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ORIGINAL RESEARCH

# AZ31 magnesium alloy recycling through friction stir extrusion process

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**Abstract** Friction Stir Extrusion is a novel technique for direct recycling of metal scrap. In the process, a dedicated tool produces both the heat and the pressure to compact and extrude the original raw material, i.e., machining chip, as a consolidated component. A proper fixture was used to carry out an experimental campaign on Friction Stir Extrusion of AZ31 magnesium alloy. Variable tool rotation and extrusion ratio were considered. Appearance of defects and fractures was related to either too high or too low power input. The extruded rods were investigated both from the metallurgical and mechanical points of view. Tensile strength up to 80 % of the parent material was found for the best combination of process parameters. A peculiar 3D helical material flow was highlighted through metallurgical observation of the specimens.

**Keywords** Recycling · Magnesium alloys · Material flow · Friction stir extrusion

## Introduction

The production cycle used for most of the mechanical components include one or more machining operation. Consequently, a huge amount of material is lost as scrap during the manufacturing of structural materials components by traditional cutting processes [1]. The produced chip is characterized by irregular geometry, presence of contaminants, and is usually made of different alloys. Currently two main techniques are used for recycling of metals, namely the "conventional" method, which implies the melting of the material to be recycled and the casting of a new billet, and the direct conversion method [2].

Chip is the most difficult kind of scrap to be recycled via fusion processes. Differently from other scraps, it is characterized by elevated surface/volume ratio, low density and it is usually oxidized and covered by different types of lubricant used for the machining process. Due to the above-cited features, conventional recycling of metals may lead to a few different drawbacks. In particular, both environmental issues, i.e., fumes and gas formation, energetic/economic issues, i.e., low percentage of obtained material and high energetic cost, and technological issues, i.e., low quality of the final product characterized by porosities, inclusions and low mechanical resistance, arise when conventional recycling is used. Recyclability by melting of both aluminum and magnesium alloys has been studied during the last few years [3, 4]. Overall, during the entire conventional recycling process the recovery rate hardly reaches 50 %. Additionally, the high energy consumption and the large number of operations needed for the conventional recycling method (e.g., cleaning, drying, compacting, melting and extrusion) make this technique inadequate for the industrial and societal needs of the next decade.

Gronostajski and Matuszak [2] first introduced the direct conversion method. The chip is segregated

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according to the chemical composition, cleaned, comminuted and finally compacted and hot extruded at temperature ranging between 500 and 550 °C. Direct chip recycling is a relatively simple technique characterized by low environmental impact with respect to the conventional method and allows for high recovery rates. Fogagnolo [5] highlighted how hot pressing before hot extrusion results in a better product quality while cold pressing results in reduced costs. Hu et al. [6] demonstrated the applicability of the direct method to magnesium alloys and analyzed the influence of three chip geometry on the quality of the final product. Overall, the final extruded product presents low porosity and high relative density, namely the ratio between the density of the extruded object and the density of parent material, equal to about 98 %. Large values of die temperature and extrusion rate are needed in order to optimize the quality of the final product. Compared to the conventional method, the direct method allows significant savings in terms of material—up to 40 %—energy—between 26 and 31 %—and labor—between 16 and 61 %. However, the shape and the dimension of the chip is a key factor for successful recycling. The irregular spiral geometry of the chip make it unsuitable for the compacting stage. Hence, the cutting and comminuting stage are needed in order to obtain good quality recycled material.

In 1993 TWI patented a new chip recycling process named Friction Stir Extrusion (FSE). Using the heat and the plastic deformation generated by the friction between a rotating tool and the chip to be recycled, the processed chip is compacted, stirred and extruded. In this way, a unique process allows the transformation of the chip into an extruded product, resulting in significant cost, energy and labor saving with respect to both conventional method and direct method. For this reason, the FSE technique appears very attractive to industry for the recycling of machining chips. Unfortunately, the process is still in its early developing stage. After the TWI patent expiration due to failure to pay maintenance fee in 2002, only very few papers can be found in literature on the process. In particular, Tang and Reynolds [7] produced AA2050 and AA2195 wires from chips using fixed extrusion force and varying tool rotation. The microstructure of the extruded wires is characterized by small equiaxed grains resulting in good mechanical properties of the wires in terms of microhardness and bend ductility. Canter [8] demonstrated that significant energy savings of more than 80 %, compared to the current melting and casting method, could be obtained.

