A Novel Approach to Enhance Mechanical Properties during Recycling of Aluminum Alloy Scrap through Friction Stir Consolidation

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Abstract
Solid state recycling (SSR) is a new approach for making metals recycling more efficient with respect to remelting based approaches. Friction stir consolidation (FSC) is a new solid-state process that is employed to recycle metallic scraps. Until now, a single-step FSC process was applied to recycled metal chips. During the single-step approach, critical processes parameters, especially processing time and rotational speed, are considered vital to control the quality and mechanical properties of the billet. However, the effectiveness of process parameters is highly restricted by challenging masses of recycling chips and machine competency. The present study first highlights the issues of the single-step FSC process, such as unconsolidated billet and poor mechanical properties at the bottom of the part, i.e. far away from the stirring action. Then for the first time, three different two-steps FSC methods were introduced as new approaches to overcome the existing challenges of the single-step method. The effectiveness of these methods was evaluated through the Vickers hardness measurements, and microstructure analysis. The results showed that two-steps FSC methods successfully led to a fully consolidated billet and considerably improved mechanical properties.

Keywords Friction stir consolidation .Solid state recycling .Recycling aluminum chips .Multi-step approaches

1 Introduction

Global demand for materials has grown considerably over the past decades. According to International Energy Agency, global demand for steel has increased by three times, cement by nearly seven times, primary aluminum by nearly six times and plastics by over ten times since 1971 [1]. However, alongside the benefits of the materials, they are also responsible for the emission of anthropogenic gases that have adverse environmental impacts. Besides, a huge amount of energy is consumed to transform raw material into the final product. In 2017, the industrial sector was reported to be responsible for nearly 40% of total final energy consumption and nearly one-quarter of direct CO_2 emissions in 2017.

Steel and aluminum are the primary metals that dominate in the industrial sectors and account for 24% and 3% of worldwide industrial emissions, respectively [2]. Although at present, only the percent share of steel is larger than aluminum among the metals in global materials consumption [3]. The demands for aluminum are rapidly increasing. It has significantly replaced steel and copper in automotive, aerospace, electronics, and structural application industries.

The growing demand for aluminum is putting immense pressure on industries to increase the production rate [4]. According to Hydro Annual Report in 2019, the global aluminum consumption was 89.9 million tons [5]. In which the share for transport was 26%, 24% for construction, 11% for each of the categories of electrical goods and machinery, 8% for each of the packaging and foil stock applications, and 6% for consumer durables.

Virgin aluminum is produced from bauxite ore. But the process is very complicated, costly, energy-intensive, and responsible for approximately up to 20% loss of pure aluminum. Besides, large amounts of waste slag are generated, which takes up considerable land resources and pollutes the environment [6]. Environmental policy efforts greatly encourage the processes that reduce primary resource use, pollution prevention, waste management, and sustainable products.

Therefore, new strategies are to be explored to minimize mining for new ore and its negative environmental impact. The potential alternatives are to meltdown, reform, and reuse metals without losing their beneficial qualities. These practices can meet much of the demand for new metals by simply recycling metals that are already in circulation. According to the European Aluminum Association (EAA, 2020), currently about 36% of European aluminum demand is met by recycled material, it is expected that recycling rate will reach to 50 percent of EU demand for aluminum by 2050 [7].

Usually, aluminum is recycled through the conventional remelting route. But processes for conventional methods of recycling consist of multiple steps of refining, remelting, and casting, which are complex and require considerable labor and power [8]. These methods have further severe implications especially during recycling aluminum chips. This kind of scraps, due to their high surface-to-volume ratio are prone to oxidation causing permanent material loss during the melting process. They cause adverse environmental impact, high cost, and significant material loss. Therefore, the researchers attempted solid-state recycling (SSR) techniques [9, 10].

SSR methods involve fewer recycling steps [11, 12]. The scrap metals are first cleaned and then directly converted into bulk products and semi-products by mechanical means or plastic deformation without undergoing remelting phase. Therefore, bypassing melting phase significantly eliminates the losses due to oxidation and leads to a recycled product with superior mechanical properties [13]. These new methods can save up to 95 % of energy, 59 % of the cost, and 46 % of material compared to the conventional melting process [14].

The idea of SSR routes for aluminum alloy scrap by direct scrap extrusion was patented in the USA in 1945 by Stern [15]. Few years later, he extended this approach by presenting an idea of continuous extrusion of aluminum scrap with the use of a rotary furnace where aluminum scrap is heated up to the extrusion temperature [16]. From that time onwards, various SSR techniques have been proposed for scrap consolidation by imposing significant plastic and shear strain.

Copper and Allwood [17] and Lela et al. [18] investigated through mathematical modelling the influence of deformation conditions in solid-state welding of aluminum. Duflou et al. [19] performed an environmental assessment of three different solid-state recycling methods comparing with the melting process as a reference. They concluded that solid-state recycling methods are more effective for machining scraps recycling.

Extrusion-based various SSR processes have been investigated for the recycling of aluminum scrap. Fogagnolo et al. [20] and Güley et al. [21] successfully recycled the scraps of aluminum alloy AA6061 and AA1050 respectively by direct hot extrusion. Chiba and Yoshimura [22] highlighted that the high extrusion ratio results in an increased level of introduced plastic strain, achieving bulk scrap-based profiles with comparable mechanical properties as the cast-based profiles. Then modified form of extrusion process were applied by integrating extrusion with Equal Channel Angular Pressing (ECAP) [23, 24] to achieve superior mechanical properties. Further, Haase and Tekkaya [25] reported that recycling aluminum chips through integrated ECAP die has superior mechanical properties with respect to those extruded through the flat-face die. To enhance the consolidation process and omit the pre-compaction and the scrap preheating step, Widerøe et al. [26] applied screw extrusion where the applied rotational movement generates extrusion pressure and introduces large shear strains.

Tang and Reynolds [27] used friction stir extrusion (FSE) for wire production. A rotating die was plunged into a cylindrical chamber containing the metal scraps. The stirring action of the tool plastically deformed the materials and forced them into the extrusion chamber. Finally, after densifying and heating, fully dense rods were extruded [28]. Kolpak et al. [29] studied the direct conversion of AA 6060 machining chips into finished parts by hot extrusion with subsequent cold extrusion. They developed a procedure to predict the minimum required weld quality value necessary to provide a process success.

Some researchers worked on the environmental assessment and finding the critical process parameters for FSE. Baffari et al. [30] reported that FSE allows a reduction in energy demand up to 74% and 63% compared to the conventional and the equal channel angular process methods, respectively. Further, they also found [31] that growing extrusion rate or decreasing processing time led to reducing significant power demand. Similarly, Tahmasbi and Mahmoodi [32] analyzed the influence of rotational speed during friction stir extrusion of 7022 aluminum alloy. The study suggested that a rise in rotational speed decreased hardness and dislocation density and increased average grain size. Besides aluminum, researchers have also successfully recycled other metals scraps through solid state methods. Lui et al. [33] recycled Ti-6Al-4V machining chips through ECAP and Topolski et al. [34] recycled titanium chips by using KOBO extrusion process.

Similar to FSE, friction stir consolidation (FSC) is a novel solid-state process. The only exception is that FSC lacks an extrusion channel within the tool. Therefore, the scrap metals are consolidated into a billet or disc shape rather than extrusion in the wire shape. FSC process has two main steps: compaction and consolidation [35]. Compaction refers to the initial pressing of aluminum chips or powder in a hollow die chamber by applying a specific load through a cylindrical tool. Then materials are further pressed and stirred through the tool's downward force and rotational speed during the consolidation phase. Baffari et al. [35] numerically modelled the welding of materials chips during FSC to predict bonding parameters.

The non-homogeneous mechanical properties of consolidated billet during single-step FSC of aluminum alloy AA6061 were first reported by Li et al. [36]. They found that the main reason for inhomogeneity was different plastic deformation and the temperature of the material in the radial direction and the advancing direction of the die. They also reported that increasing processing time, rotational speed, or consolidation force led to improved mechanical and metallurgical properties of the consolidated disc.

Buffa et al. [37] examined the influence of processing time, rotational speed, and quantity of chips on the AA 2024 consolidated disc. The report suggested that increasing processing time and speed or lowering mass resulted in a better-quality consolidated disc.

