Assessing single and multi-step friction stir consolidated recycled billets through uniaxial upsetting test

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Abstract. Friction stir consolidation (FSC) represents a solid-state recycling technique that directly transforms machining waste into billets by the stirring action and friction heat of the rotating tool. This technology has demonstrated superior energy efficiency compared to remelting-based traditional recycling methods. Concerning the FSC process mechanics, they impart temperature, strain and strain rate gradients across the billet sections leading to variations in mechanical properties such as hardness values and grain size distribution. Therefore, multi-step FSC variants were introduced to eliminate this inherent inhomogeneity inside the billets of the single-step FSC approach. However, the billets manufactured by multi-step methods showed better mechanical properties, and gradation across the billet section still persisted. This property led to the evolution of friction stir consolidation as a new manufacturing method for developing functionally graded materials. Previously, the gradation was highlighted by the Vickers hardness test and grain size distribution. Therefore, in the ongoing study, an advanced characterization technique was adopted by extracting miniaturized samples from the section of FSC billets and subjected them to the upsetting test. Employing advanced characterization techniques to get insights into the gradation and quality of billets manufactured through various process variants and will allow to draw a better conclusion regarding adoption of a more suitable FSC process variant.

Introduction

The current global demand for aluminum is around 100 million tons per year [1] that is expected to rise further due to the growing application of aluminum in lightweight transport, packaging, construction, and electronic industries. This trend is putting immense pressure on industries to increase the production rate. Concerning the environmental impact, per ton of aluminum production generates 12-16 tons of greenhouse gas (GHG) during aluminum production from primary sources, which has severe adverse environmental impacts [2] and is responsible for almost 1% of total global energy consumption [3].

To address the rising demand for aluminum and achieve sustainability objectives, researchers are exploring the implementation of recycling practices. Interestingly, aluminum is an infinitely recyclable material [3]. In Europe, almost 35 % of the aluminum demand is met by recycling aluminum scraps [4]. The conventional recycling route involves re-melting the metal, and thus it skips many of the complex steps of aluminum extraction from the primary source. Furthermore, recycling is highly energy efficient, requiring 5% of the energy compared to obtain aluminum from the primary bauxite source. Thus, recycling can shift aluminum's energy and carbon balance sustainability towards much greater sustainability.

The conventional recycling method has further limitations when dealing with the recycling of aluminum machining chips. These chips, characterized by a high surface-to-volume ratio and susceptibility to oxidation, incur permanent material losses during the melting process. This results in adverse environmental impacts, increased costs, and substantial permanent material loss [5].

Looking at the proportion, the machining scraps constitute almost 17% of the total scraps [6]. Therefore, the researchers turned to solid-state recycling (SSR) techniques. SSR are those methods that transform metal scraps directly into finished or semi-finished billet through mechanical means such as high pressure, friction, rotational speed, and force [7]. Recently, SSR processes have been analyzed as a potential alternative for recycling machining scraps. Various SRR methods have been introduced for recycling metal chips based on the process mechanics and properties of the desired product [8-10]. In fact, by omitting the melting phase, permanent losses mainly due to oxidation are avoided.

Recently FSC has proved to be one of the environment recycling techniques compared to conventional remelting [11]. This technology has proved its potential beyond the concept of recycling toward upcycling method by manufacturing functionally graded material from metal scraps [12]. FSC has two main steps: compaction and consolidation. During compaction, chips or powder are pressed in a hollow die chamber by applying a specific load through a cylindrical tool. Then compacted materials are further pressed and stirred through the tool's downward force and rotational speed during the consolidation phase [13].

A typical FSC billet, due to process mechanics such as different temperatures, strain and strain rate distribution, is characterized by variation in mechanical properties along the radial and longitudinal direction of the billet [12, 13]. Previously, this variation was validated by Vickers's hardness test and grain size distribution analysis [14]. Exploring the potential of FSC in manufacturing advanced Functionally Graded Materials (FGMs) requires highlighting gradation through advanced characterization techniques. The concept of miniaturized upsetting specimens for local sheet metal characterization was introduced by Hetz et al. [15]. The test method can also be applied to analyze the local flow properties of bulk metal parts like the billets in this contribution. Previously, the authors of the current research, Latif et al. [16], extracted miniaturized samples for single-step FSC AA2011 to emphasize gradation in FSCed samples.

In specific, this research is the extended investigation of the previous study [16] aiming to highlight gradation for billets of different aluminum alloys: AA2011 and AA7075 manufactured through various process variants, namely, single-step and multi-step. In addition, this characterization method provides valuable insights into the local mechanical properties of the billets, enabling meaningful comparisons for the selection of the most suitable FSC process variants that are otherwise not possible using conventional characterization methods. The broader objective is to find a more accurate solution for the numerical modelling of the FSC process and find the potential industrial application.

