

## MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SOLID STATE RECYCLED MG ALLOY CHIPS

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Keyword: Mg alloy chip, Recycling, Hot-extrusion, Forging, Formability

### Abstract

Recycling of Mg alloy chips generated in machining processes such as turning and sawing through a melting process is difficult because the chips are very fine and can burn easily during heating. In this study, two machined Mg alloy chips were solid-state-recycled into a bar by hot pressing and hot extrusion, and the mechanical properties of the recycled chips were examined. The recycled AZ91 and AZX911 alloys showed a fine microstructure with a grain size of less than 10  $\mu\text{m}$ . The compressed yield stresses at room temperature were 208 and 210 MPa for the recycled AZ91 and AZX911, respectively, which are higher than those for non-recycled samples. A backward extrusion test revealed that the recycled AZ91 and AZX911 alloys have good forgeability at temperatures above 573 K and slightly higher hardness than non-recycled samples. Therefore, solid-state-recycled Mg alloys have good formability for forging at elevated temperatures with good mechanical properties and have potential for use as forging material

### Introduction

Mg alloy is the lightest among commercial structural materials and used in the housing of computers, cell phones, and consumer goods. Moreover, the application of Mg alloy to automotive parts reduces the weight of the car and helps improve fuel efficiency and CO<sub>2</sub> emission reduction. The use of Mg alloy in automotive markets has increased in the last ten years. The increase in Mg alloy components has also caused an increase in their waste; thus, the recycling of Mg alloys has become more important. The production of a kilogram of Mg from raw materials consumes 35 kWh. In contrast, a kilogram of refined recycled metal requires less than 3 kWh [1], implying that Mg alloy has excellent recyclability. Most recycling is carried out by a melting process. In general, the waste of Mg alloys is categorized into several classes; among them, clean scraps without impurities or oil, such as casting scraps, are easily recycled by melting. However, recycling of fine chips generated during machining by melting is difficult. Upon heating, Mg alloy chips are easily oxidized and burned even in a cover gas before melting. Increasing the Mg alloy parts causes an increase in Mg chips; thus, there is a demand for a suitable process for recycling of Mg chips. Studies have reported [2,3] that Mg alloy chips can be solid-state-recycled by undergoing hot extrusion into a bar, which then results in a fine structure with high strength. The solid-state recycling process is useful for enhancing the mechanical properties of Mg alloys. However, little has been reported on the formability of solid-state-recycled Mg alloy. This study was aimed at clarifying the microstructure and mechanical properties of solid-state-recycled Mg alloy chips and evaluating the formability of recycled Mg alloy for forging materials.

### Experimental Procedure

In this study, AZ91 and AZX911 alloys were used; their chemical compositions are listed in Table 1. AZX911 alloy contains Ca, which improves creep resistance and flammability. The chips were produced by dry cutting non-recycled AZ91 and AZX911 bars prepared by casting and hot extrusion. The chips had a fibrous shape with a length of about 1 mm, width of 100  $\mu\text{m}$ , and thickness of 20  $\mu\text{m}$ , as shown in Fig.1. The chips were hot-pressed at a temperature of 573 K under a pressure of 350 MPa into a preform with a diameter of 70 mm and height of 50 mm; the preform was then hot-extruded at 623 K into a bar with a diameter of 18 mm. The higher the extrusion temperature, the lower is the extrusion force; however, extrusion above 673 K causes oxidation or partial melting of samples. Hot pressing and hot extrusion were carried out in air. The extrusion ratio was 1:15 because the consolidated chips with a low extrusion ratio below 1:10 showed no sound deformation during upsetting at elevated temperature. The bar prepared by the hot extrusion of chips is shown in Fig.2.

Table 1. Chemical composition of AZ91 and AZX911(wt%)