Although researchers [36, 37] successfully identified the critical process parameters for the single-step FSC process. These process parameters standalone are still insufficient to develop a fully consolidated billet and eliminate inhomogeneous mechanical properties. In addition to already reported reasons, the other possible causes for such issues could be challenging recycling masses or limited machine capacity. In the current study, first, this was proved experimentally that regulating mechanical property based solely on critical parameters is not a perfect solution. Therefore, these problems need to be addressed by other strategies. The present research tackles them in a completely different way. One potential solution is based on the finding of Buffa et al. [37] that lowering billet height led to improved mechanical properties. Therefore, half of the mass of chips is consolidated in the first step, and then the remaining half is consolidated in the second step by adding more mass over the top of the billet developed in the previous step. This results in two billets bonded together as a single product, and a new two-steps FSC process called the mass addition approach is developed. The other way is to consolidate billet both on top and bottom surface by allocating half of the processing time for each surface. This method is called the double-sided approach. The third approach was the hybrid that involves mass addition and flipping side steps of both two-steps approaches. The purpose of this hybrid approach is to combine the beneficial properties of mass addition and double-sided approaches. The performance of these methods is evaluated by comparing them to the single-step process. Hardness measurements and grain size distribution are considered the main output criteria. Besides, the broader objective of the study is to improve FSC process productivity and environmental sustainability.

2 Experimental Procedure

2.1 Materials

The experimental setup of FSC consisted of ESAB LEGIO (a dedicated friction welding machine), an H13 cylindrical steel tool with 25 mm diameter, and a die also made of H13 steel with a nominal diameter of 25 mm (tolerance 0.2-3 mm), as shown in Fig.1. A round bar of aluminum alloy AA 2024-O was reduced to chips through turning operation (parameters listed in table 1). The diameter of the bar was 30 mm and chemical composition by mass percentage was 94.1Al-0.003Cr-4.26Cu-0.57Fe-0.004Mg-0.01Mn-0.129Si-0.003Ti-0.008Zn-0.5Pb-0.12Sn. Acetone, a washing solvent commonly used for cleaning metallic chips, was utilized to clean the chips. Due to its eco-friendly approach, of course, it can also be implemented on the industrial level with some other processes such as stirring and then drying in the oven to further improve process effectiveness and reduce oxidation [38]. Then the chips were charged in the die, finally compacted, and consolidated at 5KN and 20KN force, respectively.

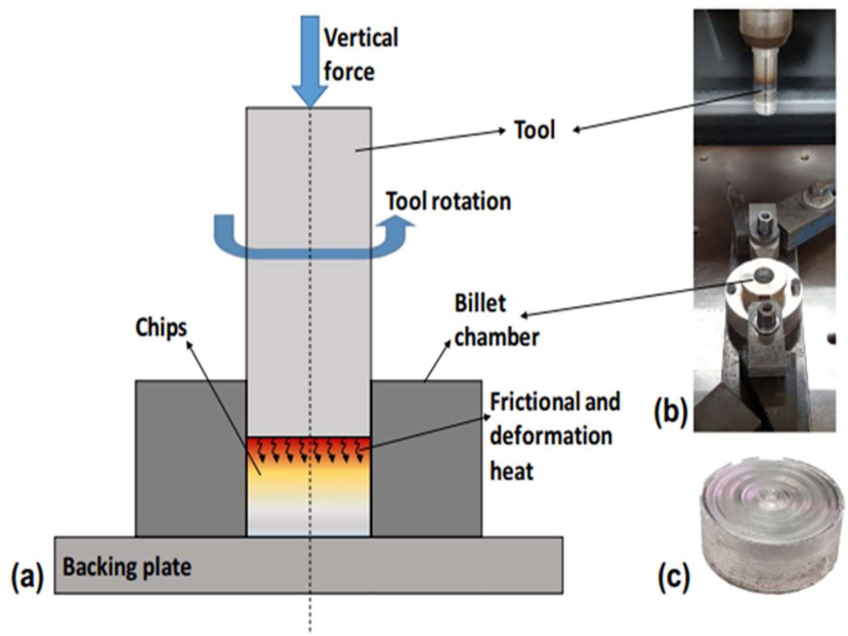


Fig. 1 a Sketch of the process, **b** utilized fixture, and **c** consolidated billet [38]

Table 1 Detailed information of chips preparation

Machine	Process	Rotational speed (rev/min)	Depth of cut (mm)	Feed (mm/rev)	Cutting angle (degree)
COMEV 180	Finishing	460	1.5	2	15°

2.2 Process

Previous studies on AA2024-O aluminum alloys have highlighted that recycling a 20 g mass was an upper bound [37]. It is worth mentioning that such an upper bound is due to the used experimental setup. In fact, other setups would very likely allow recycling more (or less) aluminum mass. Therefore, in the current research, a fixed mass of 20 g chips was recycled during a single-step FSC process. The experimental setup of previous studies [37] was employed with a constant tool force of 20KN, variable tool rotation, i.e., 1000 rpm and 2000 rpm, and variable process time, i.e., 40 and 60 s. Many studies [36, 37] reported that the higher consolidated force enhances the billet quality. Consequently, in this research study, the maximum applicable load considering the machine's safety was applied to get a quality billet. Initial attempts of the current research were based on achieving a fully consolidated billet by increasing the processing time and rotational speed. However, this approach could not lead to successful outcomes as explained in the next sections. Hence, to get a fully consolidated billet with homogenous mechanical properties, three new strategies in terms of two-steps FSC methods were applied. In this way, the experimental plan of 16 different conditions: two levels of processing time, two levels of rotational speed, and four methods (one single-step, three two-steps) were completed.

Concerning the procedure of the single-step FSC process, first, the total mass (20 g) was charged into the die and then consolidated in a single run under the experimental conditions listed in table 2.

While in two-steps methods: mass addition, double-sided, and hybrid, the same experimental conditions were considered as listed in table 2. The only exception was the number of steps (two). Besides, a pause of 30 minutes was kept between step 1 and step 2. That was a reasonable time to prepare the machine for step 2 and bring the die, tool, and machine to room temperature. The reason was to provide the same initial experimental condition (ambient temperature) for developing billet from the chips. Indeed, keeping a shorter pause can minimize the risk of oxidation. However, in this study, the effect of pause duration was not studied on billet quality in terms of oxidation. Thus, the total processing time was divided into two equal intervals (40:20+20 and 60: 30+30 seconds).

In mass addition approach, the total mass was divided into two equal parts (10 g - 10 g) to decrease the height and achieve a billet with superior mechanical properties. According to the finding of Buffa et al. [37] that lowering billet height led to improved mechanical properties. Therefore, half chips mass was consolidated in the first step, and then the remaining half was consolidated in the second step by adding more mass over the top of the billet developed in the previous step. Hence, two billets bonded together as a single product, and a new two-steps FSC process called the mass addition approach was developed as shown in Fig 2.

In the next approach, the whole mass of billet (20g) was consolidated for half of the processing time on one side, and then its sides were flipped and subjected to consolidation for the remaining half of the time as shown in Fig 2. This method was called the double-sided approach that successfully overcame unconsolidated billet at the bottom and non-homogeneous mechanical properties, especially in the advancing side of the die.

The third approach was the hybrid method. It is an intermediate method between mass addition and double-sided. The hybrid method was developed with goal to combine the beneficial properties of mass addition and double-sided approaches. The first step is the same as in mass addition. A 10 g charge was consolidated for half of the processing time. Then billet was removed from the die. After 30 minutes pause, the billet was flipped and inserted into the die like step 2 of the double-sided approach. Then 10 g more chip was added, and the total charge was consolidated for the remaining half of processing time. The schematic presentation has been given in Fig. 2.