Magnesium alloys have a great potential for parts weight and fuel consumption reduction [9]. However, most Magnesium is produced through the Pidgeon process which is characterized by intensive energy usage and generates a large amount of greenhouse gas (GHG) emissions, which may offset the potential advantage of using Mg parts in automobiles [10]. A few papers focusing on direct recycling of Magnesium can be found in literature. In particular, the effect of size chips and extrusion ratio on the main microstructural and mechanical properties [11–13] and the corrosion properties of the extruded product [14] have been investigated for solid state recycled Mg alloys.

However, no paper on FSE of Mg alloys is known by the authors. Although FSE can be considered extremely competitive even with respect to the direct method, as near-zero emissions of carbon dioxide and no emissions of metal oxide particulates are produced, the real potential of the process has not been still highlighted due to the significant knowledge gap present in literature.

In this paper, the results of an experimental campaign on the FSE of AZ31 Mg alloy are shown. The as received material has been machined and the obtained chips have been Friction Stir Extruded with varying tool rotation and extrusion ratio. During the tests, the required forces have been measured. The micro and macro mechanical properties of the extruded rods have been investigated in terms of microhardness and ultimate tensile strength.

## Material and methods

AZ31 magnesium alloy was utilized for this study. The considered magnesium alloy is characterized by yield strength equal to 150 MPa, Ultimate Tensile Strength (UTS) equal to 250 MPa. The raw material was received in plates and milled with no lubricants in order to produce a regular and clean chip. The produced chip has an average dimension of 5 mm length, 2 mm width, and 0.2 mm thickness (Fig. 1).

A dedicated fixture was designed and built for the experimental campaign. In particular, both the rotating tool and the extrusion chamber were made in AISI H13 steel quenched at 1020 °C and characterized by 52 HRc hardness. The rotating tool has an outer diameter equal to 25 mm and a 10° conical surface in order to increase the contact area between the tool

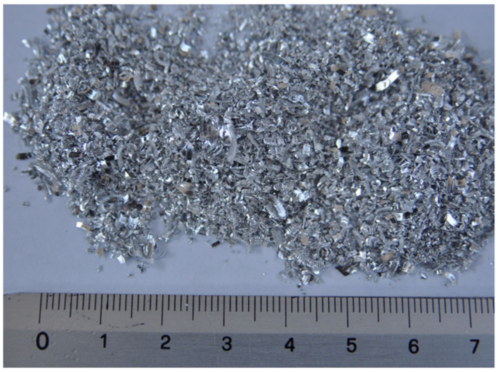

**Fig. 1** Chip used for the experiments

and the material to be extruded and to convey the material flow toward the tool center, i.e., the extrusion channel. At the end of each experiment, the entire chip previously loaded into the extrusion chamber was found either as extruded wire or as plasticized layer bonded to the conical surface of the tool.

Friction Stir Welding machine ESAB LEGIO was used for the experimental campaign. All the tests were carried out with constant extrusion rate, equal to 0.5 mm/s. Variable tool rotation, corresponding to variable heat input to the chip, and extrusion ratio were used. In particular, three different values were used for the tool rotation, namely 300, 500 and 700 rpm, while two extrusion ratios were considered, equal to 5 and 3.57, corresponding to a final diameter of the extruded parts of 5 and 7 mm, respectively. In this way a total of six different case studies were investigated. Each tests was repeated three times and, from each rod, specimens were cut for tensile tests and micrographic analysis. Figure 2 shows a sketch of the process. The chip closer to the tool, i.e., closer to the heat source, rotates together with the tool and plasticizes due to the combined effect of high temperature and stirring. Moving far from the tool interface, a transition layer is encountered, in which the chip is heated but has not been homogenized as a continuum material. The extrusion starts from the rotating plasticized layer and is influenced by the combined action of tool rotation and extrusion rate. At the end of the process the extruded material returns to room temperature by calm air cooling. The microstructure of the final extruded parts is significantly affected by this complex material flow, as it will be highlighted in the next paragraph.

The specimens were hot mounted and polished according to ASTM E407-09 standard and constantly doused with ethanol to avoid water contamination. The samples were then etched for 12 s by a picric reagent composed of 1 ml of picral 4 (4 % *w/w* ethanol solution of picric acid), 1 ml of distilled water and 7 ml of glacial acetic acid. The microstructure was then observed by an optical microscope. Microhardness was measured along the wire diameter with measuring points equally spaced by 0.5 mm.