alloy	Al	Zn	Mn	Ca
AZ91	8.7	0.81	0.21	-
AZX911	8.8	0.84	0.17	1

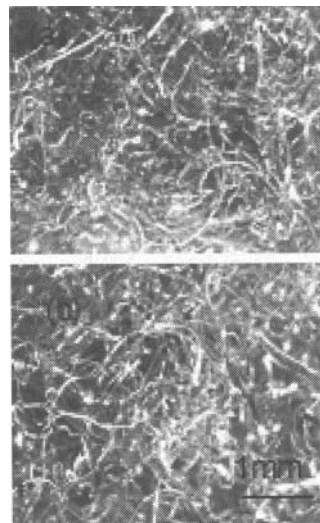


Figure 1 Mg alloy chips: (a) AZ91 and (b)AZX911

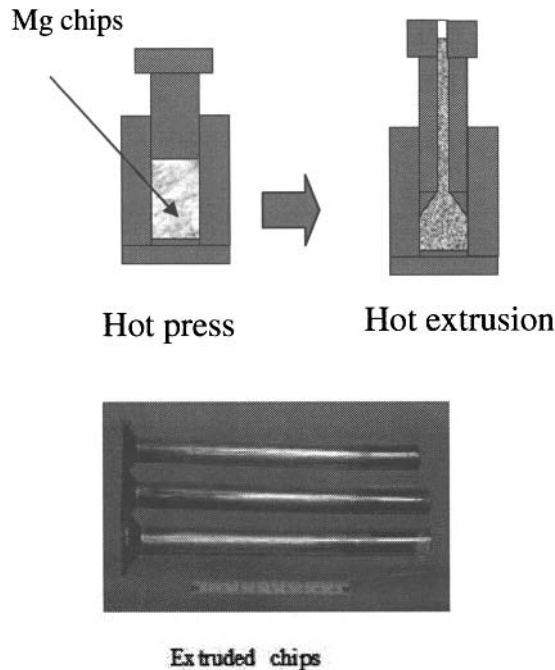


Figure 2 Consolidation process and extruded Mg alloy chips

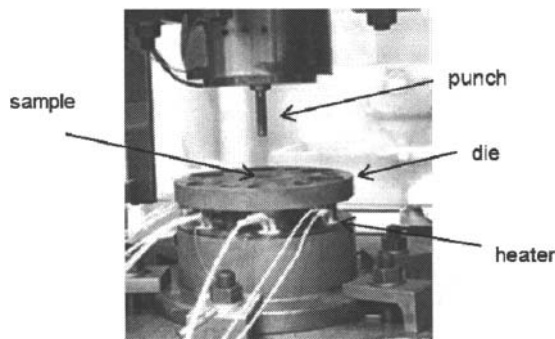


Figure 3 Die set for the backward extrusion

The microstructure of the obtained bar was examined by optical microscopy and X-ray diffractometry. The mechanical properties at room temperature were examined by a compression test. The forgeability was evaluated by a backward extrusion test. The test piece was machined from extruded bars into a shape with a diameter of 16 mm and height of 10 mm. Figure 3 shows a die set for the backward extrusion test, installed on a crank press machine. The stroke number and maximum force of the crank press machine were 86 spm and 100 tf, respectively. The inner diameter of the die is 16 mm. Molybdenum disulfide was used as the lubricant. A test piece prepared by turning the hot-extruded bar was inserted into a die heated by rod-type heaters at the desired

temperature. After maintaining these conditions for 10 min, the test piece was backward-extruded by a punch into a cup shape. The punch has a diameter of 13.4 mm. The extruded sample has an area reduction of 70%, which corresponds to an equivalent strain of about 2. Most of forged parts are subjected to deformation with an equivalent strain of 2, and therefore the area reduction of 70% is suitable for evaluating forgeability.

## Results and discussion

### Microstructure

Figure 4 shows the optical microstructure of AZ91 and AZX911 chips recycled by hot extrusion. Individual machined chips were not observed, and a fully dense structure was obtained for both samples. The density of AZ91 as measured by Archimedes' method was  $1.81 \text{ g/cm}^3$ , which is nearly the same as the theoretical value. The extruded AZ91 and AZX911 chips were composed of fine equiaxed grains. The average grain sizes were 10 and  $7 \mu\text{m}$  for AZ91 and AZX911, respectively. The non-recycled AZ91 and AZX911 alloys showed grain sizes of 100 and  $50 \mu\text{m}$ , respectively, as shown in Fig.5. A fine microstructure was obtained by solid-state recycling. This is owing to the dynamic recrystallization induced by hot extrusion. In the recycled samples, AZX911 showed a finer microstructure than AZ91. The addition of Ca was effective in preventing grain growth during recrystallization. This may be because Ca addition causes the formation of  $\text{A}_2\text{Ca}$  or an intermetallic compound containing Ca, which acts as a pin to inhibit grain growth.

