Direct hot rolling as a solid-state recycling process for green sheets production

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Abstract. With the increasing demand for sustainable solutions in the recycling of aluminum alloys, solid-state recycling (SSR) offers an energy-efficient alternative by avoiding the melting phase, which typically leads to high energy consumption and material loss. This study presents a novel SSR process utilizing direct hot rolling to recycle aluminum alloy chips (EN AW-5754). The main objective is to evaluate this process's feasibility and assess the recycled sheets' mechanical and microstructural properties. Aluminum alloy chips were produced from the turning process of EN AW-AA5754 bars. The chips were compacted and subsequently heat-treated at 400°C for 2 hours. The compacted samples were then hot rolled in multiple passes, with a final cold rolling step to achieve a final thickness of 0,8 mm. Mechanical properties and microstructure were analyzed using tensile testing machine and SEM-EBSD technique. The recycled samples demonstrated mechanical properties comparable to those of reference material. SEM/EBSD analysis revealed broken oxides and a layered grain structure due to the prior chips' boundaries. Overall, the results confirm that direct hot rolling can be a viable recycling method for aluminum alloys, offering significant energy and material savings compared to conventional processes.

Introduction

The use of aluminum has surged across various industries (including automotive, aerospace, and construction) due to its exceptional strength-to-weight ratio, corrosion resistance, superior plasticity, and excellent joinability and its utilization is expected to grow in the next decades [1]. However, conventional recycling methods, such as melting, are energy-intensive and result in significant material losses due to oxidation. These inefficiencies underscore the urgent need for innovative recycling approaches. Solid-State Recycling (SSR) has emerged as an alternative to traditional recycling methods. By avoiding the melting phase, SSR significantly reduces energy consumption and minimizes material waste [2]. Several SSR processes have been developed, among which the most common, based on severe plastic deformations, are direct Extrusion, Friction Stir Extrusion (FSE), High Pressure Torsion (HPT), Equal Channel Angular Pressing (ECAP) and Direct Hot Rolling (DHR). Common steps to every SSR process are grinding, cleaning, drying, compaction, and hot plastic or cold deformation [3]. The last step depends on the chosen method.

Chips production is required in a lot of industries where machining is a step to obtain the final mechanical part. SSR is an energy-efficient method to recycle aluminum alloys and particularly chips, where traditional recycling can lead to metal losses due to oxidation [4]. An additional advantage of SSR processes is that preventing fusion process avoids mass losses in recycling chips.

Gronostajski et al. [5] demonstrated the feasibility of production extruded products through direct recycling extrusion of chips, as well as Tekkaya et al. [6]. ECAP process as well was demonstrated to be able to consolidate metal chips [7,8]. FSE has been recently applied to recycling chips, with the production of wires [9,10] and tubes [11]. Additionally, several authors have focused on developing FEM models for these processes [12,13]. HPT was demonstrated by Abd El Aal et al. [14] as a possible recycling route for aluminum chips. It is also possible to add reinforcement particles to chips to obtain improved bonding and mechanical characteristics after SSR [15,16].

Several authors studied miscellaneous methods. Suzuki et al. [17] produced rolled product by hot rolling as a second step of the SSR process, after chips extrusion. Chiba et al. [18] explore instead the possibility of recycling chips through cold extrusion and consequently cold rolling.

Allwood et al. [19] studied the possibility of cold bonding through forging, rolling or extrusion processes, performed separately, demonstrating that bonding could occur with sufficient strain, but some voids could remain. El Mehtedi et al. studied DHR followed by Accumulative Roll Bonding process (ARB) [20].

Among SSR techniques, direct hot rolling offers a promising route for recycling aluminum alloy chips into high-quality sheets obtaining a good material’s consolidation and low environmental impact [21,22]. This study explores the feasibility of direct hot rolling as a solid-state recycling method for aluminum alloy EN AW-5754. Building on previous research presented at the 12th ICEB-Aluminium2000 international conference (Bologna, Italy) [23], it further investigates the potential of this recycling method. By assessing the mechanical and microstructural properties of the recycled sheets, this study aims to validate the efficacy of the process in producing high-quality green sheets with properties comparable to bulk material, demonstrating its potential as a sustainable alternative to conventional recycling techniques.