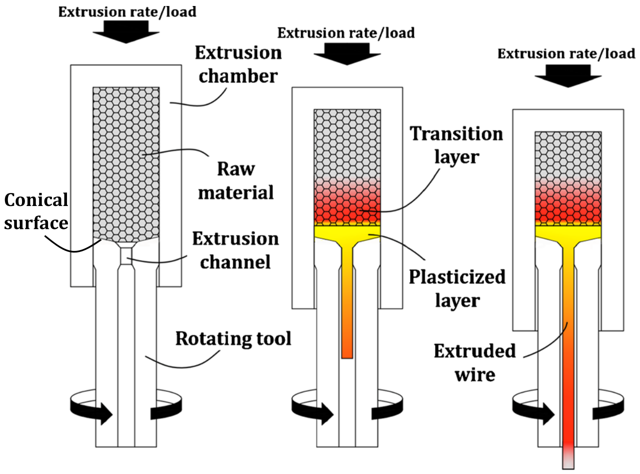

**Fig. 2** Sketch of the Friction Stir Extrusion process

## Results

Tensile tests were carried out on the produced rods. The results are reported as a percentage of the UTS of the parent material (Fig. 3). From the obtained results, it can be stated that the tool rotation plays a key role in the process efficiency. In particular, when 5 mm rods are considered, no extrusion can be obtained when rotational speed is equal to 300 rpm. As the tool rotation increases to 500 rpm, a sound rod is extruded. The mechanical performances of the extruded rods further increase with tool rotation of 700 rpm. When the 7 mm rods are taken into account, i.e., with a smaller extrusion ratio, a rod can be extruded also at 300 rpm. However, this is characterized by an extremely poor mechanical resistance and an irregular surface with several cracks can be observed on the external surface (see Fig. 3). This is due to the poor heat input conferred to the chip. In this way, proper softening conditions needed for the activation of the process are not reached. On the other hand, with increasing tool rotation, better resistance is found, with a maximum value equal to 80 % obtained for the 700 rpm case study. This specimen is also characterized by a regular outer surface with no cracks (see Fig. 3).

The extrusion force needed to carry out the process with constant extrusion rate of 0.5 mm/s was measured (Fig. 4a and b).

During the first few seconds of the process, the tool compacts the chip. Hence, only a very small load is observed and actual extrusion has not started. When the chip is sufficiently impacted to offer resistance to the extrusion tool, heat is generated due to the friction forces work, the material homogenizes and the extrusion process can initiate. This corresponds