Figure 6 shows the X-ray diffraction patterns taken from the cross-sectional area of the bar recycled by the hot extrusion of AZ91 and AZX911 chips. AZ91 alloy is mostly composed of hcp Mg. The peaks corresponding to  $\text{Mg}_{17}\text{Al}_{12}$  were not observed,

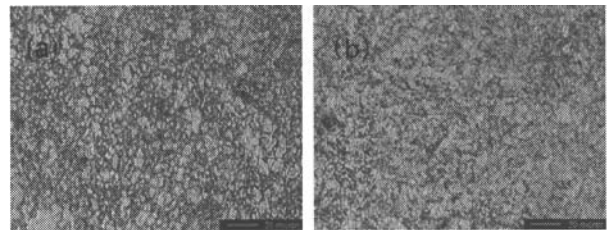


Figure 4 Optical microstructure of hot extruded AZ91 (a) and AZX911(b) alloy chips

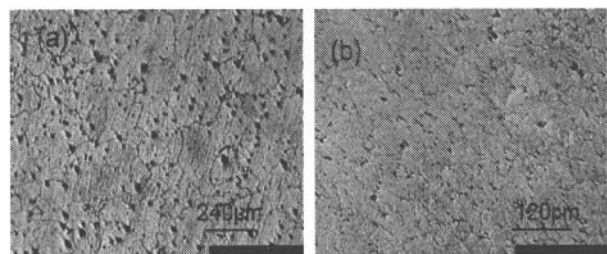


Figure 5 Optical microstructure of non-recycled AZ91(a) and AZX911 alloy(b)

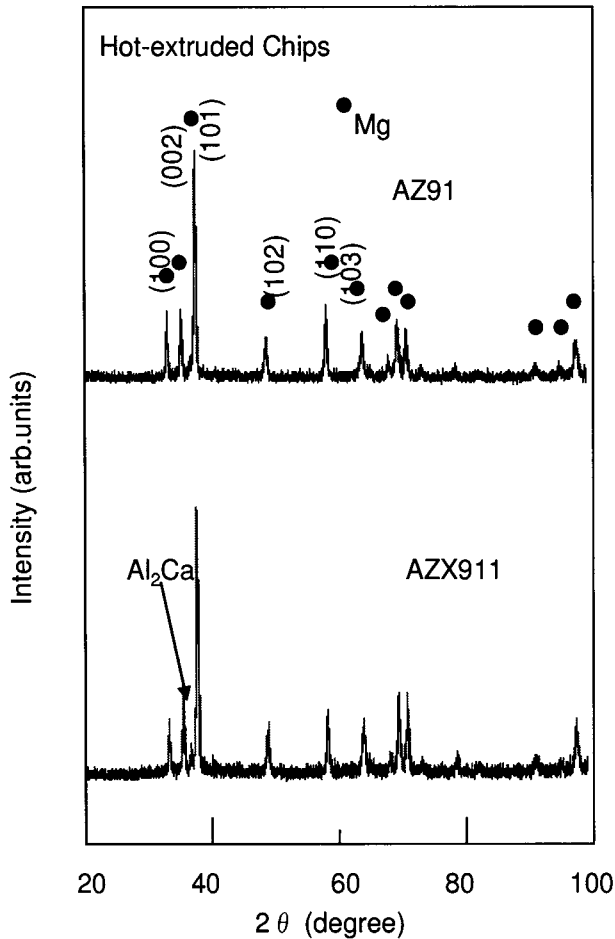


Figure 6 X-ray diffraction patterns of hot extruded AZ91 and AZX911 alloy chips

which might be because  $Mg_{17}Al_{12}$  dissolved into Mg during the hot extrusion process. In the X-ray diffraction pattern of AZX911, peaks corresponding to  $Al_2Ca$  were observed along with those corresponding to hcp Mg. The X-ray diffraction patterns of both samples were similar to those of cast samples. This means that the samples recycled by hot extrusion of the chips have no texture. In general, a Mg wrought alloy produced by hot extrusion shows an oriented structure with a basal plane of hcp Mg that is parallel to the extrusion direction. The sample without texture should have a good formability.