Experimental methods

The aluminum chips, produced from the turning of an EN AW-5754 alloy bar without the use of lubricants, have the nominal chemical composition detailed in Table 1 and an average length of 7 ± 2mm and width of 3±1mm. These chips were cold compacted into circular molds using a force of 150 kN (equivalent to a compacting pressure of 120 MPa), followed by a two-hour heat treatment at 400°C in a muffle furnace (Nabertherm N50). This process resulted in discs measuring 40 mm in diameter and 9 mm in thickness.

Table 1 – Chemical composition of the alloy EN AW-AA5754.

Element	Si	Fe	Cu	Mn	Mg	Zn	Ti	Cr	Al
content (wt.%)	<0,40	<0,40	<0,10	<0,50	2,90	<0,20	<0,15	<0,30	bal.

Subsequently, the compacted chips underwent hot rolling (HR) at 400°C over five passes, again without the use of lubricants. To maintain consistency with industrial rolling processes, the rolling schedule was carefully designed to ensure that the samples were reheated in the furnace for 10 minutes after each pass, restoring the initial temperature of 400°C. The rolls were kept at room temperature throughout.

A final cold rolling (CR) pass at room temperature was performed to achieve the desired final thickness of 0,8 mm. Table 2 details the nominal thickness progression after each pass, while Fig. 2a-c visually illustrates the samples at various stages: after compaction, following the rolling process, and after jagged edge removal, respectively.

Table 2 – Rolling Schedule.

Process	**Hot Rolling**						***Cold Rolling**
Pass number		1	2	3	4	5	6*
Thickness (mm)	9	5	4	2,5	1,75	1,25	0,8

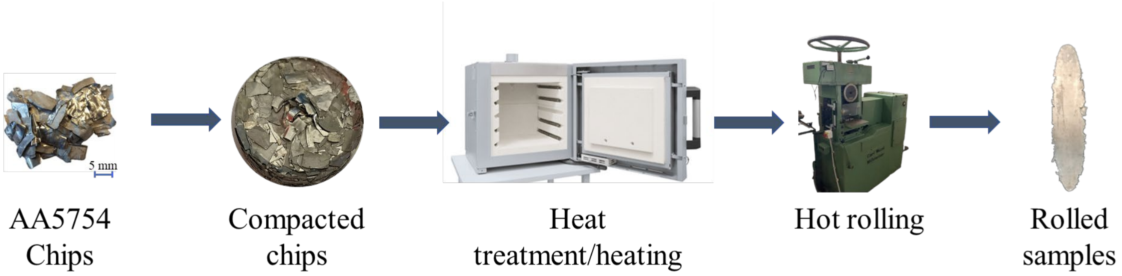
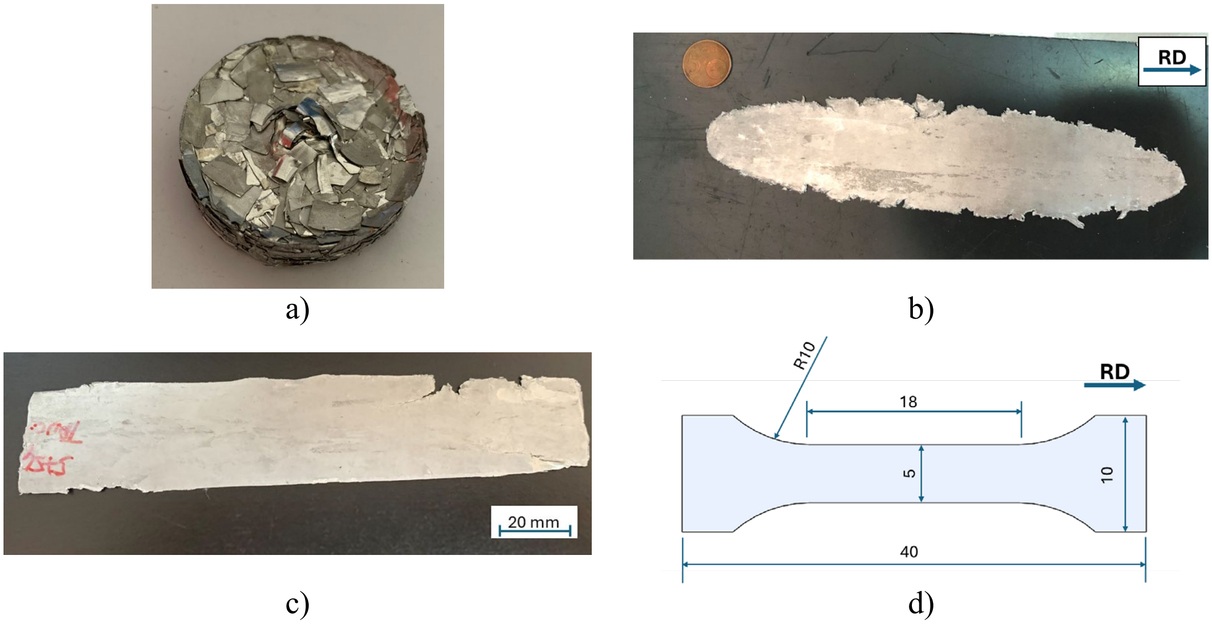