Table 2 Experimental plan

Experiment ID	Rotational speed (rpm)- time (sec)-mass (g)
Exp 1	1000-40-20
Exp 2	1000-60-20
Exp 3	2000-40-20
Exp 4	2000-60-20

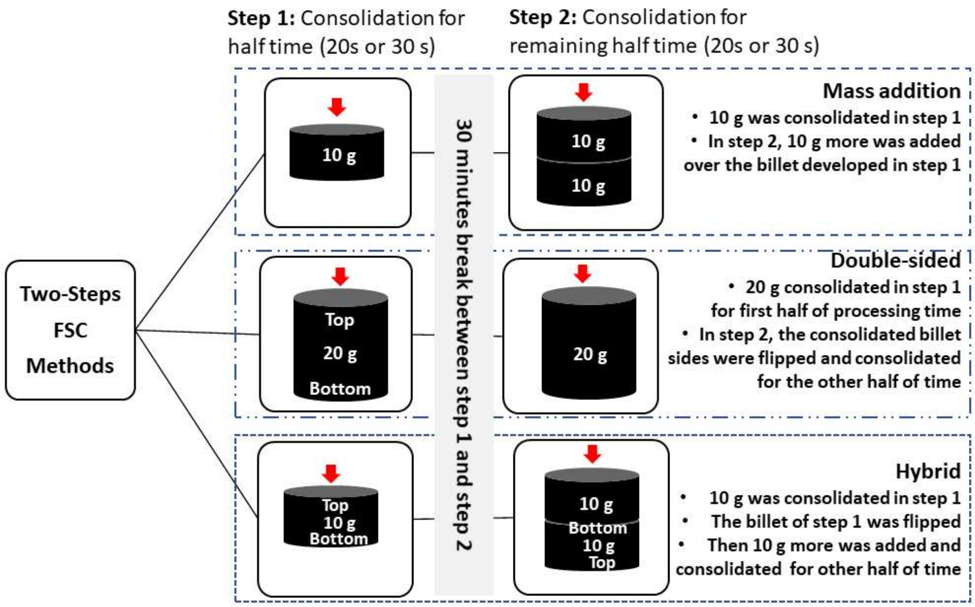


Fig. 2 Schematic of two-steps FSC methods

2.3 Analyzed Output

The consolidated disc was cut into two halves along the longitudinal axis. The disc section was polished with 0.05 μm alumina by applying a series of abrasive papers. The hardness was measured by the Vickers hardness test. A 49 N (5 kg) load was applied for 15 seconds over the four lines along the longitudinal axis. The lines were at radius, r=0 (central line), 6.50, 9.00, and 12.25 mm (near the external surface). On each line, the pitch of the load points was 0.50 mm, as shown in Fig. 3a.

Then microstructure was revealed through Keller reagent and then examined under the OLYMPUS GX51F microscope. On each line, grains were analyzed on the top, middle, and bottom, as shown in Fig. 3b.

Average hardness and average grain size were measured as representative hardness and grain size values for the entire billet section. The standard deviation was also determined to predict the possible variation and uncertainty in hardness or grain size from its corresponding average value. Therefore, a lower standard deviation value is highly desirable because it indicates a billet with more homogenous mechanical properties.

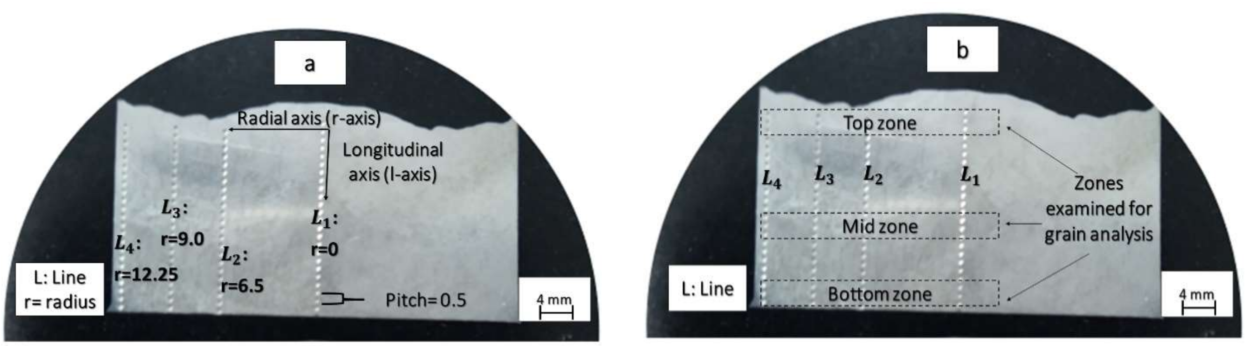


Fig. 3 Schematic diagram for **a** Vickers hardness test, and **b** microstructure analysis
All dimensions in Fig 3a. without units are in mm

3 Analysis of results

In this section, hardness and grain size distribution of single-step and two-steps methods FSC: mass addition and double-sided are compared. First, Exp 1 was considered a reference, and a comprehensive analysis was made for the hardness and grain size profile of both single-step and two-steps approaches. Then, the trend was examined in both longitudinal and radial directions. Finally, the influence of processing time and rotational speed on hardness and grain size distribution was analyzed.
A sub-section has been separately dedicated to the hybrid method. The reason is that hybrid was designed after mass addition and double-sided approach. Besides, the two-steps hybrid approach could not lead to successful outcomes like the other two-steps FSC methods

3.1 Hardness and microstructure in longitudinal and radial direction

Hardness of single step method: In single step FSC method, a billet was developed at a rotational speed of 1000 revolutions per minute (rpm) and a processing time of 40 seconds during the Exp 1 process parameters configuration.

The hardness value continuously decreased from top to bottom of the disc during Exp 1 (Fig. 4a) along the longitudinal axis. The maximum Vickers hardness value (HV) was observed at the top of the billet section around HV 70. The minimum was at the bottom almost HV 50. The hardness at the midsection was near the average value (HV 58). Besides, a high hardness value was noticed along the central line, r=0 mm, and low hardness along the line near the external surface (r=12.25 mm), respectively.

The hardness also follows a decreasing trend in the radial direction. Its value dropped from the central line to the line near the external surface. Even though a uniform hardness (HV 50) was found at the bottom of the billet, this value is remarkably lower than the average hardness value (HV 58).

The rotating tool is considered the hub of the heat. Heat flows from the top surface of the tool to the bottom, as reported by Li et al. [36]. Maximum heat is gained by the top surface, and sound bonding is developed between the layers of the chips, and therefore, the top surface has high hardness. The intensity of the heat reaching the bottom surface is quite low and is not sufficient to cause a full solid bonding of the layers of the chip at the bottom, and thereby, an unconsolidated zone was observed at the bottom of the billet with a relatively low hardness value.

Microstructure of single step method: The billet section was assessed at the top, mid, and bottom zones to examine grain size in the longitudinal direction. Based on the variation in grain size distribution, four different zones were found, as shown in Fig. 5a. First, coarse grains were found at the top of the central line (Fig. 5b). Then, 1.5-2 mm below this zone, a grain growth occurred (Fig. 5c) until the midsection of the billet, where the grain size started to reduce (Fig. 5d), and defects emerged. Finally, grains disappeared, and the number of defects increased at the bottom zone (Fig. 5e).

The grain analysis was also extended to the other three lines (Fig. 6a) at r=6.50, 9.00, and 12.25 mm to investigate grain size tends in the radial direction. The zone near the top surface was characterized by coarse grains of uniform size regardless of their distance from the central line. Larger size grains were found on the central line at the middle section, but their size diminished continuously in the radial direction. At the bottom, grains disappeared. Besides, the region with no visible grains expanded, and defects increased from the central line towards the external surface.

Hardness of two-steps FSC methods

Initially two-step FSC methods: mass addition and double-sided were employed. The aim was to get a uniform hardness value across the billet section and improve the hardness value, especially at the bottom. The experimental strategy of each method was slightly different from the other. Nevertheless, alike hardness profile was noticed for all.

Mass addition: For the Exp 1 of the mass addition approach, the hardness was more uniform both in the longitudinal and radial directions.

Hardness value slightly dropped from the top to midsection then rose again at the bottom. Similarly, a uniform hardness profile was also observed in the radial direction. However, at the bottom of the at r=12.25 mm, the hardness value significantly deviated from other lines (Fig. 4b). Li et al [36] called this "dead zone" comparable to the zone found in extrusion.

The mass addition led approach to a fully consolidated billet and a more homogenous hardness profile. Its working principle is based on a key finding of Buffa et al. [37] studies that a lower billet height owned superior mechanical properties. Therefore, the mass addition approach recycles a smaller mass (lower height) in a short period of processing time. First, a billet of a smaller height is developed, then on its top, a new billet is created, and finally, a product of desired height is achieved by the end of the process. Hence, the mass addition approach provides an alternate solution to a fully consolidated billet with improved mechanical properties compared to the single-step FSC approach.

Double sided: During Exp 1 of the double-sided approach, the hardness value dropped from the top to midsection then grew again at the bottom. The lowest hard-ness value occurred near the midsection of the billet, but still, it was almost equal to the average value (HV 58). While in the radial direction, the hardness was uniform (Fig. 4c) except at the line r=12.25 mm, where almost all hardness values significantly deviated from other lines .

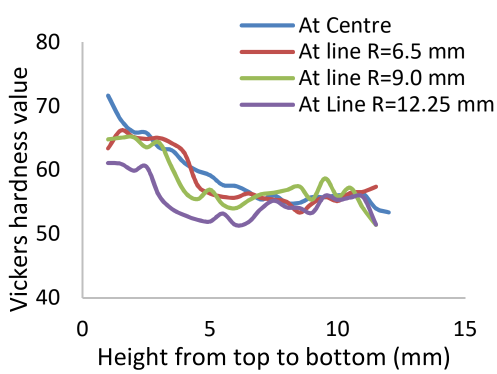
During the double-sided approach, both sides of the billet come into direct contact with the rotating tool. Therefore, a fully consolidated billet was achieved with a uniform hardness at the top surface as well as at the bottom surface.