#### Mechanical Properties

Figure 7 shows the compressive mechanical properties of solid-state-recycled AZ91 and AZX911 alloys at room temperature. For comparison, the compressive mechanical properties of non-recycled AZ91 and AZX911 are shown in Fig.8. The fracture stress ( $\sigma_f$ ) and yield stress ( $\sigma_{0.2}$ ) of recycled AZ91 alloy are 334 and 208 MPa, respectively. These values are higher than those of non-recycled AZ91 alloys. For AZX911, a similar tendency was observed. The recycled AZX911 also showed a higher strength,

with  $\sigma_f$  of 327 MPa and  $\sigma_{0.2}$  of 210 MPa, compared to a non-recycled sample. It is notable that Mg alloys produced by solid-state recycling have improved mechanical properties. This is owing to the grain refinement by dynamic recrystallization. The fracture strains were 0.47 and 0.38 for recycled AZ91 and AZX911, respectively, which are also higher than those for non-recycled AZ91 and AZX911. AZX911 showed a slightly lower fracture strain than AZ91, although AZX911 showed a finer microstructure than AZ91. This is caused by precipitation of intermetallic compounds such as  $Al_2Ca$ . However, the fracture strain of solid-state-recycled Mg alloy is higher than those of non-recycled materials, suggesting that ductility is also improved by solid-state recycling.

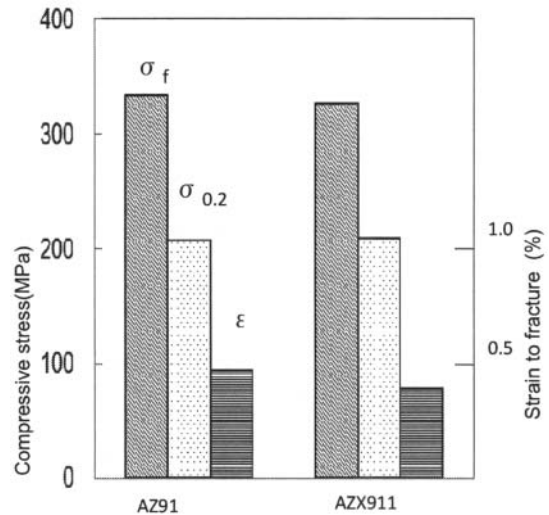


Figure 7 Compressive mechanical properties of solid state recycled AZ91 and AZX911 alloys

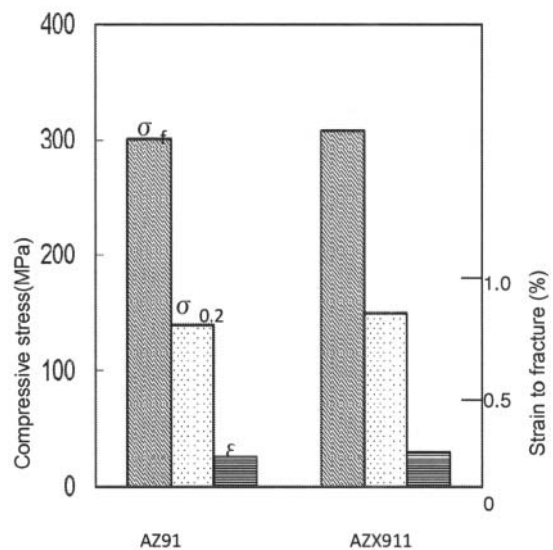


Figure 8 Compressive mechanical properties of solid state non-recycled AZ91 and AZX911 alloys

## Forgeability

In order to evaluate the forgeability, the solid-state-recycled AZ91 and AZX911 alloys were backward-extruded into a cup shape at elevated temperatures. Figure 9 shows the outer surface of recycled and non-recycled AZ91 alloys after backward extrusion in the temperature range of 473–573 K. At 473 K, several cracks were observed on the surface of recycled AZ91, and the top of the cup was broken. The fracture surface was declined at 45° to the extruded direction, implying the occurrence of shear fracture. At 573 K, the recycled AZ91 showed no cracks on the surface, but a

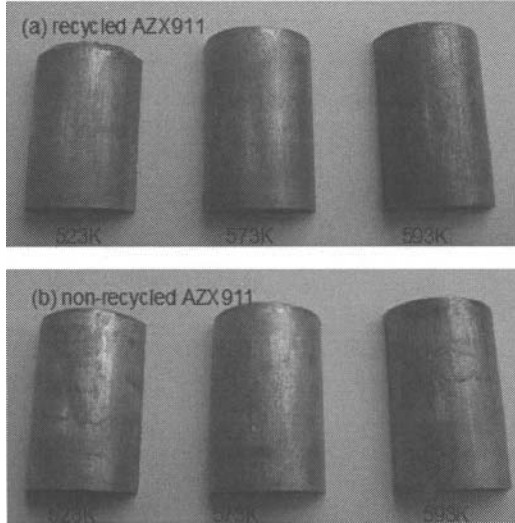


Figure 9 External appearance of recycled and non-recycled AZ91 alloy after backward extrusion at various temperatures