Figure 1 – Representation of process steps [23].



a) b) c) d)

Figure 2 – a) EN AW-5754 chips compacted prior to recycling, b) a sample after the direct hot rolling process, c) a sample post cold rolling with jagged edges removed (RD aligned with scale bar) [23], d) tensile specimen geometry (all measures in mm).

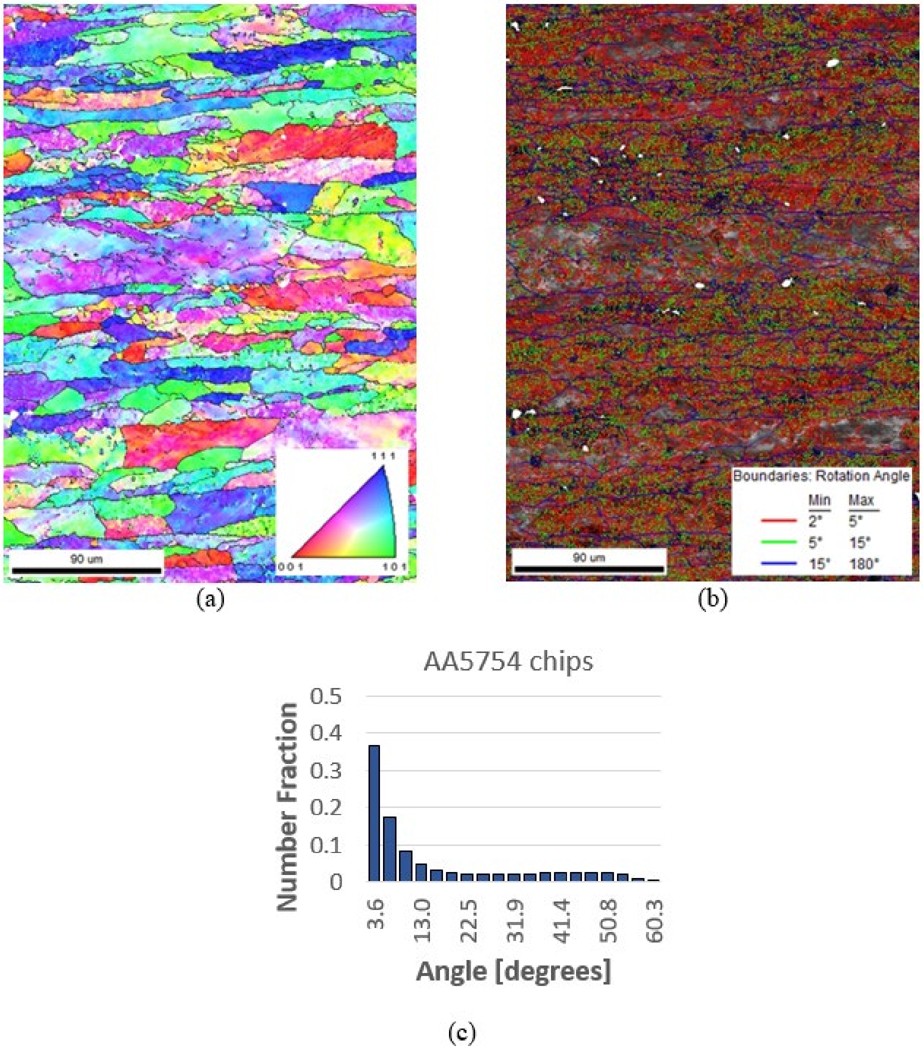
The recycling efficiency of the direct rolling process was evaluated by analyzing each sample's mechanical properties (tensile tests) and microstructure (SEM and EBSD).
Mechanical properties were investigated on every sample produced by hot rolling: at least 3 tensile samples from recycled chips and samples from the bulk material were tested in rolling direction (RD). The specimens were extracted from the middle of the rolled sheet and oriented along the RD (Fig. 2d). Tensile tests were carried out according with ASTM E8/E8M and BS EN 895, with the usage of a material testing machine a Zwick Z050.