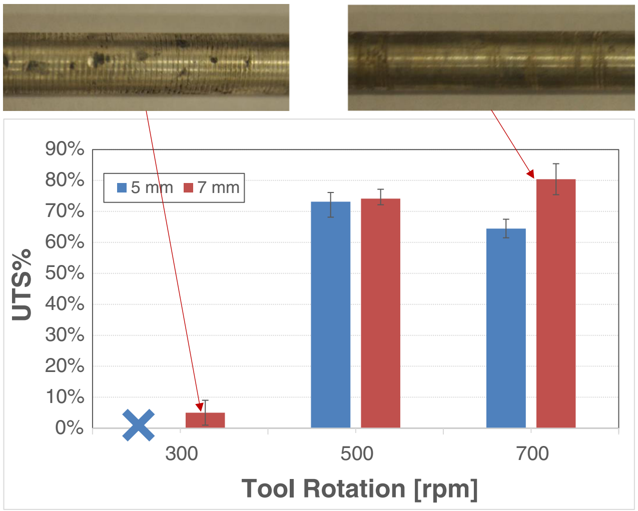

**Fig. 3** Tensile tests as a percentage of the UTS of the parent material for the considered case studies

to an increase in the force measured. Looking at the variation of the extrusion force with rotational speed for the 7 mm case studied, it arises that higher forces are obtained with small tool rotation values, indicating that the material is colder and the activation energy of the extrusion process is higher. Maximum force values, obtained at the end of the process, when only a small amount of material is left in the extrusion chamber, range between 12 and 18 kN. This result is consisted with the one obtained in [7], in which an aluminum alloy, with mechanical resistance comparable to the used AZ31 Mg alloy, was extruded with constant extrusion force of 18 kN. It is worth noticing that small differences in the initial compacting can delay the onset of the extrusion process. This can explain why between 15 and 25 s the 700 rpm case study requires force higher than the 500 rpm case study. When the forces measured for the two extrusion ratios are compared for two different case studies, a significant difference is found. Similar trends are observed for the 700 rpm case studies, both resulting in sound rods, with larger force values measured for the larger extrusion ratio, as expected. As the 300 rpm case studies are considered, again the load obtained for the 7 mm case study shows a similar trend with respect to the previous tests. On the contrary, when 300 rpm and 5 mm are selected, the extrusion force rapidly increases. Due to the low heat input, the extrusion process is not activated and the force vs time profile is similar to the one of a closed die forging process. In these conditions, the maximum load of the utilized machine is reached after about 23 s from the beginning of the process and the rod cannot be produced.

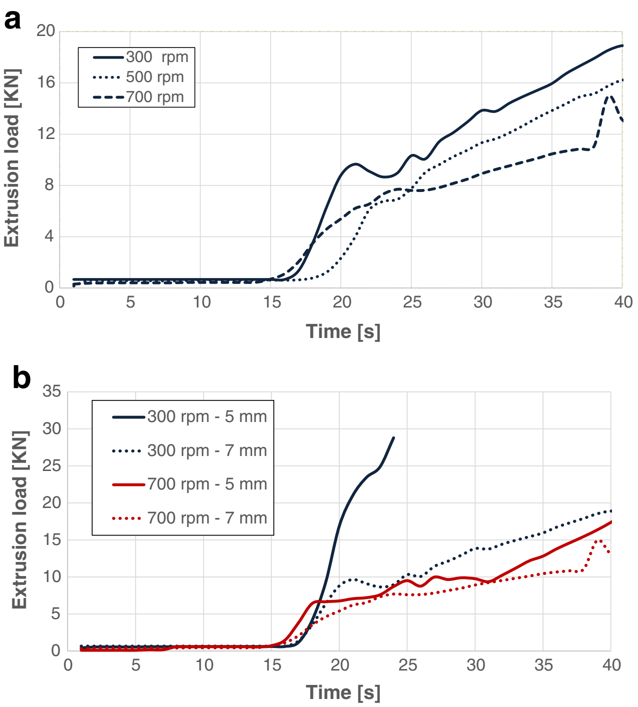

**Fig. 4** **a** Extrusion force measured for the 7 mm case studies and **b** comparison between 5 mm and 7 mm for the 300 rpm and 700 rpm case studies

In order to better understand the process mechanics, macro and micro images of the cross sections of the produced wires have been acquired. In Fig. 5, the macro images of the considered case studies are shown.

As it could be expected from the tensile test results, when 300 rpm are selected a large flow defect is found in the cross section of the specimen. In particular, only the outer surface of the rod was extruded and a final tubular shape is obtained, thus explaining the extremely poor resistance obtained. As the 5 mm–300 rpm is taken into account, no extrusion could be obtained, due to the effect of the "too cold" material and the increased extrusion ratio. It is worth noticing that, although the last aspect is usually beneficial to the chip consolidation, the required load in these conditions increases. As already specified, the machine load limit was reached (see again Fig. 4b) and the process could not be completed. As the tool rotation increases to 500 rpm, a sound extrusion is found for the 5 mm rod. On the contrary, a small flow defect is found in the 7 mm specimen. Finally, using tool rotation equal to 700 rpm, sound extrusion parts are produced with both the extrusion ratio values considered in this study. This rotation value correspond to the maximum resistance for the 7 mm rods. On the other hand, looking at the 5 mm case studies, the mechanical performance obtained with 700 rpm is lower than the one obtained with 500 rpm. Although both the latter specimens show a consolidated cross area, the etched cross section of the 700 rpm case study shows a microstructure of the outer area similar to the one obtained in FSW when the so called swirl defect takes place due to the turbulent material flow close to the backplate [15]. In the FSE process, the lateral surface of the extrusion channel acts as a break for the rotating material thus inducing the swirl defect in case of large particle velocity. In order to better analyze the occurring material flow, micro observations have been carried out on a light microscope (Fig. 6).

