although these techniques significantly enhance fatigue resistance, strength, and hardness, they often have high production and complex equipment requirements [10,11]. Among them, multi-axial forging (MAF) is very appealing since it effectively creates ultrafine-grained (UFG) structures by applying high compressive true strain [12,13]. Multi-Axial Forging (MAF) has several practical and technological drawbacks, despite being a successful technique for producing Ultrafine Grained (UFG) materials through Severe Plastic Deformation (SPD). The procedure is time-consuming and less suitable for large-scale production, as it requires several successive deformation processes along multiple axes.

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To preserve microstructural consistency, each forging cycle necessitates exact control over temperature, strain path, and strain rate, which increases process complexity. Moreover, oxidation, heat loss, and dimensional errors might result from frequent handling and warming in between passes. Grain refining is heterogeneous because the accumulated strain is frequently not uniform across the sample, especially in the vicinity of edges and corners. Due to the requirement for high-capacity presses that can apply significant deformation stresses, MAF also entails significant tooling and equipment expenditures. These drawbacks limit its industrial application, particularly when compared with alternative SPD techniques, such as cryorolling or accumulative roll bonding, which provide greater productivity and easier setups.

Ultrafine-grained (UFG) metallic materials with outstanding mechanical performance can be produced very effectively by combining Multi-Axial Forging (MAF) and Cryorolling (CR) as a hybrid Severe Plastic Deformation (SPD) approach. MAF promotes a uniform distribution of strain and the start of grain refining throughout the bulk material by facilitating the introduction of significant cumulative strains by serial forging along different axes. The low-temperature deformation efficiently reduces dynamic recovery when this pre-deformed material is cryorolled, which further increases dislocation density and improves grain refinement and strengthening. This hybrid approach minimizes process restrictions inherent to individual techniques, such as limited strain accumulation in CR and non-uniform deformation in MAF, while also accelerating the attainment of UFG structures. The MAF-CR hybrid process is therefore a viable technique for large-scale processing of high-performance aluminum alloys and other structural materials since it produces a better balance between strength and ductility, enhanced thermal stability, and refined microstructural uniformity. In order to create ultrafine-grained (UFG) structures in aluminum and related alloys, hybrid severe plastic deformation (SPD) methods have recently attracted a lot of interest. To attain improved mechanical characteristics and microstructural uniformity, combinations such multi-axial forging (MAF) followed by rolling or cryo-rolling (CR) have been successfully used. According to Bhatt et al. [14], a hybrid MAF–CR process was employed to manufacture UFG AA2099 alloy, which exhibited notable increases in high cycle fatigue strength and impact toughness as a result of the refinement and equiaxed grain structure. Similar hybrid deformation techniques, such as MAF followed by hot rolling, have been investigated in Mg–Zn–Gd alloys to enhance fracture toughness and tensile strength through grain refinement and texture alteration [15]. In Cu–Ti alloys, Ramesh et al. [16] employed a combined MAF and rolling process, which yielded increased durability and resistance to corrosion as a result of the development of sub grains and an increased dislocation density. Additionally, hybrid cryo-deformation and annealing treatments have been effectively applied to improve the ductility and fatigue life of Al-based alloys [17,18]. Together, these investigations demonstrate that incorporating multi-step cryo-deformation pathways, like hybrid MAF–CR processing, can successfully modify microstructure, regulate anisotropy, and enhance fatigue resistance in lightweight aluminum–lithium systems.

While AA2099 alloys' mechanical performance under fatigue loading has been extensively studied, its mechanical anisotropy and fracture behavior after cryogenic deformation have received less attention. The development of ultrafine-grained (UFG) structures, which improve strength and fatigue resistance, has shown great promise using cryo-rolling and related severe plastic deformation (SPD) processes. However, the majority of earlier research has concentrated on single-step cryo-deformation or traditional thermo-mechanical processing; for Al–Cu–Li alloys like AA2099, the impact of hybrid SPD routes, which combine cryo-deformation with subsequent thermal treatments, is still mainly unknown. Furthermore, it is currently unclear how anisotropy and low-cycle fatigue (LCF) behavior are influenced by cryo-deformation-induced substructures, misorientation distributions, and grain boundary features. By methodically examining the microstructural evolution, anisotropy, and mechanical performance of AA2099 produced using a hybrid SPD approach that incorporates cryo-deformation and post-deformation annealing, the current study fills in these gaps. The central aim of this study is to achieve ultrafine-grained microstructures and to evaluate how multi-axial forging followed by cryo-rolling, together with subsequent annealing, influences microtexture development, grain boundary character, kernel average misorientation (KAM), and low-cycle fatigue (LCF) performance. To create a thorough relationship between processing, microstructure, and mechanical behavior, mechanical and microstructural features were examined using LCF testing with assistance from optical microscopy, SEM, and EBSD.