Microstructure of two-steps methods: The two-step methods were applied to enhance the grain size distribution and eliminate the defects of the bottom section. Overall, these methods led to a similar grain size distribution profile.

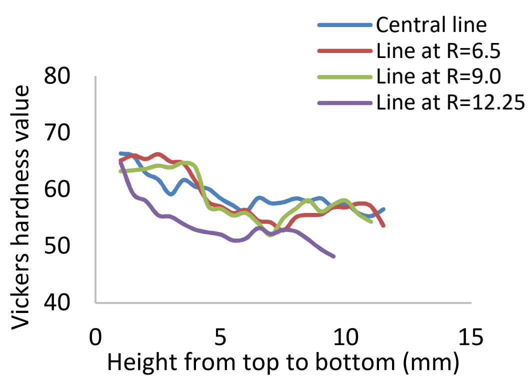
Mass Addition: For Exp 1 of the mass addition approach, grain size decreased from the top to midsection as shown in Fig. 5f, Fig. 5g, and Fig. 5h. Then grain size slightly increased again at the bottom (Fig. 5i). However, grain size continuously decreased along the line at r=12.25 mm from top to bottom (Fig. 6b). On the other hand, a fluctuating trend was observed in the radial direction.

Double-sided: For the Exp 1 double-sided approach, grain size decreased steadily from the top along the longitudinal direction (Fig. 6c). The drop in grain size became more evident for the other lines and especially at line r=12.25 mm.

Similarly, in the radial direction, grain size also decreases. However, grains were observed throughout the section of the billet.



(a)



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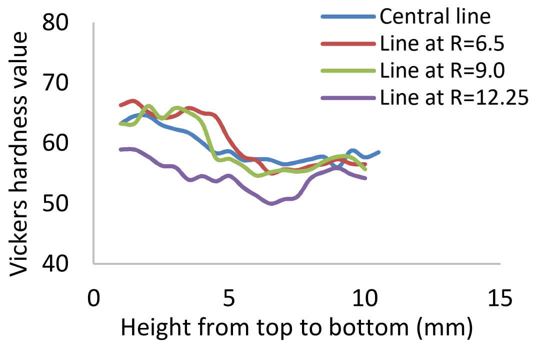
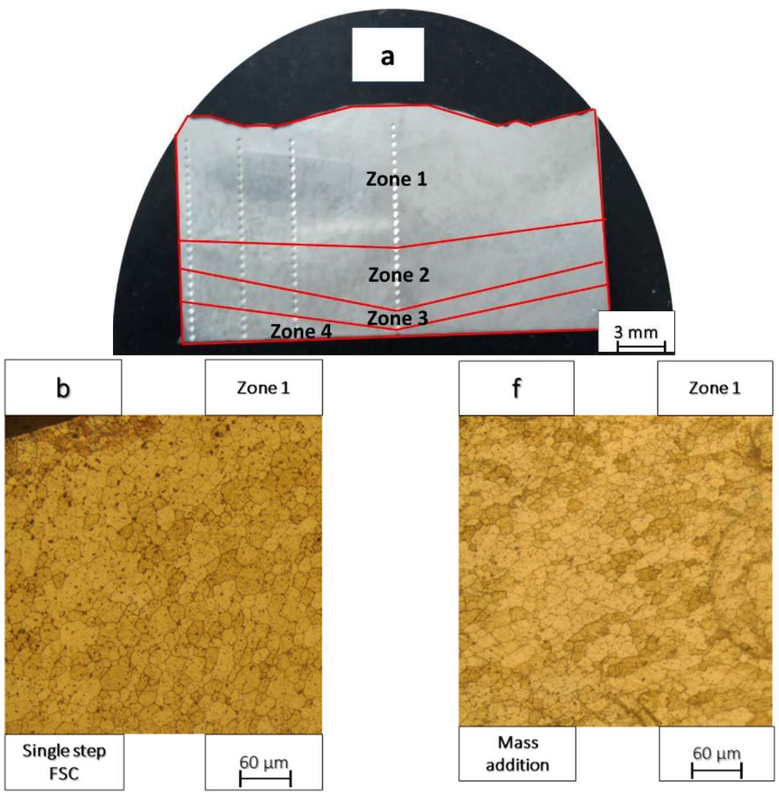


(c)

Fig. 4 Hardness of Exp 1 porcess parameters configutarion for **a** single-step, **b** mass addition, and **c** double-sided



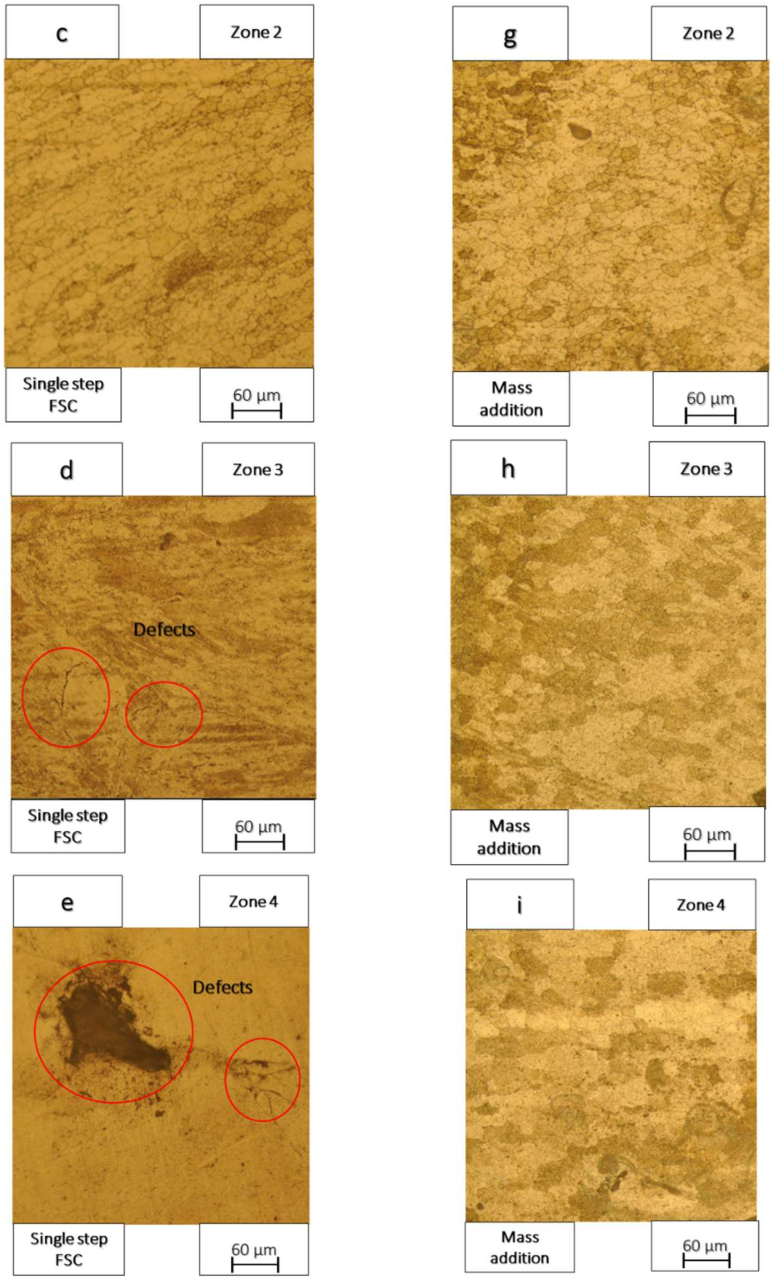


Fig 5. a Four zones with different grain size distribution along the billet section. Grain size distribution of Exp 1 (**b** to **e**) for single-step on left side and (**f** to **i**) for mass addition on right side on 20X