Material and methods

Material and process set-up

The employed experimental setup consisted of ESAB LEGIO, a dedicated friction welding machine with 35 kN maximum capacity. A tool of H13 steel with 25 mm nominal diameter was integrated to the head of ESAB machine and a H13 steel die cylinder die with 25mm was fixed onto the working table as shown in Fig. 1.

As-received materials were a 20x20 mm squared bar of AA 2011-T3 and a 3 mm thick rolled sheet of AA7075. Both materials were reduced into chips by a milling operation with a rotational speed of 1250 rpm, a feed rate of 28 mm/min and a cutting depth of 1 mm. The chips were kept submerged in acetone for effective cleaning for 30 minutes which has proved as a potential washing solvent for cleaning metal scraps both at laboratory and industrial scales.

After drying, a constant mass of 15g cleaned chips was loaded inside the die and compacted with 5 kN load. The step of chips compaction was necessary in order to eliminate the possibility of sprinkling out chips during the following consolidation step. Finally, chips were consolidated with load of 20 kN pressing, 30s of processing and 1500 rpm through different FSC process variants according to the experimental plan as shown in Table 1. The choice of the process condition was driven by previous studies [12, 14].

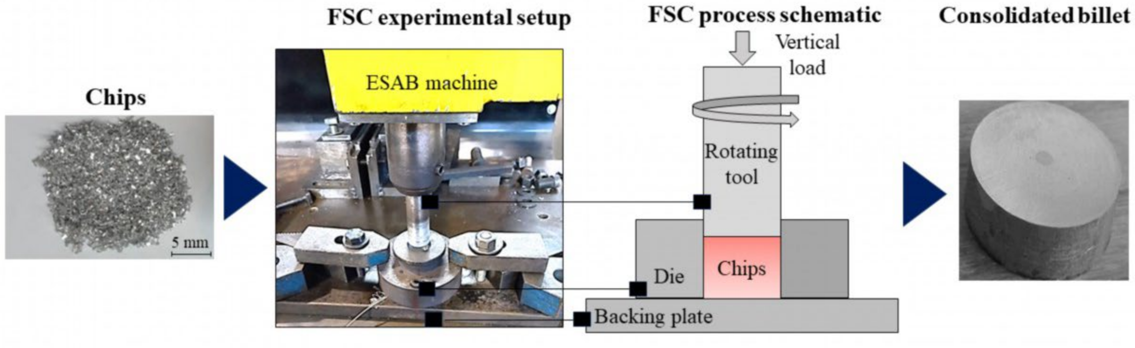

Fig. 1 Process chain of FSC process from scraps to consolidated billet

In particular three process variants: single step, double sided and mass addition methods were used for the billet manufacturing. During single step method, all input mass (15g) were compacted and consolidated in a single run for the total processing time of 30s. In double step method, likewise single step all input mass was consolidated for half of the total process time (15s) , then billet was removed from the die and reinserted up-side down by applying consolidation step for the half of the processing time. During mass addition approach, half of the total input mass (7.5 g) was consolidated for half of the total processing time while remaining half input mass was added and then total mass (15g) was consolidated for the remaining half of the processing time. The purpose of flipping side and step wise consolidation during double sided and mass addition methods respectively was to achieve billets with improved mechanical properties compared to single step FSC billet.

Table.1 Experimental plan for developing billets using different variants of FSC process

Exp ID	**Material**	**FSC process variants**	**Process conditions RPM-mass [g]-time [s]**
Exp 1	AA2011	Single step	1500-15-30
Exp 2	AA7075	Single step	1500-15-30
Exp 3	AA2011	Double sided	1500-15-30
Exp 4	AA7075	Double sided	1500-15-30
Exp 5	AA2011	Mass addition	1500-15-30
Exp 6	AA7075	Mass addition	1500-15-30

Measured outputs

After FSC consolidated step, the obtained billets had an average height and a diameter of 10.0 mm and 25.0 mm, respectively. The top and bottom surfaces of the FSC cylindrical billet were well polished to get a very flat surface for exact alignment of the billet in the following specimen extraction process as shown in Fig. 2. Then slices were cut (perpendicular to the cylindrical axis of the billet) from the top, middle, and bottom sections with a thickness of 1.5 mm and a diameter equal to the original billet diameter (25 mm) using die-sinking EDM. From each disc slice, five miniaturized cylindrical upsetting samples were extracted (Fig. 2) that had a height (h) of 1.5 mm (slice thickness) and a diameter (d) of 1 mm through micro-EDM on an SX-200-HPM (Sarix SA) as shown in Fig. 3. One sample (x1) was extracted from the center (r=0) and the remaining four samples (x2, x3, x4, and x5) were extracted at r=10 mm from the center of the billet. Upsetting tests were performed at a strain rate of 0.5%/s with upsetting sample dimensions given in Fig. 3. The height-to-diameter ratio (1.5) of the upsetting was selected based on the following equation [15].