SEM samples were prepared by mechanically polishing the surface. For EBSD measurements, the samples were further electropolished to remove any scratches and the deformation layer. The electropolishing process was conducted using a Struers Lectropol-5 and Struers AC2 electrolyte, applying 20V for 20 seconds. The SEM/EBSD evaluation was carried out on a FEG-SEM Zeiss Ultra 55 Gemini.

Results and discussion

The microstructures of the recycled sheet samples and the hardness was analyzed in details in the previous work [23]. The samples exhibit a highly layered microstructure due to the characteristics of the original material. It is characterized by higher elongated grains in the RD with respect to the cross-section.

EBSD Analysis. The images in Fig. 3 depict Electron Backscatter Diffraction (EBSD) measurements of EN AW-AA5754 recycled sample.



(a)

(b)

(c)

Figure 3 – EBSD measurement of EN AW-AA5754 chips. (a) IPF map (RD aligned with the scale bar), (b) low boundaries map and (c) histogram of angle distribution.

Fig. 3.a shows an EBSD map with diverse color patterns, which represents the varied crystallographic orientations of the individual grains within the alloy. Each color corresponds to a different crystallographic orientation, as illustrated by the inverse pole figure map located in the lower-left corner of the composite image. The black lines correspond to a misorientation angle higher than 15° and show the boundary of grains. This image shows a significant number of grains with different orientations, indicating a microstructure that it is strongly affected by the base material (chips) and by the presence of Mg atoms in the matrix that promotes the grain refinement [24,25].

Fig. 3.b illustrates the orientation of grains (IPF map) in the recycled EN AW-AA5754 alloy chips with different color patterns showing a high number of low-angle boundaries and medium-angle boundaries. The presence of Magnesium atoms dissolved in the aluminum matrix creates a

solid solution that can strengthen the alloy. The Mg atoms, which are different in size compared to the aluminum atoms, create lattice distortions. The histogram (Fig. 3.c) indicates the distribution of misorientation angles for grain boundaries within the EN AW-AA5754 chips and confirms what is already said about the boundary map. Most boundaries seem to have low misorientation angles, as indicated by the taller bars on the left side of the histogram.

SEM and EDS analysis. In Fig. 4, an EDS map of EN AW-AA5754 recycled chips at 3000 magnification is represented, revealing the spatial distribution and concentration of different elements within the sample. This comprehensive EDS mapping, in conjunction with the SEM image, provides critical insights into the distribution of alloying elements, the formation of specific intermetallic phases, and the presence of defects and inclusions in the recycled chips.

The Fe map reveals localized iron-rich regions, which are co-locate with Manganese and silicon rich regions, supporting the presence of α-Al(FeMn)Si and β-AlFeSi intermetallic [26]. In the SEM image in gray scale this particle appears to be light grey/white. No voids or inter-chips defects are visible in the analyzed samples. Yellow regions in the Si map indicate the presence of silicon-rich areas, which are not only in conjunction with the Fe map, but also with Mg rich zone, potentially indicating Mg_2Si precipitates formation. The red regions on the O map denote areas with oxide particles. Given the absence of the other elements in those regions on the EDS maps except for Mg, these are likely aluminum oxides and magnesium oxides. In the SEM image in gray scale these oxide particles appear dark gray. The presence of dark spots within this map may indicate areas with other intermetallic compounds and oxide inclusions that correspond with the previously mentioned particles.

In recycled chips, it's common to have more inclusions and higher oxygen content due to oxidation during prior processing. Whenever Magnesium (Mg) is present as an alloying element, Aluminum oxide (Al_2O_3) consistently coexists with Magnesium oxide (MgO). This association has been reported by several studies in the literature [27,28]. The MgO thickness is influenced by the annealing temperature and the oxidation that occurs at each step of the process conducted at high temperatures. In particular, from the color map of Oxygen and Magnesium it is clear that MgO and Al_2O_3 are organized along parallel line (aligned along the RD) and appear to be broken into numerous parts. Chino et al. [29], achieved a uniform dispersion of oxide layers around aluminum chips by employing an extrusion ratio of 1600:1, corresponding to an ideal real strain of 7,4. In contrast, the rolling process described in this study involves significantly lower deformations. The maximum deformation observed was 0,6 during the first rolling pass, with a cumulative real strain of 2,4 from the initial thickness to the final pass. Despite the lower deformation levels, the oxide layer around the chips was observed to be cracked in multiple points, allowing for continuity within the aluminum alloy matrix.