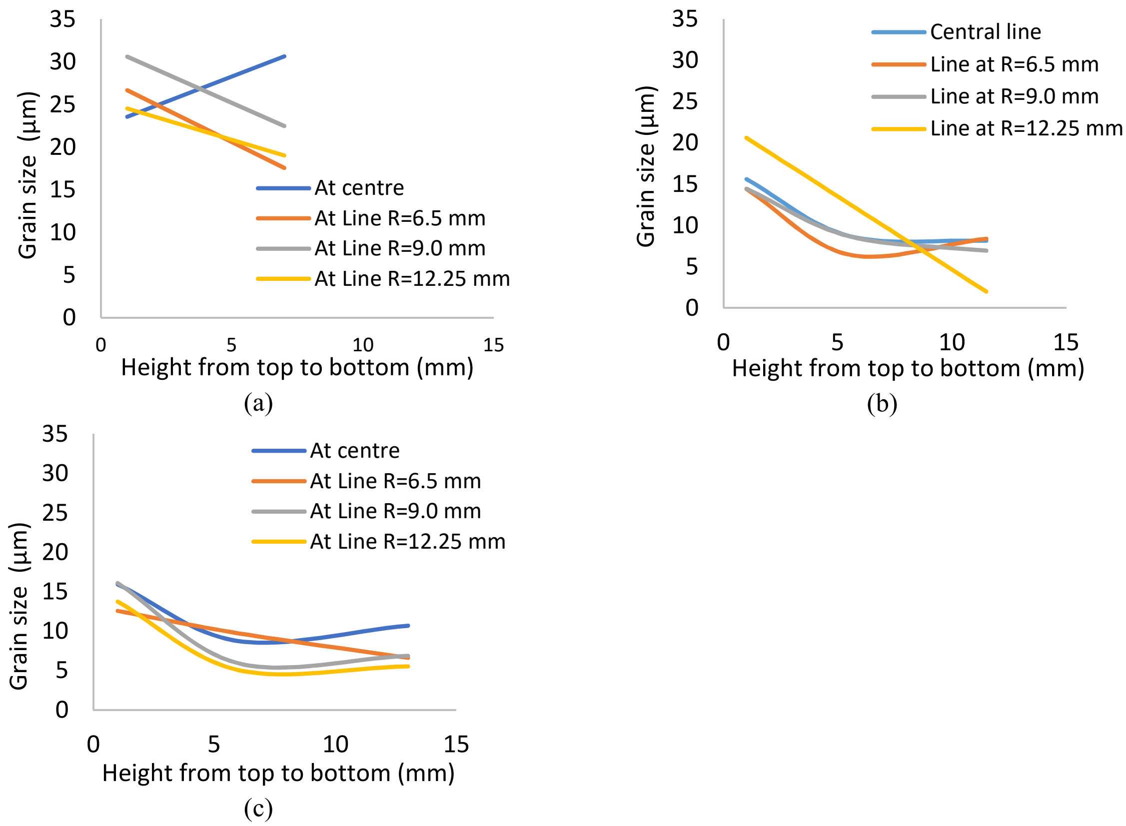


Fig. 6 Grain size distribution of Exp 1 for **a** single-step, **b** mass addition, and **c** double-sided

3.2 Influence of processing time and rotational speed on hardness and microstructure

Hardness of single step method: For simplicity, the hardness of the central line was considered to study the influence of processing time and rotational speed (Fig. 7). The trend of hardness for all four experiments was found to decrease from the top to the bottom of the billet (Fig. 7a).

With rising processing time and rotational speed, the average hardness value showed no notable change and still remained constant around HV 58 (Fig. 8a). In comparison, the standard deviation of hardness values rose (Fig. 8b). This fact depicts that growing rotational speed and processing time caused non-uniform hardness across the billet section.

Microstructure of single step: During Exp 2, the consolidation time was increased from 40 to 60 sec. It caused further grain growth near the top surface. Nevertheless, the trend of the grain size distribution for Exp 2 was found similar to Exp 1.

Then in Exp 3, the rotational speed was doubled (from 1000 to 2000 rev/min), and processing time was also increased to 60 sec during Exp 4. However, such attempts could not lead to a fully consolidated billet. Actually, at the bottom of the billet unconsolidated chips are still visible (Fig. 9). The growing processing time and rotational speed caused grains growth near the top surface and increased the average grain size from 15 to around 30 µm (Fig. 10a). However, the grain size remained unaffected in other parts of the billet section, such as mid and bottom zones, and the standard deviation doubled from 4.5 to 9 (Fig. 10b). Hence, grain distribution further agitated across the billet section.

Hardness of two-steps methods: The effect of processing time and rotational speed was studied by comparing the hardness of the central line (Fig. 7b, and Fig. 7c). Overall, the average hardness remained constant around

HV 58. Its value seemed less sensitive to the kind of approach, and level of rotational speed, and processing time used during FSC.

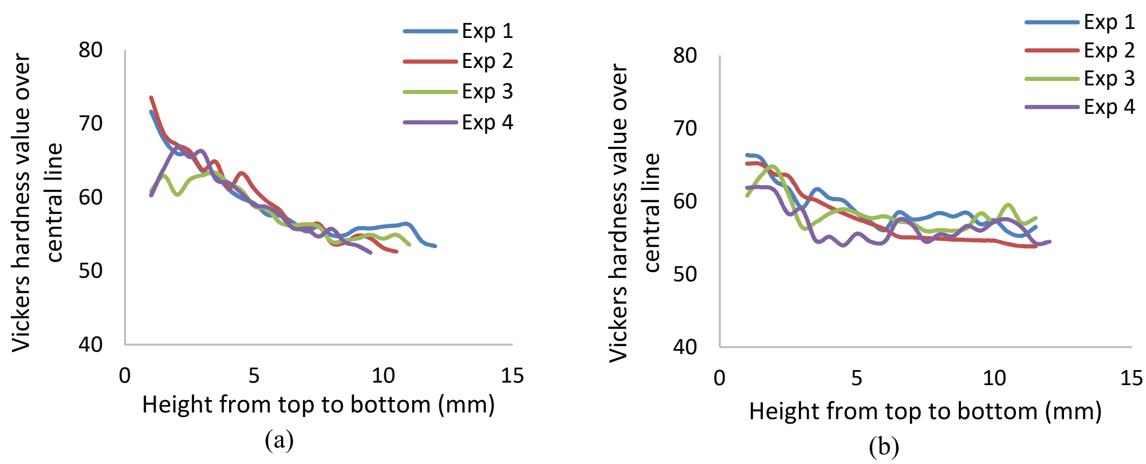
Mass addition: During the mass addition approach, the average hardness fluctuated between HV 57-58. On the other hand, the standard deviation decreased from 4.5 (Exp 1) to 2.9 for Exp 4 (Fig. 8a, and Fig. 8b). This means that rising processing time and rotational speed boost to develop a billet with more consistent and uniform hardness. The reason is that in each step of the mass addition approach, only 10 g of chips (a lower mass) are consolidated, and therefore, the standard deviation in hardness decreased, and hardness became more consistent with rising rotational speed and time. These results agreed to the investigation as reported by Buffa et al. [37]. In comparison, a higher mass (20 g) is consolidated in the case of the single-step process. Therefore, rising rotational speed and time have limited effectiveness on the hardness value (up to the middle section of the billet) and leave the remaining section with high inconsistency in hardness value.

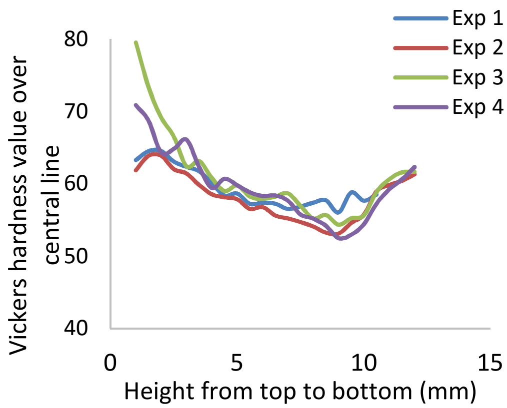
Double-sided: While for the double-sided approach, average hardness although was above HV 58. However, the standard deviation grew up from 4.2 to 4.5, and it stood near to 5 for the rest of the experiments (Fig. 8a, and Fig. 8b). Thus, this approach improved hardness at the bottom section but led to the least consistent and uniform hardness profile.

Fig. 11 provides a schematic presentation of the hardness profile across the section of the billet during Exp 1. The color codes show different levels of hardness. In specific, Fig. 11a represents the hardness of single-step, and Fig. 11b represents hardness for mass addition. It is cleared from the diagram that hardness at the most sectional area of the mass addition is closed to average hardness and thus leading to a more uniform hardness profile.

Microstructure of two-steps methods: With the rise in processing time and rotational speed during the mass addition approach, the average grain size slightly increased from around 11 to 13 µm (Fig. 10a). In contrast, the standard deviation sharply rose from almost 3 to 7.5 (Fig. 10b) leading to less uniform grain size across the billet section. However, grains were still visible throughout the billet section, and defects were minimized at the bottom.