$$1 \le \frac{h}{d} \le 2. \tag{1}$$

The selection of the height-to-diameter ratio was based on findings from a previous study [16]. Further, according to this equation (1), a higher ratio exceeding the maximum limit leads to the buckling of the upsetting sample, which is considered a premature failure. On the other hand, a lower ratio increases the effect of the frictional influence. Therefore, Dionol ST V 1725-2 (MKU-Chemie GmbH) lubricant was applied to minimize friction.

In addition, hardness was measured across the section of the of FSC billet at constant pitch of 1.5 mm in radial and cylindrical directions. In particular, Vickers hardness test was performed by applying 49 N load for dwell time of 15s. The results of both upsetting test and hardness measurements were compared to get better insights into quality and gradation.

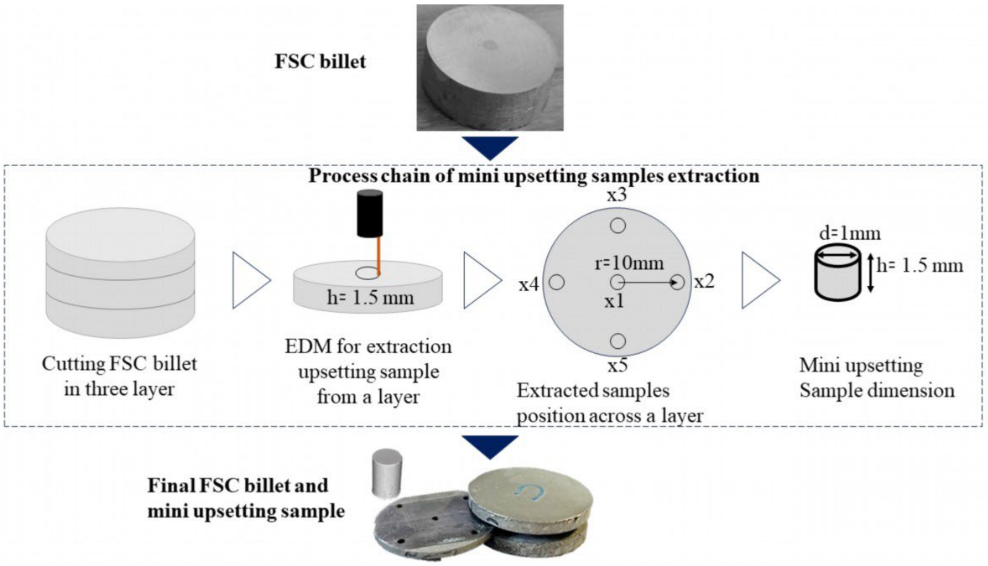

Fig. 2 Process sketch miniaturized upsetting sample extraction

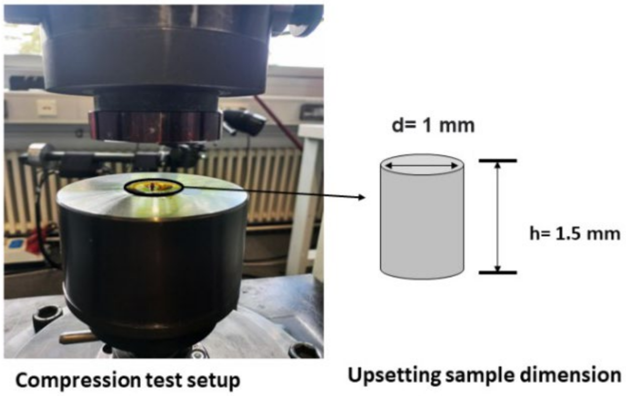

Fig. 3 Upsetting test setup and sample dimensions

Results and Discussion

This section provides details information about the true strain-strain curves of the FSC billet of AA2011 and AA 7075 manufactured through single step and multi-step methods. AA2011 single billet that has been already analyzed by authors of the current study [16], is taken as reference for detailed discussion and then influence of change in material and process variants are analyzed. Further, FSC billets plastic material behavior is then compared to as-received material ones to understand the changes made by FSC process in the mechanical properties of the materials. In addition, a correlation is also established between strength and hardness of FSC billet by analyzing the ultimate strength and hardness values for the zones of the interest particularly in top, middle and bottom layers of the FSC billet.

True-stress strain analysis of AA2011 single step FSC billet

This section gives details information about the true strain-strain curves of the FSC 2011 billet with evidence of how far true stress-strain graphs agree with the hardness distribution profile across the FSC billet section. Further, a comparison was also made between the true stress-strain curve FSC recycled billet and as-received material AA2011 bar.