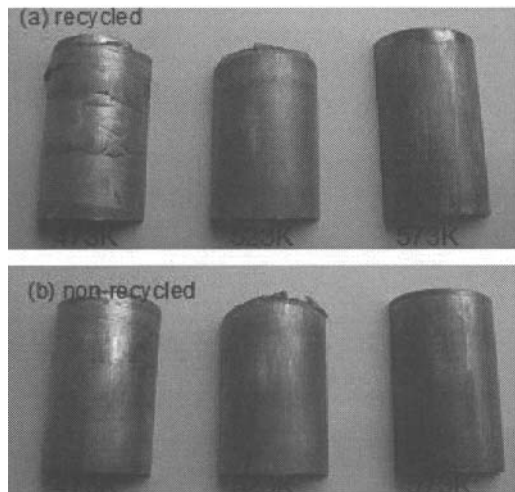


Figure 10 External appearance of recycled and non-recycled AZX911 alloy after backward extrusion at various temperatures

small fracture occurred on the top part. For the non-recycled AZ91, a similar tendency was observed. A further increase in test temperature improved the formability. However, a backward extrusion test at above 573 K caused partial melting owing to the generation of heat by working. Figure 10 shows the results of backward extrusion at 473–593 K for the recycled and non-recycled AZX911 alloys. For recycled AZX911, cracks were observed at 473 K and they decreased with increasing extrusion temperature. The cup extruded at 593 K shows no cracks on the surface. In contrast, non-recycled AZX911 showed cracks on the surface of the cup backward-extruded at 593 K. The forgeability of solid-state-recycled AZ91 and AZX911 was comparable to that of the non-recycled alloys. However, the solid-state-recycled Mg alloy has enough workability for forging at elevated temperatures. The generation of cracks may be caused by the temperature difference between the punch and the samples; this can be controlled by changing the forging condition. The punch geometry also influences the deformation behavior. An appropriate geometry of the punch should improve the forgeability. Furthermore, the deformation rate also influences the formability. In particular, Mg alloys show a strong strain rate dependence. The higher the strain rates, the lower is the elongation. In the present backward extrusion test, the punch speed was estimated to be 0.22 m/s, which indicates a high deformation rate. A lower deformation would make it possible to achieve a sound deformation.

Figure 11 shows an optical microstructure of the recycled AZ91 alloy after the backward extrusion test at 573 K. A grain flow is clearly found in the wall part and bend part. This metal flow introduced by plastic deformation helps enhance the mechanical properties, in which the structure is not obtained by casting. The backward-extruded AZ91 shows a fine microstructure with an average grain size of 5  $\mu\text{m}$ , which is finer than the recycled bar. Further grain refinement occurs by dynamic recrystallization during backward extrusion. The backward-extruded AZX911 shows a similar fibrous and fine structure with an average grain size of less than 5  $\mu\text{m}$ .

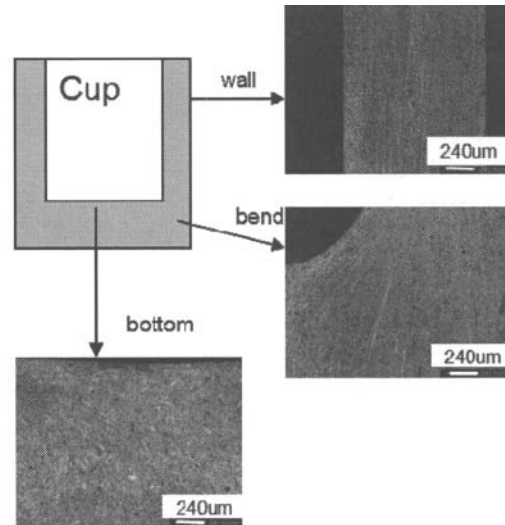


Figure 11 Optical microstructure of backward extruded AZ91 alloy at 573K prepared by hot extrusion of the chip