The use of a hybrid cryo-deformation-based thermo-mechanical method for AA2099, which has not been widely documented in the literature, is what makes this study new. By showing how controlled cryo-rolling and subsequent low-temperature annealing can modify grain boundary character, reduce mechanical anisotropy, and improve fatigue resistance in advanced Al–Cu–Li alloys, this integrated approach offers new insights into the microstructure–anisotropy–fatigue relationship.

2. Experimental Setup

Bulk AA2099 (Al–Cu–Li) alloy was purchased from Bharat Aerospace Metals in Mumbai, India, and machined into prismatic specimens measuring 27 mm by 30 mm by 33 mm under four distinct processing conditions: solution-treated (ST), multi-axial forging followed by cryo-rolling MAF + CR and MAF + CR followed by annealing at 150°C = MAF+CR+150, and MAF+CR followed by annealing at 200°C = MAF+CR+200. Table 1 displays the alloy's chemical composition, while Table 2 lists the processing routes along with the relevant acronyms.

To achieve a homogenized microstructure, all specimens were solution treated at 530°C for two hours and then water quenched. Figure 1 shows a schematic illustration of the MAF and CR processing routes used in this investigation.

With a total strain of 0.6 each cycle, each MAF cycle comprised three successive compressions along mutually perpendicular axes (actual strain of ~0.2 per pass). After six cycles of MAF, a total accumulated strain of 3.6 was obtained. Figure 1 illustrates a schematic of a complete MAF cycle. To achieve a thickness reduction of up to 90%, the forged samples were further sectioned into plates (30 mm × 25 mm × 10 mm) along the rolling direction and cryo-rolled (CR). Liquid nitrogen was used as the cooling medium during cryo-rolling, which was done at about -196°C. The MAF+CR samples were then annealed in an air-circulating furnace for 45 minutes at 150°C and 200°C. Samples for microstructural examination were mechanically polished using SiC sheets (320–2000 grit) and then electropolished in order to prepare them for EBSD characterization. A Quanta 400 Focused Ion Beam Scanning Electron Microscope was used for the EBSD and SEM studies.

3. Data Processing

The gauge sections were physically polished to remove surface flaws, and tensile and low-cycle fatigue (LCF) specimens were machined along the rolling direction. With a strain ratio ($R = -1$) and total strain amplitudes ranging from 0.2% to 0.6%, all LCF experiments were carried out at room temperature under strain-controlled conditions at a frequency of 0.25 Hz.