On the top slice (Fig.4 a and b), the central sample (x1) showed higher strength compared to the remaining upsetting samples (x2 to x5). The flow curves of the remaining four were found almost aligned with each other. The reason was samples x2 to x5 were extracted from the same height (top slice) and same radial distance (r=10 mm). Interestingly, the variation in properties is enabled by FSC process mechanics, i.e., different strain rate and temperature distributions across the billet section, as reported by Tang et al. [14]. In addition, a preliminary analysis has been provided by Puleo at al. [17] but it is quite evident that the die wall and back plate serve as heat sinks, reducing the temperature. Due to this, chips close to the die wall and back plate don't receive sufficient heat input and deformation to be fully recrystallized, but some bonding is, however, achieved.

Likewise, the top slice flow curves, almost the same trend was found for the middle slice. The central specimen (y1) showed higher strength than the other samples y2 and y3 (Fig. 4c).

For all samples of the bottom slice, almost all flow curves were found aligned with each other indicating less variation in mechanical properties. It is evident that the strength of the material varies both in the radial and longitudinal direction of the billet. Further, the top part of the billet shows higher yield strength than the bottom part, and along the radial direction the central part is stronger than the region near the external surface. Also, along the radial direction, the difference in true stress-strain curves between the samples at the center (x1) and the other samples (x2 and x3) is higher for the top slice, then difference diminishes at the middle slices, and finally, all curves are aligned with each other for the bottom slice (Fig. 4c).

Interestingly, the trend of true stress-strain profile was found consistent with the hardness distribution trend (Fig. 4c and d). For example, the zone of the billet section that exhibited higher hardness value also showed higher strength. The hardness measurements were performed on the same material, AA2011, manufactured under the same process conditions.

Again in Fig. 4d the middle of the billet section shows a minor variation in hardness value along the radial direction. A similar relationship can be found for the flow curves of middle slice y1 and y2/y3 in Fig.4c.

For the bottom section, a lower but uniform hardness value can be noticed. Likewise, lower strengths but alignment of curves were noticed for the true stress-strain graphs of the bottom section (z1, z2, and z3).

It is evident from true stress-strain curves that ultimate strength followed exactly the same trend as found in the case of hardness distribution. Overall, the central, and top zones show higher strength and higher hardness values in comparison to the part near the external surface and bottom section, where relatively low hardness values were noticed.