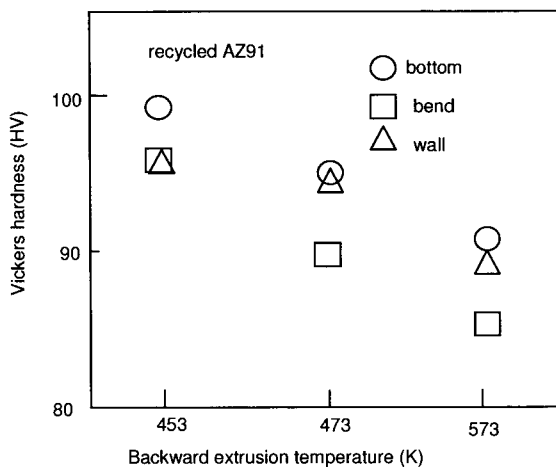


Figure 12 Change in the Vickers hardness of the recycled AZ91 alloy as a function of back ward extrusion temperature

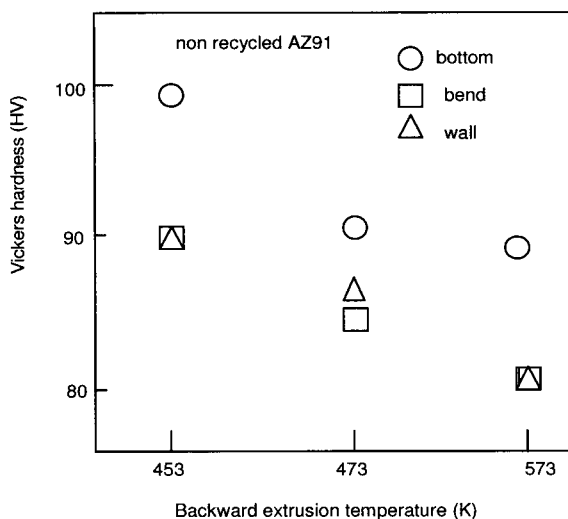


Figure 13 Change in the Vickers hardness of non-recycled AZ91 alloy as a function of back ward extrusion temperature

Figure 12 shows the change in Vickers hardness of the bottom, bend, and wall parts of the backward-extruded AZ91 alloy prepared by solid-state recycling as a function of backward extrusion temperature. Hardness was found to decrease with increasing extrusion temperature. The cup deformed at 573 K showed a hardness of 85–90 HV. The hardness of the AZ91 alloy prepared by backward extrusion of the non-recycled sample is shown in Fig.13. A similar change was observed, and the hardness of the recycled AZ91 was slightly higher than that of non-recycled AZ91. For AZX911, a similar tendency was observed. This suggests that the mechanical properties of the sample forged using the solid-state-recycled Mg alloy are superior to those of the sample forged using the non-recycled Mg alloy. Finally, the solid-state-recycled Mg alloy

showed good forgeability, and the forged sample showed good mechanical properties. The AZ91 and AZX911 alloys used in this study are generally casting alloys with poor plastic formability. Thus, it is notable that the solid-state-recycled AZ91 and AZX911 alloys showed enough formability for forging, making it possible to use solid-state-recycled Mg alloy as forging materials. This is also caused by grain refinement by dynamic recrystallization. Furthermore, the recycling process investigated here was carried out at temperatures below 673 K without melting. This means that the energy consumption of solid-state recycling is less than that of conventional melting methods. In addition, the flux or cover gas used in the melting process is not necessary for solid-state recycling. Therefore, this is an environmentally friendly recycling process. In this study, dry processed Mg alloy chips were used. In most cases, machining of Mg alloys is carried out using machine oil, and thus, the Mg alloy chips are coated with oil. For solid-state recycling, deoiling treatment is needed because the coated oil prevents the connection of chips. We previously reported [4] that superheated steam is useful for cleaning oily chips; solid-state recycling with superheated steam treatment is under investigation.

## Conclusion

Machined AZ91 and AZX911 alloy chips were solid-state-recycled into bars using hot extrusion. The obtained bars showed a fine microstructure with a grain size of less than 10  $\mu\text{m}$  and higher strength than non-recycled materials. A backward extrusion test revealed that the solid-state-recycled AZ91 and AZX911 were forged into a cup without cracks at 573 and 593 K, respectively. The forged sample prepared by solid-state recycling showed a favorable grain flow and higher strength than the non-recycled samples. Therefore, we can conclude that the solid-state recycling process is useful for application to Mg alloy chips and that the materials recycled by this process can be used as a forging billet.

## Acknowledgement

This work was supported by New Energy and Industrial and Technology Development Organization (NEDO) of Japan.

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