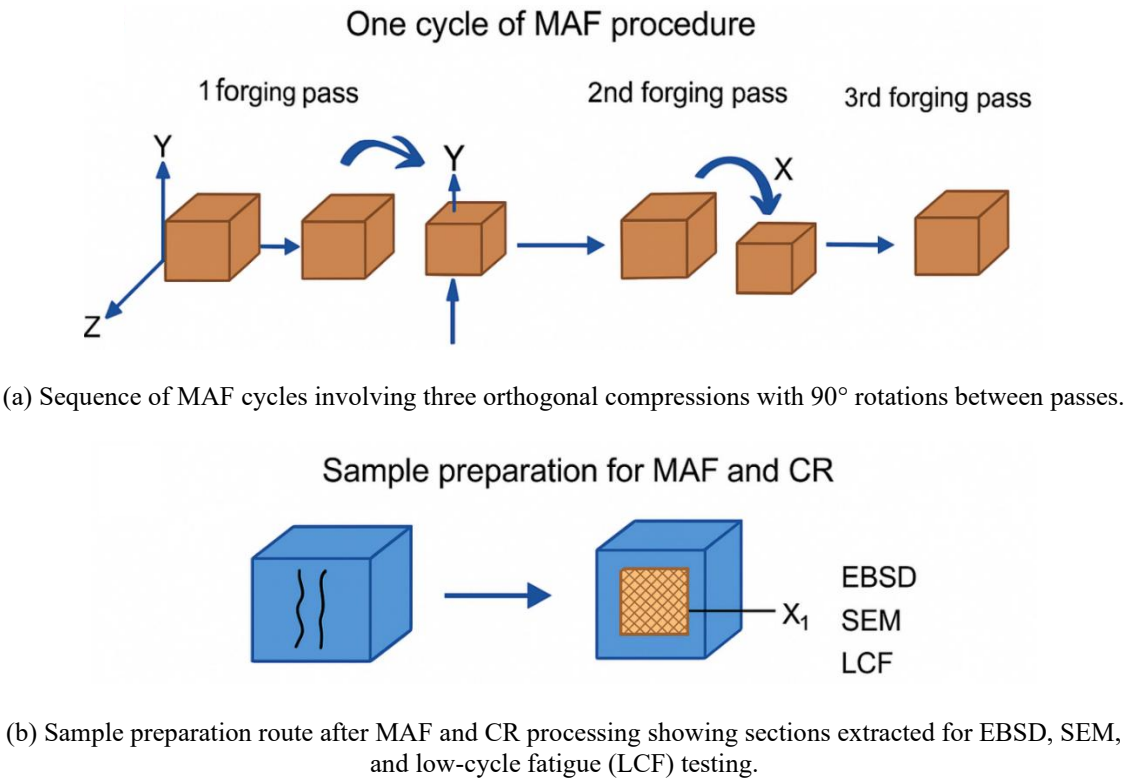


Fig. 1. Schematic representation of the multi-axial forging (MAF) and cryo-rolling (CR) processing route followed in the present study.

Table 1: Nominal chemical composition of the Al 2099 aluminium–lithium alloy used for the present investigation.

Elements	Cu	Li	Zn	Mg	Mn	Zr	Al
Wt%	2.4-3.0	1.6-2.0	0.4-1.0	0.1-0.5	0.1-0.5	0.05-0.12	Balance

Table 2: Details of processing routes and abbreviations used for hybrid cryo-deformation and annealing treatments of AA2099 alloy.

Abbreviation	Description
ST	Solution-treated at 535 °C for 2 h
MAF + CR	Solution-treated at 535 °C followed by cryo-rolling to a 90% thickness reduction
MAF + CR + 150 °C	Solution-treated at 535 °C, cryo-rolled to 90% thickness reduction, and subsequently annealed at 150 °C
MAF + CR + 200 °C	Solution-treated at 535 °C, cryo-rolled to 90% thickness reduction, and subsequently annealed at 200 °C

4. Results and Discussion

4.1 Electron backscatter diffraction (EBSD) Microstructure and Texture Evolution of Al 2099 Alloy after Hybrid SPD and Annealing

Figure 2 shows the EBSD microstructures of Al 2099 alloy under different processing settings after hybrid severe plastic deformation (SPD) and post-deformation annealing. Grain refinement, crystallographic reorientation, and microstructural evolution under various deformation and annealing settings are highlighted by the inverse pole figure (IPF) maps in Figure 2. The transition from solution treatment (ST) to MAF+CR processing results in significant grain size reduction and textural alterations, which are then further altered by annealing at high temperatures [19–21]. Inverse pole figure (IPF) maps corresponding to the solution-treated (ST) state, the MAF + CR processed condition, and the specimens annealed at 150°C and 200°C after multi-axial forging + cryo-rolling. samples are shown in Figures 2a–d. The MAF+CR condition shows elongated ultrafine-grained (UFG) structures with refined average grain size ($\sim 0.42 \mu\text{m}$), whereas the ST sample shows relatively coarse, equiaxed grains. Grain size increases to about $0.84 \mu\text{m}$ and $0.75 \mu\text{m}$, respectively, 150°C and 200°C after annealing, although grains maintain an elongated shape. Following annealing, high-angle grain boundary (HAGB) fractions rise concurrently (Figures 2g, 2h), suggesting partial recrystallization [22–23]. All things considered, these IPF maps support notable texture evolution and grain fragmentation brought about by the applied thermomechanical procedure [24].

The distribution of grain boundary misorientation angles for each of the four situations is shown in Figure 3. All deformed samples show a significant population of low-angle grain boundaries (LAGBs, less than 15°), with MAF+CR samples showing the largest LAGB proportion, indicating widespread dislocation structures [25]. The fraction of high-angle grain boundaries (HAGBs) slightly increases after annealing, indicating the start of recrystallization processes and structural recovery, adjusting the grain boundary character for better mechanical properties [26]. For MAF+CR and post-annealed samples, texture evolution was investigated using $\{001\}$, $\{111\}$, and $\{110\}$ pole figures, as seen in Figure 4. A weak, dispersed texture with a maximum intensity at roughly 1.9 multiples of random distribution (m.r.d.) is seen in the MAF+CR condition (Figure 4a). Texture intensity is increased (~ 3.7 m.r.d.) after annealing at 150°C (Figure 4b), indicating preferred grain orientation formation upon recrystallization. The texture components are still present at 200°C, MAF + CR + PA sample (Figure 4c), but they are less intense, suggesting a partial recovery [27].

The combined copper-type ($\{112\} \langle 111 \rangle$) and brass-type ($\{011\} \langle 211 \rangle$) textures are present in both ST and MAF+CR samples, with increased texture intensities after annealing. Local lattice misorientations are quantified as markers of geometrically required dislocation (GND) densities using Kernel Average Misorientation maps (Figure 5). Low misorientation patches (blue-green), which are associated with high strain localization, predominate in the MAF+CR sample (Figure 5a). Increased proportions of intermediate misorientation values ($1\text{--}3^\circ$) after annealing (Figures 5b, 5c) show partial recovery and recrystallization with persisting plastic strain. In line with changing microstructural recovery, the color-coded KAM data verify lower average strain levels with increasing annealing temperatures [26, 28].