The microstructure of the cross section of the joint clearly highlights the presence of two distinct areas. The outer grain is heavily deformed and a helical clockwise flow is visible, going from the rod periphery to its center. On the contrary, in the

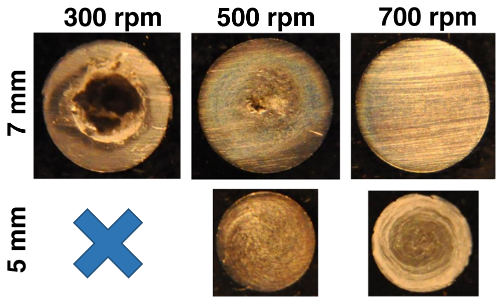

**Fig. 5** Macro images of the considered case studies

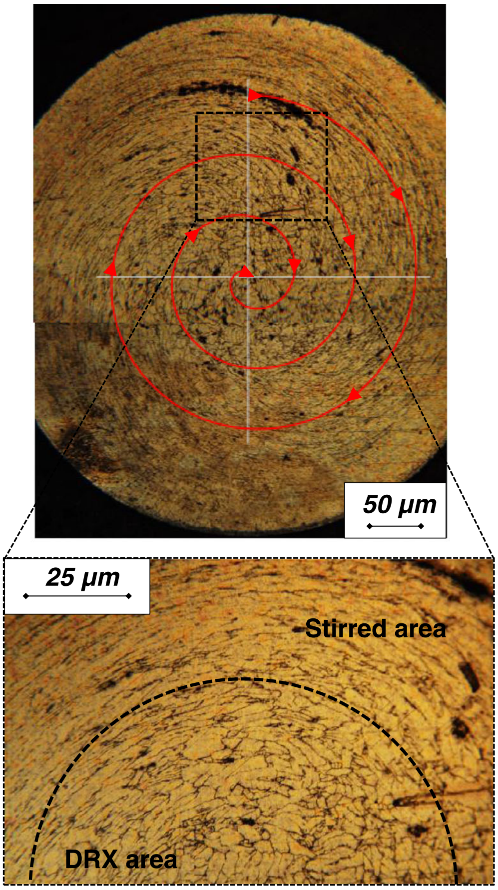

**Fig. 6** Etched cross section of the 5 mm–500 rpm case study

central area of the joint, a recrystallized grain is found. Based on the above observation and on the information acquired through the macro analysis (see Fig. 5), it can be assumed that, during the process, a 3D material flow is generated due to the combination of: (i) material rotation due to the tool action; (ii) centripetal flow enhanced by the conical shape of the base of the tool; (iii) vertical flow along the extrusion channel. In this way, only when the proper combination of these three distinct material flows is obtained the mechanical properties of the joint are maximized.

Finally, microhardness measurements have been performed along the diameter of the extruded rods. Figure 7 shows the microhardness profiles, measured along a diameter, for the 7 mm rods extruded with tool rotation of 500 and 700 rpm. The obtained HV values are consistent with the ones obtained in FSW of AZ31magnesium alloy [16].

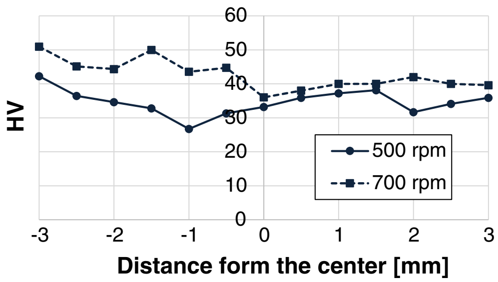

**Fig. 7** Microhardness profiles along a diameter for the 7 mm rods extruded with tool rotation equal to 500 and 700 rpm

The 7 mm–500 rpm case study, characterized by a flow defect in the central area of the cross section, shows smaller microhardness values with respect to the 7 mm–700 rpm one. The material around this area has not been sufficiently consolidated and the local lower mechanical properties have an impact on the tensile strength of the extruded component as shown in Fig. 3.

## Conclusions

In the paper, the results of an experimental study on the recyclability of AZ31 magnesium alloy by Friction Stir Extrusion is presented. The obtained results shows that the process is feasible and mechanical resistance of about 80 % of the base material can be obtained. Tool rotation is key process parameter for the effectiveness of the process. With low rotation values, corresponding to low heat input, no extrusion is obtained. On the contrary, the combination of large rotation values and high strain can result in swirl defects compromising the specimen mechanical properties. A complex 3D helical material flow is generated by the tool action, and distinct areas are observed in the cross section of the extruded parts, with heavily stretched grain in the periphery and recrystallized grain in the center.

Future works include a detailed analysis of the material flow for a more effective design of the process geometrical and technological parameters.

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