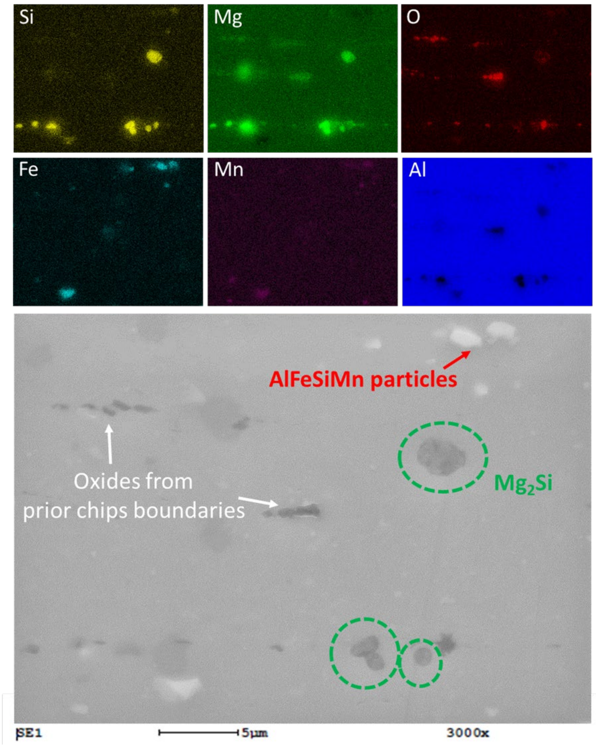


Figure 4 – EDS map of EN AW-AA5754 recycled chips, rolling direction at 3000x. RD aligned with the scale bar.

Mechanical Properties. In Fig. 5, a stress-strain curve is depicted, which provides a comprehensive portrayal of the mechanical properties of recycled-chips EN AW-AA5754 in the rolling direction (RD). This average curve is representative of the material's response under tensile loading.

The curve demonstrates a rapid initial increase in stress with strain in the elastic deformation region, followed by plastic deformation, showing a typical trend for ductile material. The peak of this curve, the ultimate tensile strength (UTS) of the material, registered a mean value of 316MPa. The oscillatory trend in the plastic region of the tensile curves visible in Fig. 5 is very common in the 5xxx alloys family and is due to the dynamic interaction between dislocations and solute atoms of Mg during plastic deformation, known as the Portevin-Le Chatelier effect (PLC) [30].

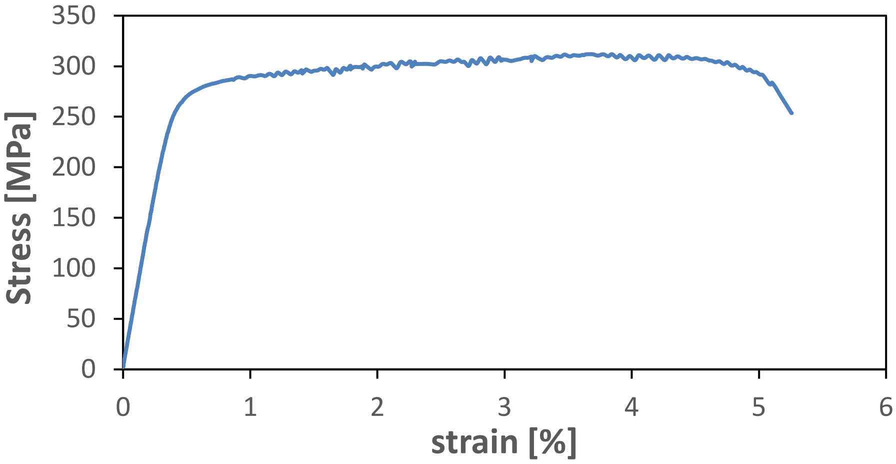


Figure 5 – Representative average tensile curve of recycled-chips EN AW-AA5754 sheet.

Table 3 shows the other mechanical parameters for the recycled-chips sheet: Yield Strength ($\sigma_{0.2}$) of 279 MPa and Elongation at Break (A%) of 5,7 %.