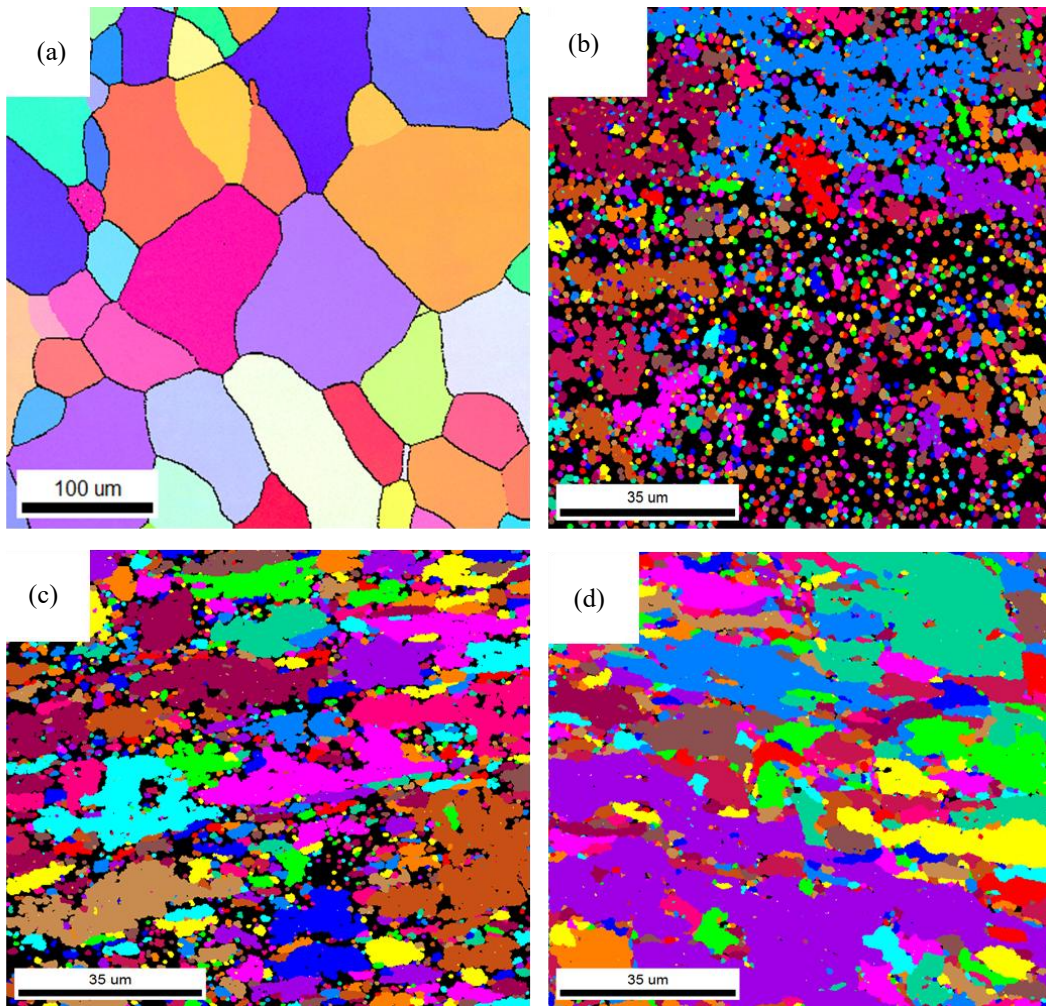


Fig. 2. EBSD orientation maps representing microstructural evolution under four conditions: (a) Solution Treated (ST) condition, (b) MAF + CR processed state, (c) MAF + CR followed by post annealing at 150 °C, and (d) MAF + CR followed by post annealing at 200°C. These maps demonstrate progressive grain refinement and texture modification with additional processing and post-annealing treatments

4.2. Low Cycle Fatigue Response and Fracture Behavior

4.2.1. Cyclic Stress–Strain Behavior

The MAF+CR+150°C sample achieves the highest peak stress (± 270 MPa) and the largest loop area, reflecting optimal cyclic hardening and maximum energy dissipation, owing to dislocation–precipitate interactions and effective precipitation strengthening [30, 31]. By contrast, MAF+CR+200°C exhibits reduced cyclic strength and a narrower hysteresis loop (± 240 MPa), likely due to partial recovery and precipitate coarsening during overaging, which results in a decreased dislocation density and precipitate efficacy [31, 32]. Table 3 summarizes the cyclic response and mechanisms for each processing condition.