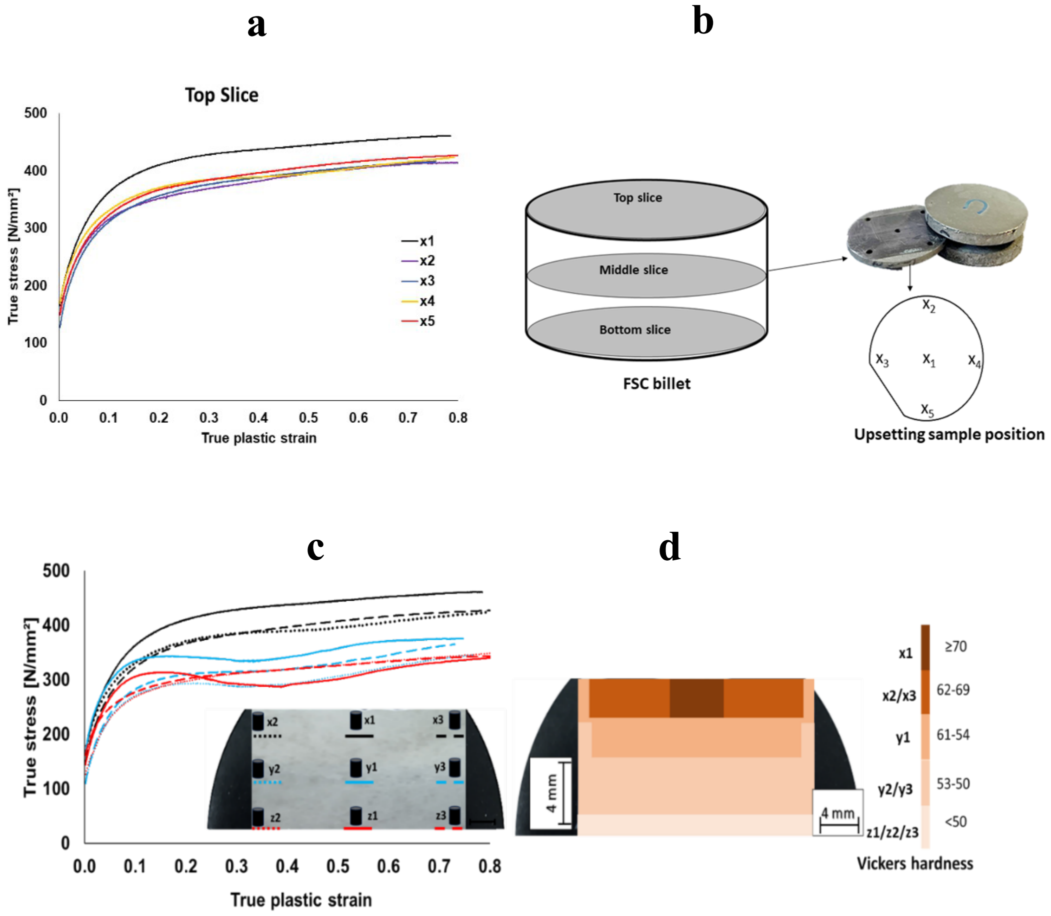

Fig.4: True stress-strain curves for top, middle and bottom slices of AA2011 FSC billet and comparision with hardness [16]

Influence of materials by analyzing strength of single step FSC AA7075 billet

To assess the impact of materials, upsetting tests were conducted on miniaturized samples extracted from the center of each from AA7075 FSC billet section.

The trend for strength profiles of AA7075 FSC billets was found similar to AA2011. The highest strength was observed for the specimen at the top section followed by the sample at the middle slice. Concerning the bottom section of AA7075, the lowest strength value could be expected but due to poor chips bonding and the existence of defects such as air cavities, it was not possible to extract the upsetting sample Fig. 5. These issues are primarily attributed to the difficulty in consolidating AA7075 material. Comparing to AA2011 FSC billets for the specimen of top slice (Fig. 6) extracted from center, AA7075 upsetting sample showed higher strength but lower plastic deformation under the same manufacturing process and upsetting conditions. This trend can be attributed to the fact that AA7075 presents the class of aluminum alloys with the highest strength and relatively lower ductility compared to the other series of aluminum alloys.

On comparing the true stress-strain curves of the single-step FSC billet AA7075, extracted from the center of the top slice, with the as-received AA7075 sheet, the FSC billet exhibited lower strength. However, it is evident that this decrease in strength can be attributed to the premature failure of the specimen, as all samples extracted from the FSC billet AA7075 failed during upsetting tests.

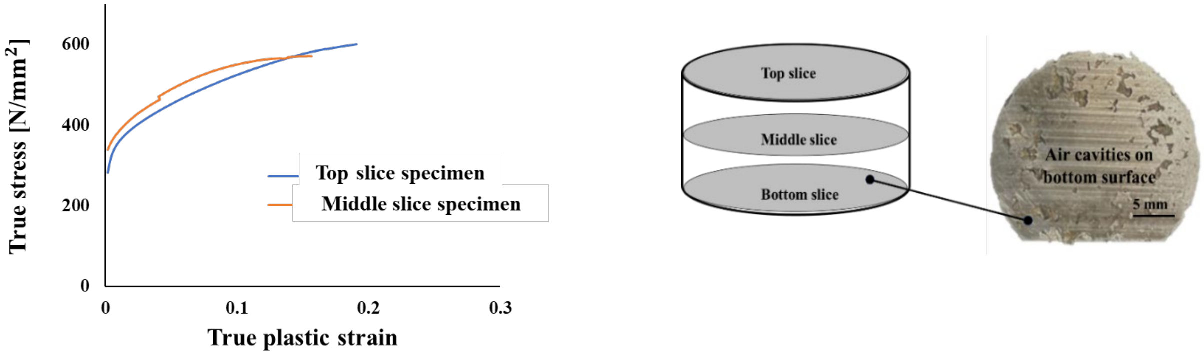

Fig. 5 True stress-strain curves of AA7075 for single step FSC billet samples x1 and y1

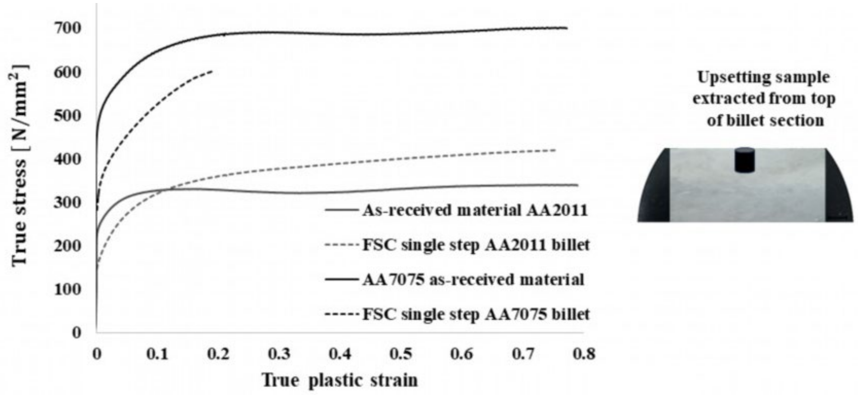

Fig. 6 True stress-strain curves for AA7075 and AA2011 as-received materials and single step FSC billets sample x1