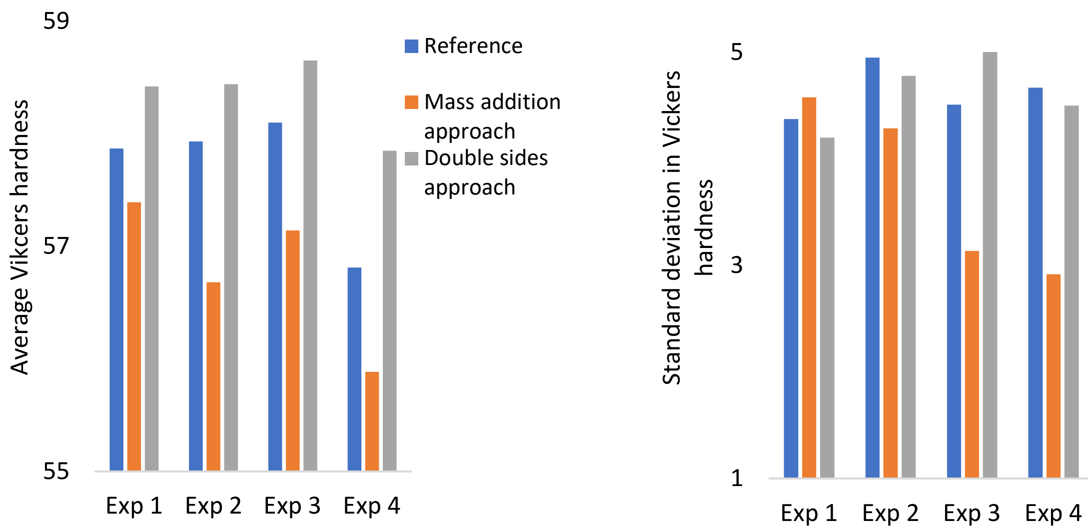
In the double-sided approach, both the average grain size and standard remained less sensitive to process parameters. Their values stabled around 11 µm and 4.5, respectively. Besides, the best uniform grain size profile was noticed in the case of the double-sided approach.





(c)

Fig. 7 Hardness at central line of all experiments **a** single-step, **b** mass addition, and **c** double-sided



(a) (b)

Fig. 8 a Average Vickers hardness, and **b** standard deviation of hardness

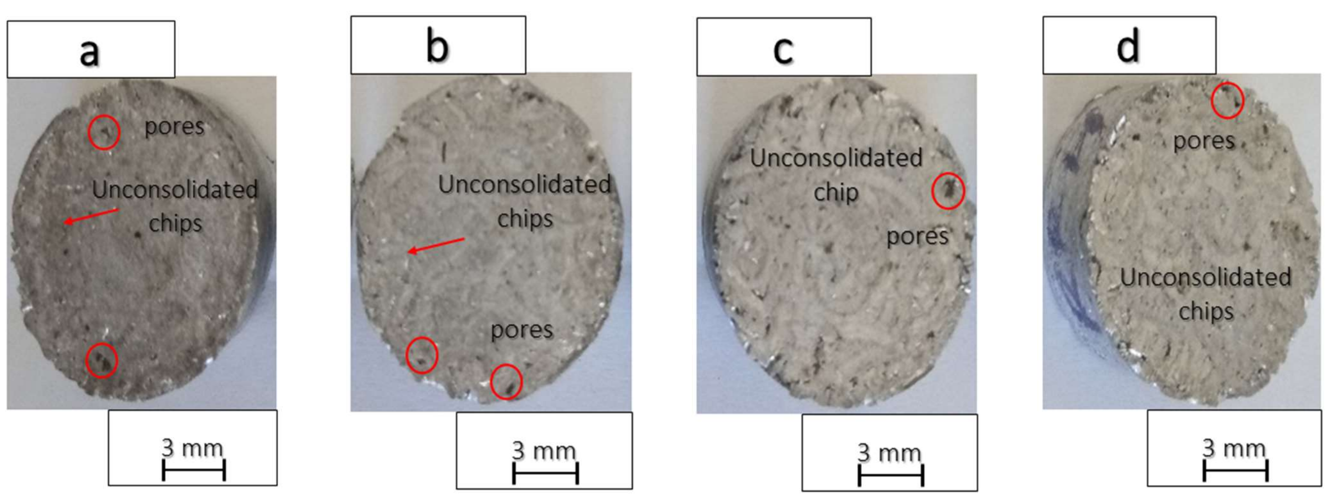
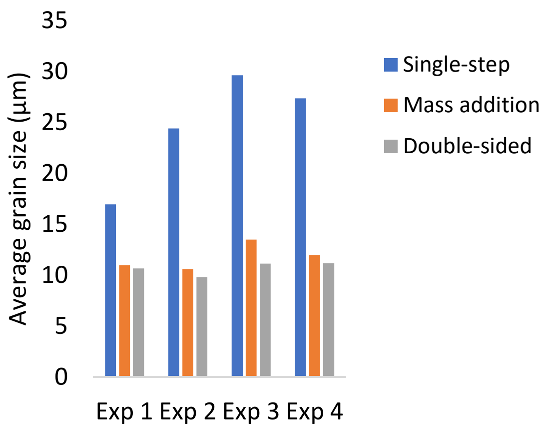
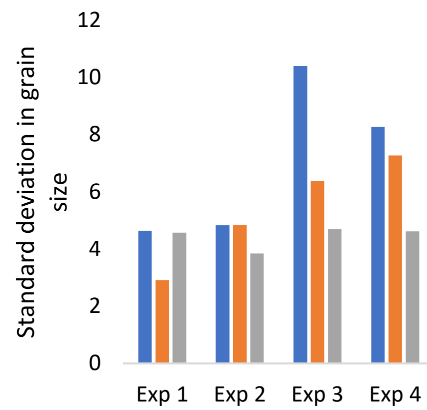


Fig. 9 For single-step FSC, unconsolidated chips at the bottom of the billet **a** Exp 1, **b** Exp 2, **c** Exp 3, and **d** Exp 4



(a)



(b)

Fig. 10 **a** Average grain size (µm), and **b** standard deviation of hardness

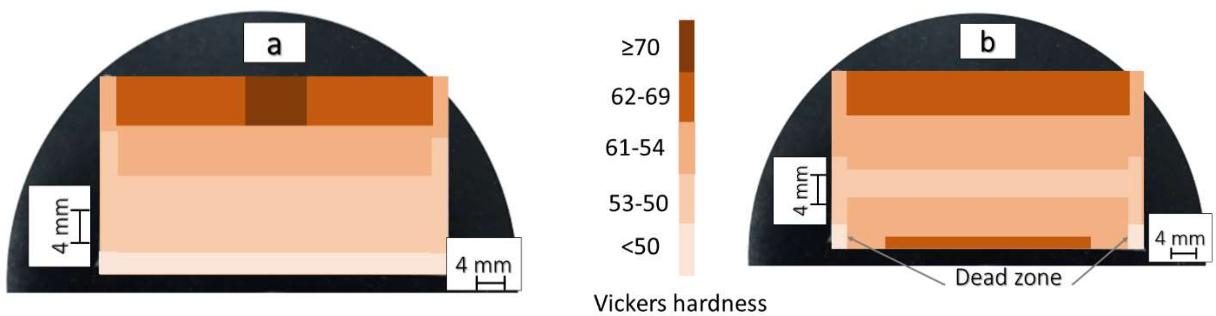


Fig. 11 Schematic diagram for hardness profile **a** single-step, and **b** two-steps methods

3.3 Hybrid Approach

In terms of hardness profile, the mass addition showed excellent results (Fig. 8) . On the other hand, relatively a uniform grain size distribution was observed for the double-sided approach (Fig. 10). Therefore, both these approaches were merged in a single method called the "hybrid approach." The aim of the hybrid method was to achieve both a homogenous hardness as well as a uniform grain size profile. The results showed that hybrid is the potential method with superior mechanical properties compared to the other two-step approaches.
For simplicity, the hardness profile for Exp 2 at the line at r=9.0 mm and grain size distribution for Exp 1 at central were considered where it is more evident that mechanical properties of the hybrid method (Fig. 12 a and b) are in between both two-steps approaches.