Due to dislocation–precipitate interactions and efficient precipitation strengthening, the MAF+CR+150°C sample reaches the highest peak stress (± 270 MPa) and the biggest loop area, indicating excellent cyclic hardness and maximum energy dissipation [30, 31]. In contrast, MAF+CR+200°C shows a tighter hysteresis loop (± 240 MPa) and decreased cyclic strength, probably as a result of partial recovery and precipitate coarsening during overaging, which reduces precipitate efficacy and dislocation density [31, 32]. The cyclic response and processes for each processing situation are compiled in Table 3.

Table 3: Summary of cyclic stress response, loop area, and fatigue mechanisms for Al 2099 alloy under different SPD and annealing treatments.

Condition	Peak Stress (approx.)	Loop Area	Cyclic Hardening	Fatigue Resistance	Mechanism
ST	±200 MPa	Smallest	Low	Poor	Coarse grains, low dislocation density
MAF + CR	±230 MPa	Moderate	Moderate	Improved	Grain refinement, dislocation
MAF + CR +150°C	±270 MPa	Largest	High	Excellent	Precipitation + dislocation
MAF + CR +200°C	±240 MPa	Moderate	Mild	Intermediate	Partial recovery, over aging

The LCF findings indicate a great reliance of fatigue conduct on microstructural development: the ST circumstance indicates little hardening, brief life, hybrid SPD and moderate aging (MAF+CR+150°C) denotes better fatigue protection because of optimal grain framework and precaution impacts. Superior fatigue properties are the properties acquired when the age of the process is 200oC with the onset of processes such as softening taking over [32].