Effect of Process variants

An improvement was noticed for hardness values and grain size distribution with minimal defects at the bottom of the billet in the case of multi-step FSC methods [14]. Therefore, the same upsetting testing method was also applied to the billets of multi-step FSC approach to compare results with the single-step approach. Employing advanced characterization techniques to assess the billet quality will allow to draw a better conclusion regarding the adoption of a more suitable FSC process variant.

Double sided method: This FSC approach was primarily introduced to enhance the mechanical properties especially at the bottom zone of the FSC billet. It is clear from Fig. 7 and Fig. 8 that an improvement occurred at bottom zone's strength compared to the middle zone for both AA2011 and AA7075. A similar enhancement was also noted in previous studies [14] during hardness measurements for AA2024-O. In the case of ductile material (AA2011), upsetting tests were successfully conducted for samples from all zones (top, middle, and bottom). However, for AA7075, premature failure occurred, possibly due to its less ductile nature inherited from the as-received material. In addition, highest plastic deformation was shown by specimens at top zone, followed by specimen at bottom zone and the lowest strength and plastic deformation shown by the upsetting specimen at the middle zone of the double sided FSC AA7075 billet.

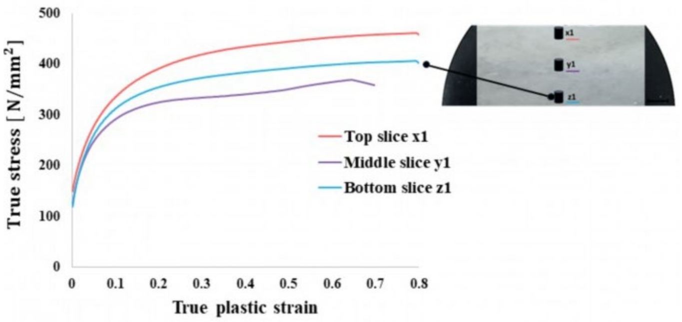

Fig.7 True stress-strain curves for FSC AA2011 billet obtained double sided approach (Exp 3)

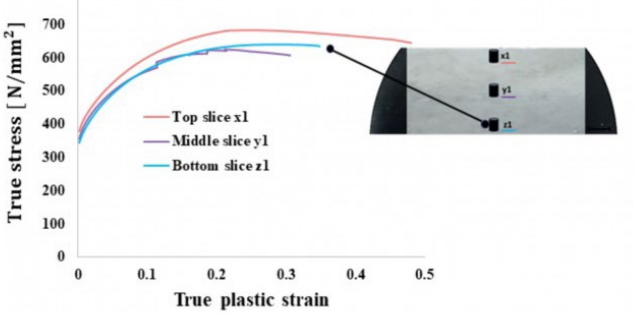

Fig.8 True stress-strain curves for FSC AA7075 billet obtained double sided approach (Exp 4)

Mass addition method: The mass addition approach involves recycling the desired input mass in a stepwise manner, typically in two steps. Primarily this method was also introduced to enhance the mechanical properties, especially in the bottom zone of the FSC billet.

Likewise double sided approach, mass addition also led to increase the strength of the bottom zone of the billet compared to the strength of middle section especially in the case of ductile material AA2011 (Fig. 9). On the other hand, the middle section was affected by air cavities and several other defects possibly indicating that favorable bonding conditions were not achieved between the billet of step 1 and step 2 during the mass addition approach for AA7075. Comparing to single-step approach, the defective zone with air cavities moved from bottom to middle section (Fig. 10).

However, both the top and bottom specimens exhibited the highest plastic deformation when compared to the single-step and double-sided approaches.

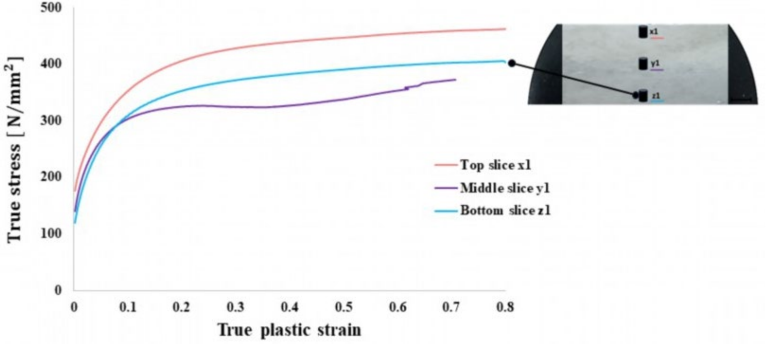

Fig.9 True stress-strain curves for FSC AA2011 billet obtained through mass addition approach (Exp 5)