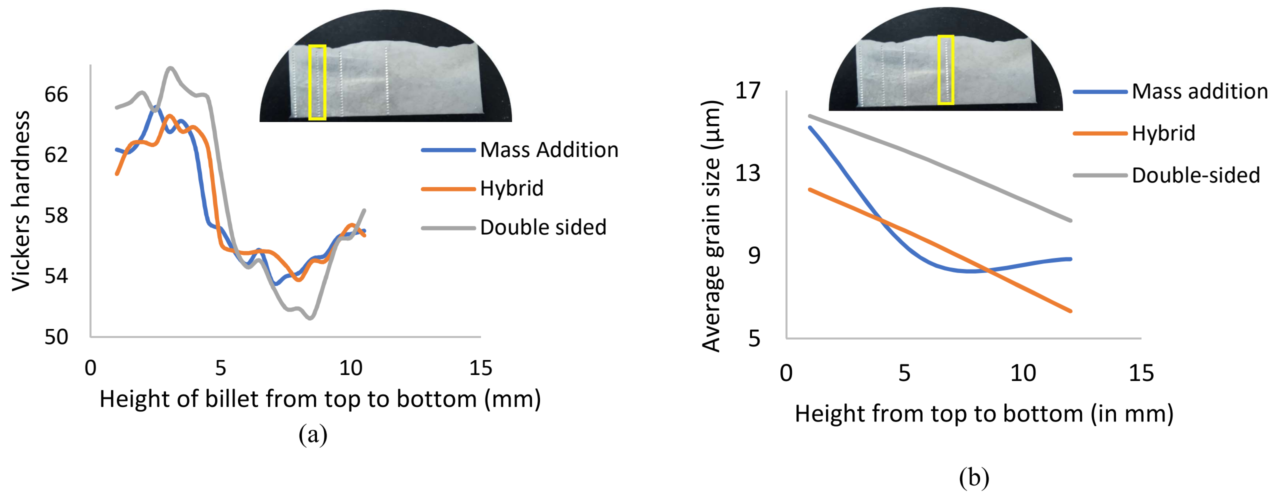
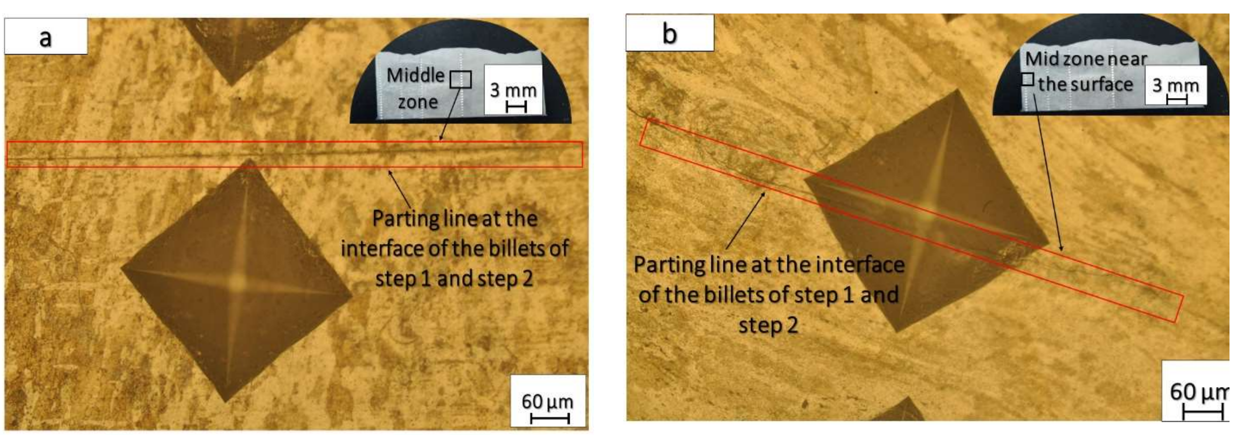


Fig. 12 The two-steps approach comparison of **a** hardness for Exp 2 at line r=9.0 mm, and **b** grain size distribution for Exp 1 at central

However, a sound bonding could not achieve between the billets of step 1 and step 2. In Exp 1 and Exp 2, a parting line (Fig. 13 a and b) was noticed by examining the cross-section. This separating line extended to the external surface during Exp 3, and finally, two separate billets were observed during Exp 4 (Fig. 13 c and d). The billet side that is in contact with the tool has a high surface roughness while the other surface is relatively smooth. Hence, this failure occurred because once the billet sides were flipped before step 2. The chips were added over the smooth surface (Fig. 14). As a result, the smooth surface did not enable bonding between the billet of step 1 and newly added chips. In fact, a smooth surface reduces friction induced temperature rise, such phenomenon does not allow bonding condition to be reached. Therefore, increasing tool speed further reduced friction between billets of step 1 and developing billet of step 2 that finally led to two different detached billets.



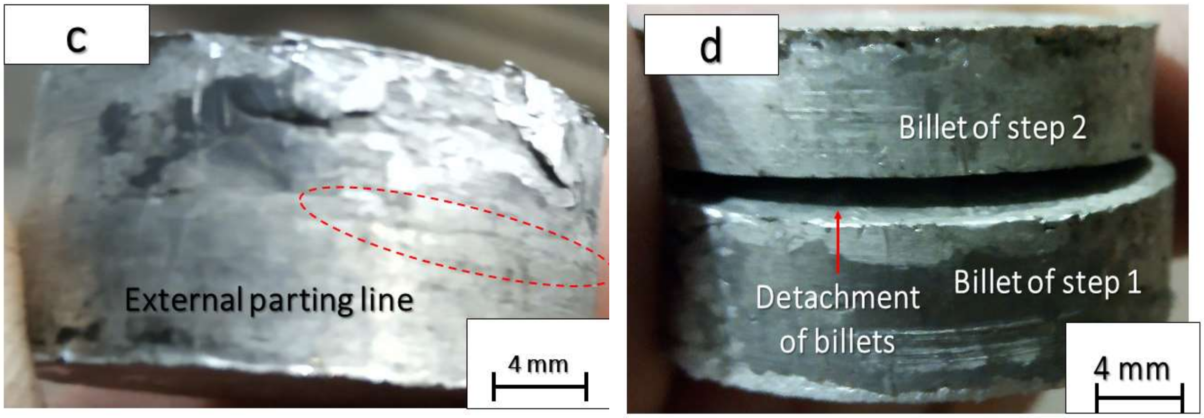


Fig. 13 Defects during two-steps hybrid approach: internal defects as partling line **a** Exp 1 and **b** Exp 2, and external defects **c** Exp 3, and **d** Exp 4

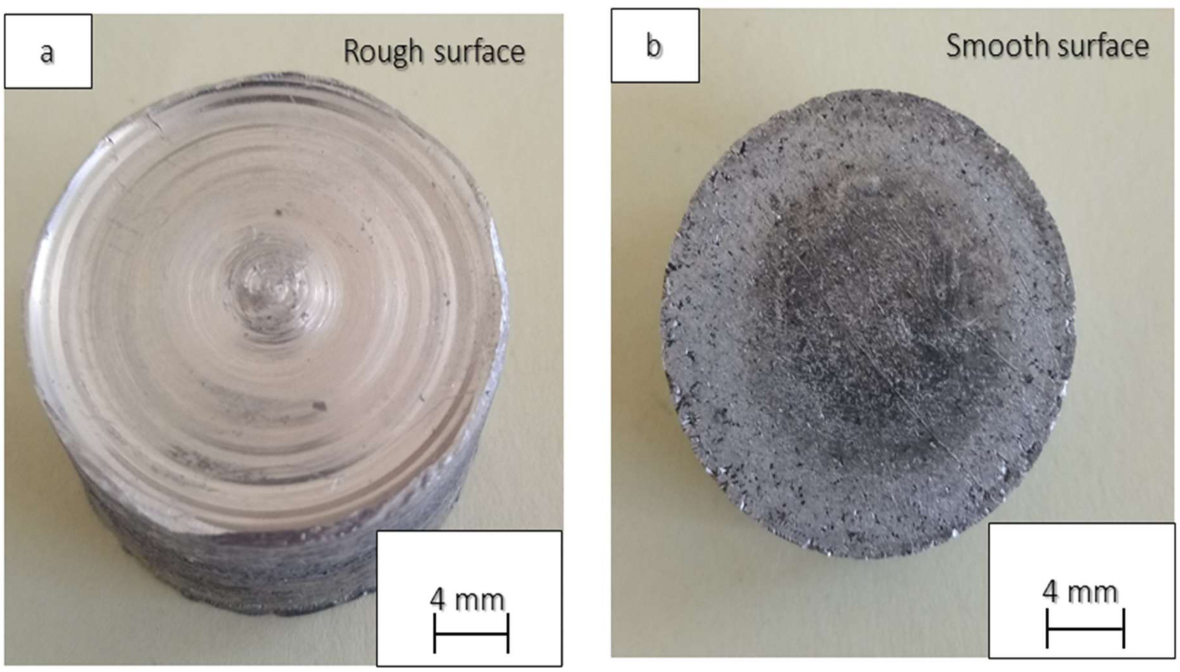


Fig. 14 Surfaces of the billet after consolidation **a** top surface, and **b** bottom surface

A 3-steps hybrid approach was applied to develop a sound bond between and validate the guess that a rough surface is crucial for bonding the two billets. Exp 4 was considered for this trial because it showed the worst results.