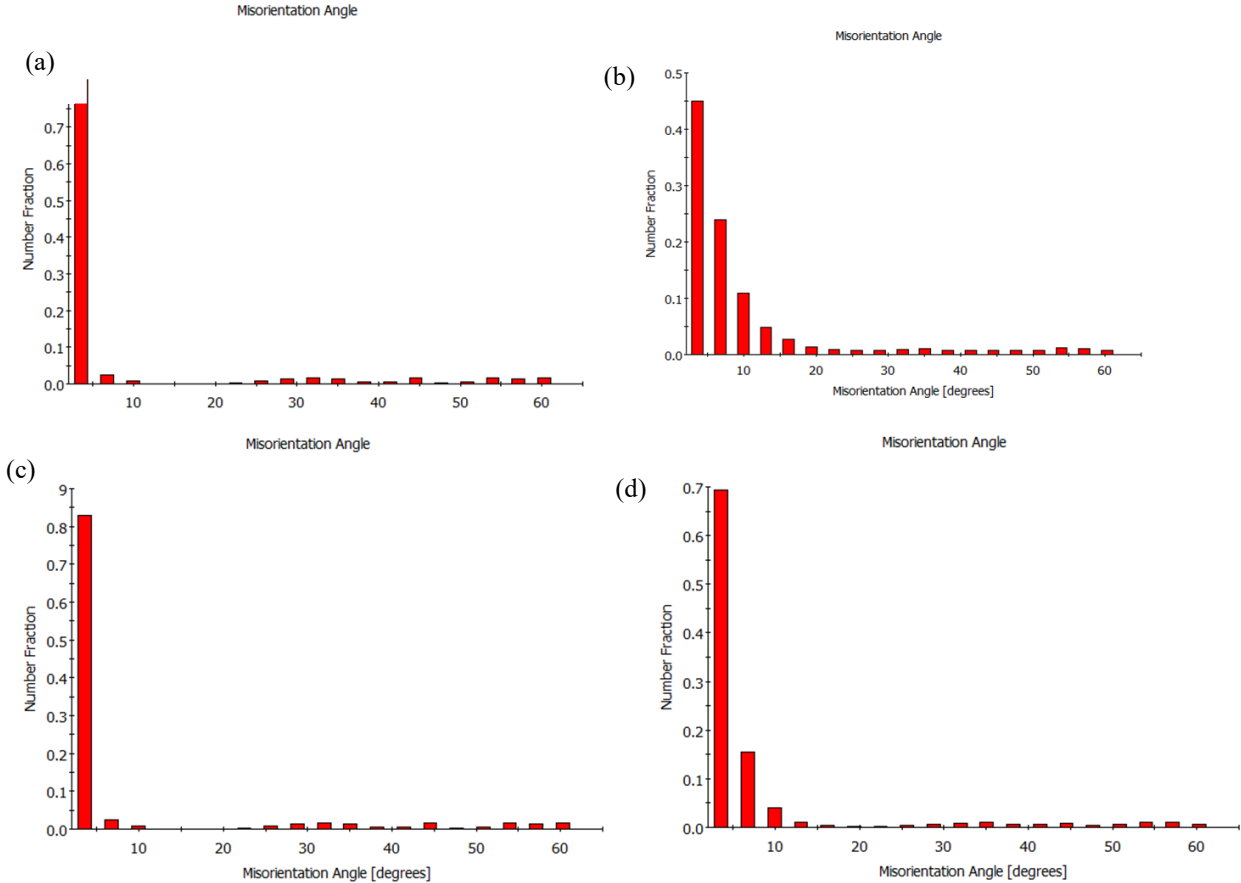


Fig. 3. Distribution of grain boundary misorientation angles for (a) Solution Treated (ST) condition (b) MAF + CR processed state (c) MAF + CR followed by post annealing at 150 °C (d) MAF + CR followed by post annealing at 200°C. The number fraction of boundaries versus misorientation angle illustrates the evolution of low and high angle boundaries after processing and post-annealing treatments

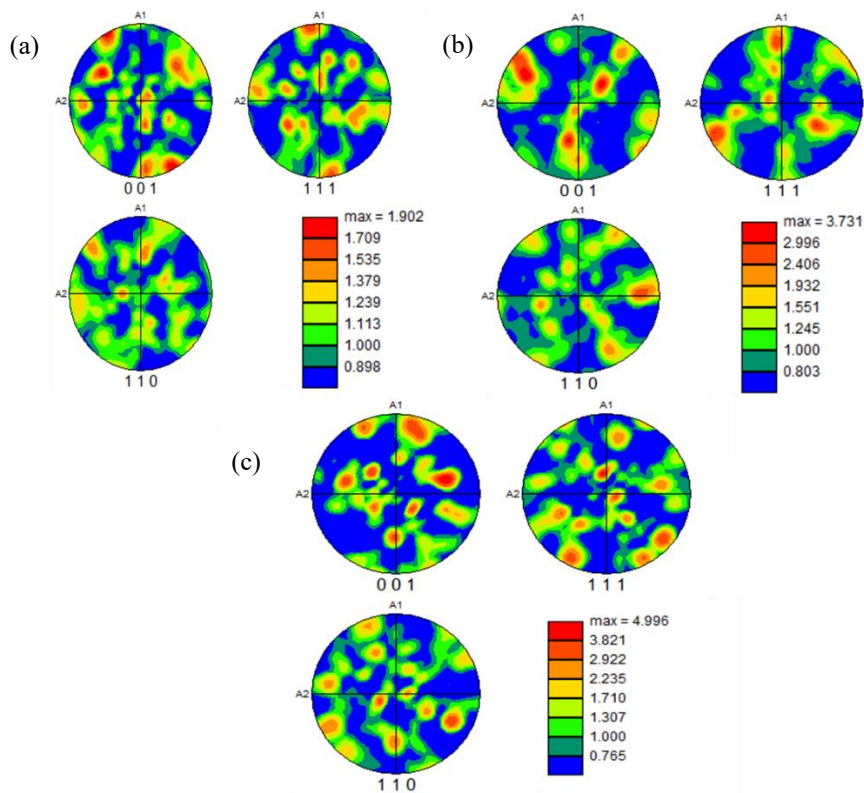


Fig. 4. {001}, {111}, and {110} pole figures for (a) MAF or CR processed state, (b) MAF or CR followed by post annealing at 150 °C (c) MAF or CR followed by post annealing at 200°C conditions. The orientation distribution highlights the evolution of crystallographic texture with deformation and post annealing treatments, as indicated by color intensity in Multiples of Random Distribution (M.R.D.)