Furthermore, a comparative assessment is offered against standard datasheets and prior literature. The Aalco UK [31] datasheet for AA5754 H26 reports a minimum yield strength of 190 MPa and a UTS range of 265 - 305 MPa, with a minimum elongation at break of 4%. The study by Yakut et al. [32] on AA5754 H26 suggests a yield strength of 170 MPa, an elastic modulus of 68 MPa, a UTS of 290 MPa, and an elongation at break of 10%. In comparison, the recycled-chips EN AW-5754 exhibits higher yield strength and ultimate tensile strength than both the Aalco UK datasheet values and the findings of Yakut et al. The elastic modulus is consistent with the Yakut et al. study, and the elongation at break for the recycled-chips falls between the two referenced benchmarks. This result demonstrates that the recycling process was successful.

Table 3 – Tensile test results for EN AW-AA5754 recycled-chips.

Source	**$\sigma_{0.2}$ [MPa]**	**E [GPa]**	**UTS [MPa]**	**A [%]**
Present study	279 ± 2,8	69 ± 2,6	316,1 ± 5,9	5,7 ± 0,7
Aalco UK [31] AA5754 H26	190 min.	-	265 - 305	4 min.
Yakut et al. [32] AA5754 H26	170	68	290	10

Fracture Surface Analysis. Fig. 6 shows SEM pictures of fracture surface of recycled chips tensile sample. The fracture surface exhibits a porous and uneven morphology. These voids or pores could be a result of defects inherent with bonding between chips.

The structure seems to have an interconnected network of dimples and ridges. The presence of such dimples indicates a ductile mode of fracture. Dimples form because of microvoids coalescence, where small voids or defects in the material come together to form a larger crack, leading to failure. The images also indicate material inhomogeneity, potentially arising from variations in local composition, such as precipitates within the alloy. This is supported by the EDS analysis, which reveals potential sites for crack initiation due to the presence of intermetallic compounds and precipitates.

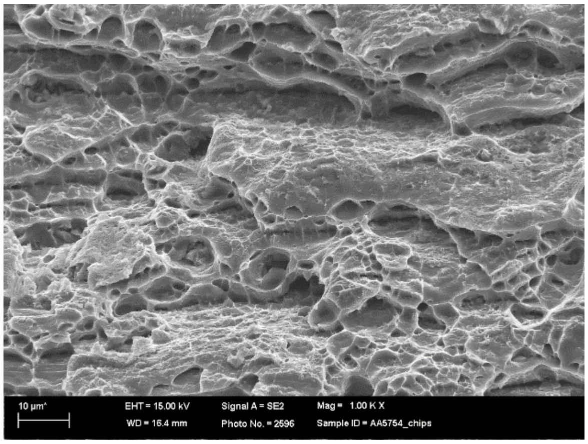


Figure 6 – SEM image of the fracture surface of recycled EN AW-AA5754 chips, at 1000x.

The fracture surface exhibits also a highly porous morphology with interconnected voids and cavities, likely resulting from imperfections in bonding during the recycling process. It maintains ductility, evident from the dimples, but shows the presence of ruggedness and larger voids. Variations in surface texture and composition is present, indicating possible inhomogeneities or inclusions within the recycled material. The microstructure appears layered and uneven, with some evidence of the original chip boundaries potentially influencing the fracture characteristics.

Conclusions

This study demonstrates the feasibility and effectiveness of Direct Hot Rolling (DHR) as a solid-state recycling method for producing high-quality aluminum sheets from EN AW-5754 chips. The results can be summarized as follows:

- The recycled aluminum sheets exhibited mechanical properties comparable to those of reference material from literature, including a tensile strength exceeding standard benchmarks for EN AW-AA5754 alloys. This result highlights the potential of the DHR process to meet industrial performance requirements.
- The recycled samples revealed a layered microstructure resulting from the consolidation of the chips. EBSD/SEM analysis showed a significant presence of grain refinement, influenced by the magnesium content in the matrix and that of the oxide layers from prior chips boundaries were effectively broken at multiple points, allowing strong bonding within the matrix and contributing to the mechanical integrity of the recycled sheets.
- The fracture surface analysis indicated a ductile mode of failure, characterized by dimples and interconnected voids, consistent with effective chip bonding. Minor inclusions and crack traces were observed, but they did not significantly compromise the mechanical performance.

Overall, the Direct Hot Rolling process has proven to be a promising method for recycling aluminum alloy chips into high-quality sheets. Future research should focus on optimizing process parameters, such as chip cleaning and compaction, to further enhance efficiency and reduce the environmental impact of the process. This work underlines the potential of DHR to contribute to a more sustainable circular economy for aluminum recycling as the process achieves significant energy savings compared to traditional recycling methods, making it a more environmentally friendly alternative for aluminum recycling.

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