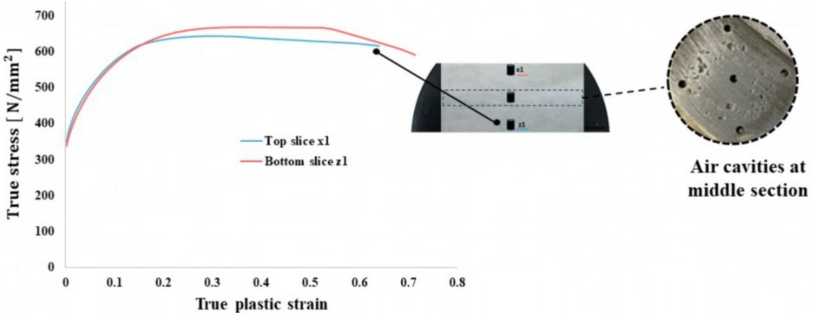

Fig. 10 True stress-strain curves for FSC AA7075 billet obtained through mass addition approach (Exp 6).

Overall, the local strength of the FSC billet depends on various factors, including the input material, process variant, and process conditions. The double-sided approach of the FSC process exhibited the highest strength, followed by mass addition and single-step methods, respectively.

In the context of material, the performance of the FSC billet improved with the ductility of the material. For the sake of better understanding the top zone was selected as no FSC billet was found defective at this zone (Fig. 11). The top zone exhibited significantly higher strength than the as-received strength in the case of AA2011 for all process variants. For AA7075, the strength of FSC multi-step approach billet was comparable to as-received while single step approach billet strength was relatively very low. Further, the bottom zone was found defective in single step, which then moved to the middle section in the case of mass-addition method. This suggests that the double-sided approach is the only potential FSC process variant for recycling very high or low ductile aluminum alloy chips.

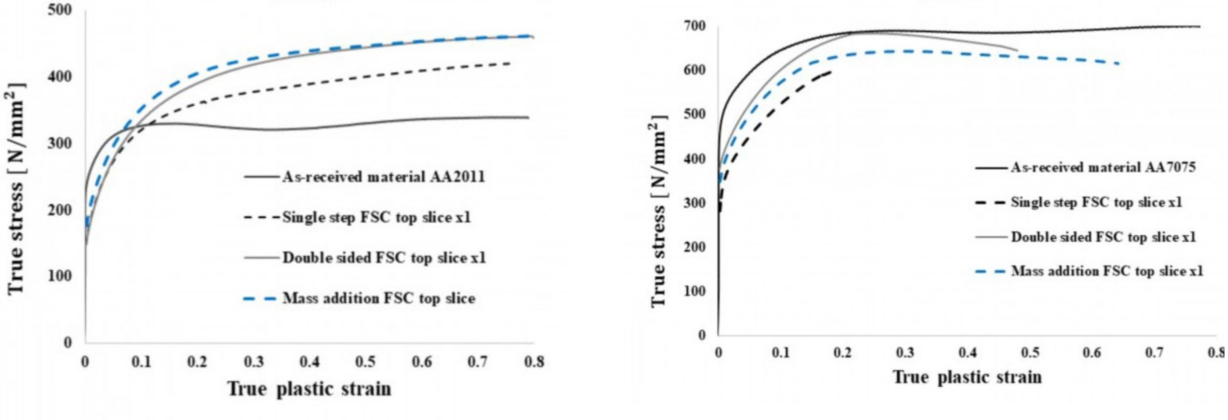

Fig.11 Comparison of true stress-strain curves for as received material and top slice (x1 sample) of different FSC billets for AA2011 (on left), and AA7075 (on right)

Conclusion

Overall, the performance of upsetting tests on miniaturized samples of FSC billet proved effective in obtaining local mechanical properties. This study also provides supportive results in confirming the gradation in the properties of FSC billet.

The flow curves were found in good agreement with the hardness trend across the billet section. A higher hardness value in a specific zone corresponds to increased strength, and conversely, a lower hardness value in another zone corresponds to reduced strength.

Further, this study highlighted the unique feature of the mass addition approach during recycling AA7075 chips. In particular, the failure of upsetting samples was noticed at the mid-zone, due to porosity and defects. This aspect was not possible to analyze during hardness measurement.

Concerning the process variants, double sided approach proved effective for producing FSC billets and offering relatively better consolidation to the FSCed billet and therefore outperformed single-step and mass addition methods.

Regarding the material aspect, upsetting samples were successfully performed on billets of AA2011 while failures occurred during AA7075 billet for all FSC process variants, highlighting the positive influence of material ductility on upsetting test outcomes.