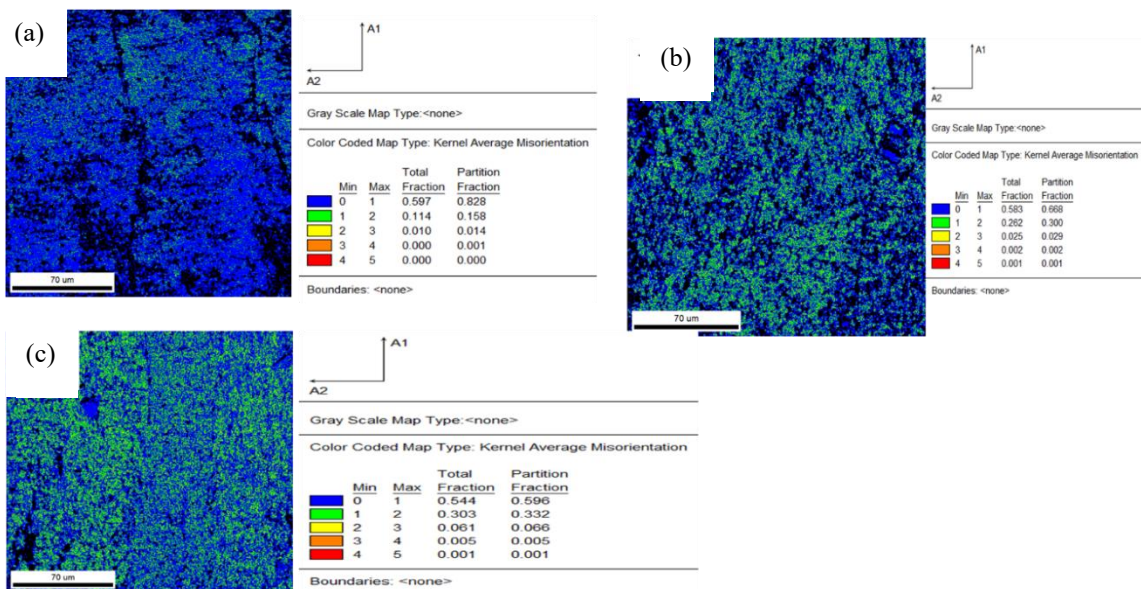


Fig. 5. Kernel Average Misorientation (KAM) maps for (a) MAF or CR processed state (b) MAF or CR followed by post annealing at 150 °C (c) MAF or CR followed by post-annealing at 200°C conditions. The color-coded maps reveal the distribution of local lattice distortions, highlighting increased internal misorientation and strain following multi step deformation and post-annealing thermal treatments.

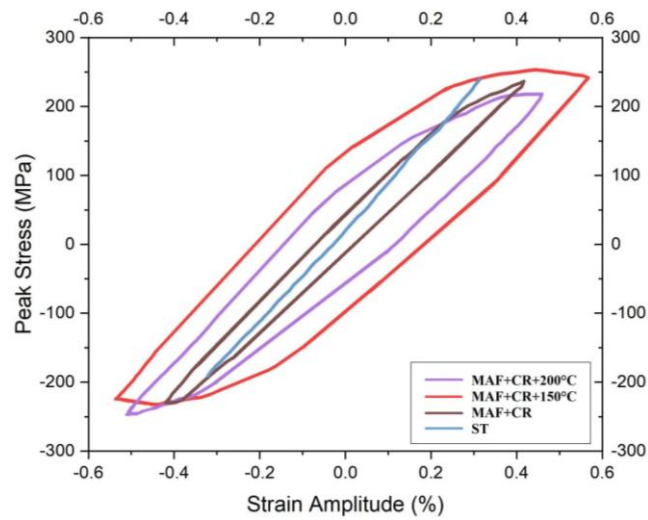


Fig. 6. Stress-strain hysteresis loops comparing solution treated (ST) with multi-axial forged and cold rolled (MAF+CR) under different annealing conditions (room temperature, 150°C, and 200°C).