First, 10 g chips were consolidated for 20 sec, then the sides were flipped and consolidated the flat surface of the billet for 10 sec to get a rough surface. Finally, adding 10 g more chips and consolidated it for 30 sec (Fig. 15).

By adding this intermediate step, a sound bond was achieved between the two billets and proper mixed zone created between two billets as shown in Fig. 16a and Fig. 16b.

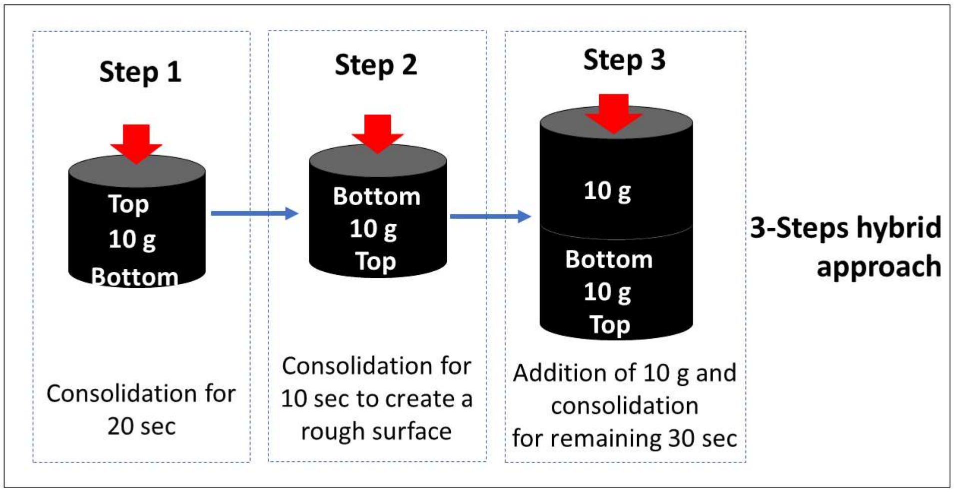


Fig. 15 Schematic of 3-steps hybrid approach

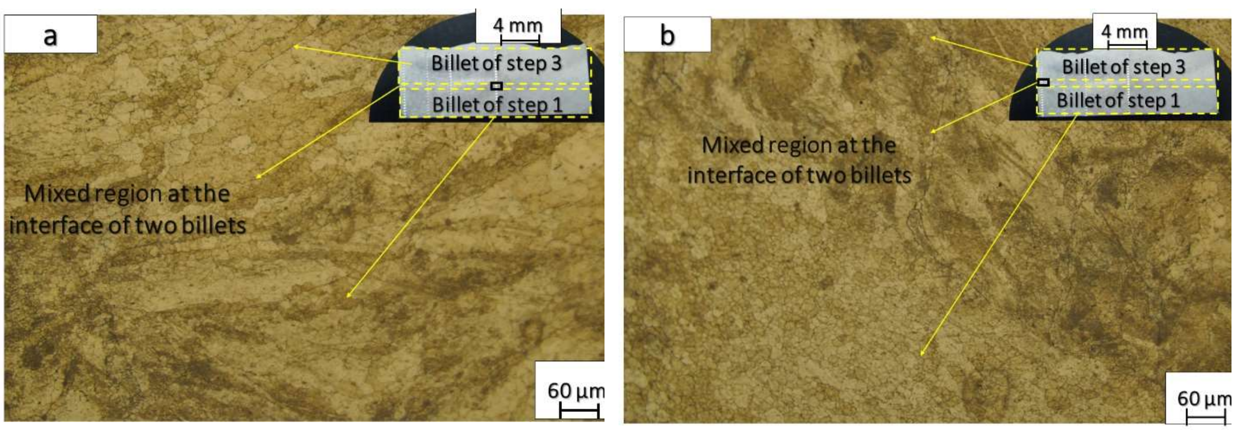


Fig. 16 Interface of two billets of three-steps hybrid approach at experimental conditions of Exp 4 configuration on lines at **a** r= 0 mm, and **b** r=12.25 mm

3.4 General discussion

This section provides a general overview of single-step and two-steps FSC methods. First, the entire area of the billet section was analyzed to find the non-defective area. Then, the billet section was assumed rectangular, and the total area was calculated using the diameter and height of the billet. It was noticed that the non-defective area (both absolute and percent) of all two-steps methods are far higher than the single-step approach, as shown in table 3.

A detailed comparison between single-step and two-steps FSC methods has been outlined in table 4. Overall, two-steps methods lead the single-step approach in all respects, including superior mechanical properties and the quality billet. However, the single-step approach is simple because the whole mass of the chips is consolidated in a single run. Therefore, it is comparatively time-efficient.

Table 3 Percent non-defective area during single-step and two-steps FSC process

<table>
<tr><th colspan="2">Approach</th><th>Exp #</th><th>% of non-defective area out of the total area</th></tr>
<tr><td colspan="2">Single-step</td><td>Exp 1
Exp 2
Exp 3
Exp 4</td><td>75.97
78.49
87.83
84.37</td></tr>
<tr><td rowspan="3">Two -steps</td><td>Mass addition</td><td>Exp 1
Exp 2
Exp 3
Exp 4</td><td>97.69
98.54
95.53
87.87</td></tr>
<tr><td>Double-sided</td><td>Exp 1
Exp 2
Exp 3
Exp 4</td><td>97.53
86.07
95.56
93.96</td></tr>
<tr><td>Hybrid</td><td>Exp 1
Exp 2
Exp 3
Exp 4</td><td>97.54
96.9
100
-</td></tr>
</table>

Table 4 Overview of single-step and two-steps FSC methods

Properties	Single step	Mass Addition	Double-sided	Hybrid
Full consolidation	No	Yes	Yes	No
Percentage area of no visible grain of the total area	15-25 %	1.5-12 %	2.5-14 %	0-4 %
Uniformity in hardness	Poor	Excellent	Good	Very good
Uniformity in grain size	Very poor	Very Good	Excellent	Very good
Process simplicity	Very Simple	Simple	Very complex	complex

4 Conclusion and further development

During the single-step FSC method, unconsolidated chips at the bottom and non-uniform mechanical properties across the billet section were the main challenges in recycling an upper bound of chips mass. To eliminate these issues, the value of rotational speed and processing time were increased based on previous studies. However, this strategy was ineffective, and hence new approaches were adopted in the present study to address these challenges. Thus, for the first time, two-steps FSC methods were introduced that successfully led to a fully consolidated billet with more uniform mechanical properties. Based on post-analysis of experimental data, the following conclusions are drawn:

- All two-steps FSC methods except the hybrid method successfully led to a fully consolidated billet that was the most challenging issue in recycling 20 g chips during the single-step FSC process.
- In terms of homogenous mechanical properties, a more uniform hardness profile was observed for mass addition that further improved with rising process parameters. A more uniform grain size distribution was noticed for the double-sided approach. However, the single-step FSC approach showed poor results in terms of homogeneity in hardness and grain size distribution profiles.
- Overall, the mass addition approach has the best mechanical properties and quality of billet among all FSC methods, including single-step and two steps.

- The mechanical properties of the hybrid approach were in between those of mass addition and double-sided approaches. However, the two-steps hybrid approach failed because the rough surface was a prerequisite for developing bonds between the billet of step 1 and the developing billet of step 2. Bond development was achieved by introducing an intermediate step to generate a rough surface.

It has been proved that multi-step FSC methods have superior mechanical properties and billet quality over the single-step FSC approach. However, it is also essential to fully explore the feasibility of the multi-step approach in the future. The potential factors for such assessment are energy consumption, power demands, and torque trends that can further lead to evaluate the environmental impact of multi-step FSC methods.

Declarations

-Ethical Approval the Authors Disclose potential conflicts of interest; also, the research here presented does not involve neither Human Participants or Animals.

-Consent to Participate: Not Applicable

-Consent to Publish: Not Applicable

-Authors Contributions:

Abdul Latif: Performing experimental campaign and hardness and grain size analyses, draft writing.

Giuseppe Ingarao: Conceiving the idea, analysis of the results and draft revision.

Marco Gucciardi: Performing experimental campaign.

Livan Fratini: Overall revision and research coordination.

-Funding: Not Applicable

-Competing Interests: The Authors disclose any financial and non-financial competing interests that could inappropriately influence, or be perceived to influence, this work.

-Availability of data and materials: Not Applicable

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