The future work can be dedicated to assessing the formability of recycled billets through forgeability tests. Additionally, new possibilities should be explored for developing more advanced materials using the FSC process.

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References

[1] Information on https://alucycle.international-aluminium.org/public-access/

[2] T.G. Gutowski, S. Sahni, J.M. Allwood, M.F. Ashby, E. Worrell, The energy required to produce materials: constraints on energy-intensity improvements, parameters of demand, Philosophical Transactions of the Royal Society A: Mathematical, Physical and Engineering Sciences 371.1986 (2013): 20120003. https://doi.org/10.1098/rsta.2012.0003

[3] D. Raabe et al., Making sustainable aluminum by recycling scrap: The science of "dirty" alloys, Progress in Materials Science. (2022): 100947. https://doi.org/10.1016/j.pmatsci.2022.100947

[4] Information on https://www.iea.org/reports/material-efficiency-in-clean-energy-transitions

[5] J.R. Duflou, A.E. Tekkaya, M. Haase, T. Welo, K. Vanmeensel, K Kellens, W Dewulf, D. Paraskevas, Environmental assessment of solid state recycling routes for aluminium alloys: can solid state processes significantly reduce the environmental impact of aluminium recycling?. CIRP Annals (2015) 64(1) pp 37-40. https://doi.org/10.1016/j.cirp.2015.04.051

[6] U.M.J. Boin and M. Bertram. Melting standardized aluminum scrap: A mass balance model for Europe. Jom, 57(8), pp.26-33. https://doi.org/10.1007/s11837-005-0164-4

[7] B. Wan, W. Chen, T. Lu, F. Liu, Z. Jiang, M. Mao, Review of solid state recycling of aluminum chips, Resources, Conservation and Recycling.125 (2017):37-47. https://doi.org/10.1016/j.resconrec.2017.06.004

[8] M. Haase, N.B. Khalifa, A.E. Tekkaya, W.Z. Misiolek, Improving mechanical properties of chip-based aluminum extrudates by integrated extrusion and equal channel angular pressing (iECAP) Materials Science and Engineering A (2012) 539 pp 194-204. https://doi.org/10.1016/j.msea.2012.01.081

[9] F. Widerøe, T. Welo, Using contrast material techniques to determine metal flow in screw extrusion of aluminium. Journal of Materials Processing Technology (2013) 213(7) pp 1007-18. https://doi.org/10.1016/j.jmatprotec.2012.11.013

[10] D. Paraskevas, S. Dadbakhsh, J. Vleugels, K Vanmeensel, W Dewulf, J Duflou, Solid state recycling of pure Mg and AZ31 Mg machining chips via spark plasma sintering Materials & Design (2016) 109 520-29. https://doi.org/10.1016/j.matdes.2016.07.082

[11] G. Buffa, D. Baffari, G. Ingarao, L. Fratini. Uncovering technological and environmental potentials of aluminum alloy scraps recycling through friction stir consolidation, International Journal of Precision Engineering and Manufacturing-Green Technology (2020) 7/5 pp 955-64. https://doi.org/10.1007/s40684-019-00159-5

[12] A. Latif, G. Ingarao and L. Fratini. Multi-material based functionally graded billets manufacturing through friction stir consolidation of aluminium alloys chips. CIRP Annals.

[13] W. Tang, A.P. Reynolds, Friction consolidation of aluminum chips, Friction Stir Welding and Processing VI. 289 (2011). https://doi.org/10.1002/9781118062302.ch34

[14] A. Latif, G. Ingarao, M. Gucciardi, L. Fratini, A novel approach to enhance mechanical properties during recycling of aluminum alloy scrap through friction stir consolidation The International Journal of Advanced Manufacturing Technology (2022) 119(3) pp 1989-2005]. https://doi.org/10.1007/s00170-021-08346-y

[15] P. Hetz, P, M. Kraus and M. Merklein. Characterization of sheet metal components by using an upsetting test with miniaturized cylindrical specimen. CIRP Annals.

[16] LATIF, A., INGARAO, G., FRATINI, L., HETZ, P., & MERKLEIN, M. (2023). Characterization of friction stir consolidated recycled billet by uniaxial compression tests with miniaturized cylindrical specimen. MATERIALS RESEARCH PROCEEDINGS, 28, 1997-2004. https://doi.org/10.21741/9781644902479-215

[17] Puleo, R., Latif, A., Ingarao, G., Di Lorenzo, R., & Fratini, L. (2023). Solid bonding criteria design for aluminum chips recycling through Friction Stir Consolidation. Journal of Materials Processing Technology, 319, 118080. https://doi.org/10.1016/j.jmatprotec.2023.118080