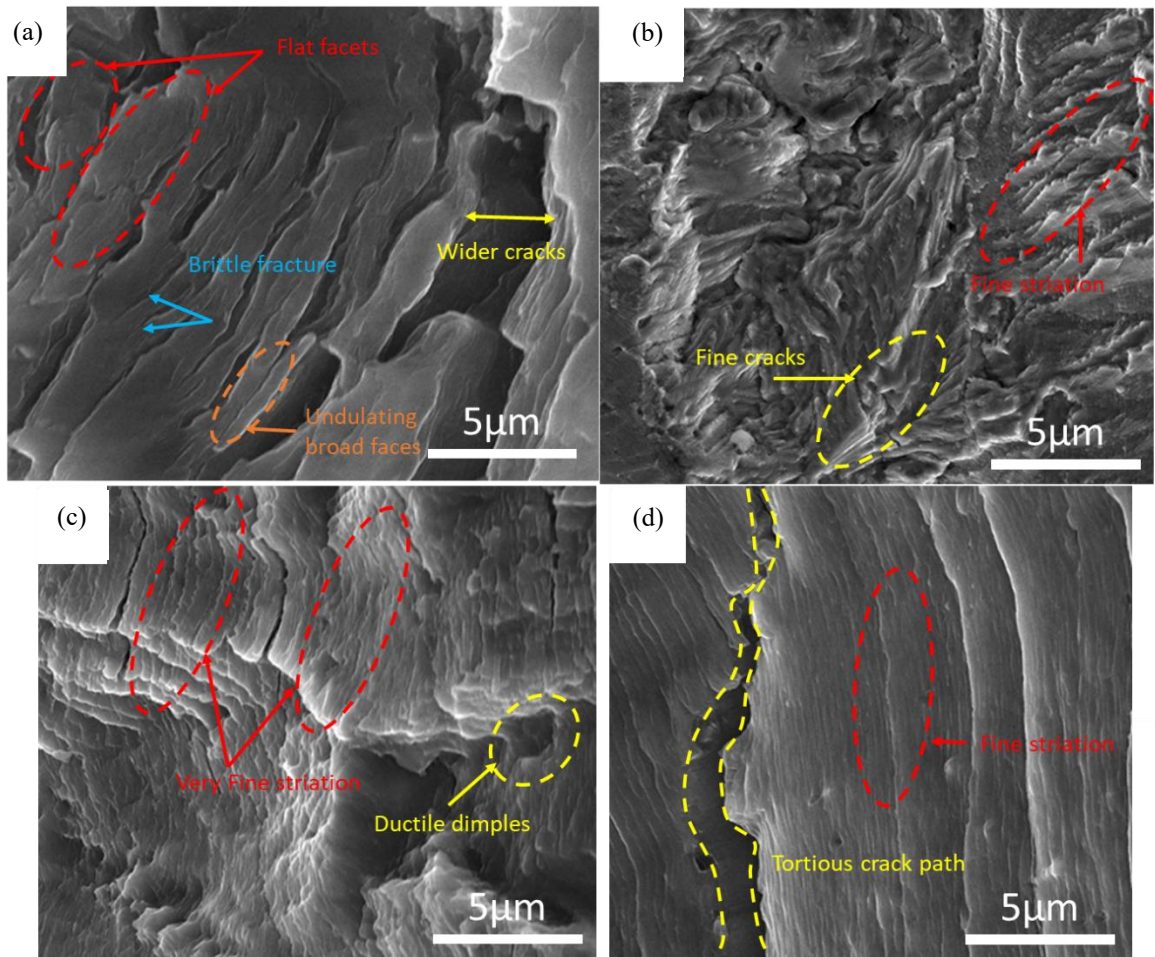


Fig. 7. SEM micrographs showing fracture surface morphologies of (a) ST displaying flat facets, brittle fracture, and wider cracks; (b) MAF+CR showing fine cracks and torn edges; (c) MAF+CR+150°C exhibiting dimorphic features with ductile dimples; and (d) MAF+CR+200°C demonstrating tortuous crack path with fine intergranular cracking.

4.2.2. Fracture Surface Morphology after LCF

The morphology of the fracture surfaces following LCF test is provided in Fig. 7. The ST condition has long planar facets, thin cracks, which suggests that it is cleavage-like and trans granular brittle failure, and has a low cyclic strength as it does [29]. In MAF+CR, the surface is coarser with areas of ductile tearing and shallow dimples which are indicative of a tortuous crack path and increased energy absorption due to refinement of the grain structure and increased density of the dislocations [30].

The most ductile characteristics are observed in the MAF+CR+150 °C sample, which has fine and equiaxed dimples and large microvoid coalescence, which are attributed to the widest hysteresis loop and the largest energy absorption [31]. In the case of the MAF+CR +200 °C, the fracture surface has a more coarse dimples and intergranular, which is the case of hardened boundaries of the grain and lowered strength through overaging [32].

5. Conclusion

This research proves that hybrid MAF-CR processing and annealing after deformation are successful in improving the fatigue property of AA2099 Al-Cu-Li alloy microstructural and low-cycle. The study was systematic to assess the impact of both harsh plastic deformation and associated thermal treatments on the refinement of grains, development of the texture and the performance of the mechanical strength. The major inferences are the following:

- The hybrid MAF+CR process was successful in forming ultrafine-grained structures ($\sim 0.42\mu\text{m}$) with a high density of dislocations and low-angle grain boundaries, whereas post-annealing at 150°C and 200°C led to a controlled growth of grains to $0.84\mu\text{m}$ and $0.75\mu\text{m}$ with a high
- The MAF + CR + 150°C condition had the best low-cycle fatigue resistance and peak cyclic stress of about $\pm 270\text{ MPa}$ and maximum energy dissipation due to efficient precipitation strengthening and dislocation precipitate interactions.
- The fatigue properties ($\pm 240\text{ MPa}$) degraded by annealing at 200°C, indicating the immensely important role of controlled thermal treatment.
- Fracture surface analysis showed that in MAF+CR+150 °C, there were mostly ductile failures with fine equiaxed dimples, whereas the solution-treated samples and overaged samples were brittle and intergranular, respectively.
- It is estimated in the article that the hybrid MAF-CR route with optimized annealing at 150°C offers a feasible strategy when attempting to produce a high-performance ultrafine-grained Al-Cu-Li alloy in aerospace applications.

Nomenclature

ST	Solution treated at 535 °C for 2 h
MAF + CR	Solution treated at 535 °C followed by cryo-rolling to a 90% thickness reduction
MAF or CR or 150 °C	Solution treated at 535 °C, cryo-rolled to a 90% thickness reduction, and subsequently annealed at 150 °C
MAF or CR or 200 °C	Solution treated at 535 °C, cryo-rolled to a 90% thickness reduction, and subsequently annealed at 200 °C

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