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Consolidation behavior of Mg–10Gd–2Y–0.5Zr chips during solid-state recycling

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ABSTRACT

Unlike that of sintering of fine powders, chips are consolidated by hot deformation in the solid-state recycling. In this work, conventional extrusion (CE) and cyclic extrusion compression (CEC) are used to investigate single and multi-passes shear deformation on the consolidation of chips during solid-state recycling. The results show that utilization of Mg chips with smaller specific surface area (i.e. coarser powder) contributes to easier solid-state bonding because of the decrease in specific surface area which promotes suppression of oxide contamination in the recycled specimens. Enhanced consolidation of chips is not only ascribed to the physical mechanisms caused by plastic deformation, but also to atom diffusion between chips triggered by shear plastic deformation at elevated temperatures. Multi-passes shear deformation breaks the oxide films easier into small particles which are dispersed within the grains or at grain boundaries. It is postulated that the shear deformation of chips is responsible for the better consolidation of chips by high-temperature deformation.

1. Introduction

Magnesium alloys currently as the lightest metallic structure materials are gaining increasing importance for applications including aerospace, automotive, materials handling, and portable electronic appliances [1–6]. With the increasing of magnesium alloys in quality, it is very important to recycle these materials to ensure the sustainable development of society. In order to increase the applicability of Mg alloys, it is necessary not only to attain excellent properties, but also to develop low-cost methods to efficiently reclaim them from scrap or machined chips. Several recycling processes, such as re-melting, electro refining in molten salt and vacuum distillation, have been proposed and some of which have been carried out [7].

Recently, solid-state recycling by hot extrusion has been proposed as an advanced recycling method for machined chips because of its relatively low cost, and environmental benefit [7–9]. The Mg alloys recycled by this means showed relatively higher strength due to the grain refinement and uniform dispersion of oxide contaminant compared with those extruded specimens from the original ingot [10–15]. Besides, with the development of hydrostatic technology, the solid-state recycling is also popular because the huge specimens can be directly extruded at high temperature and the size of the recycled specimens is increased.

In previous studies, on the mechanical properties of the AZ31, AZ91, AZ80 and ZK60 magnesium alloys produced by solid-state recycling have been investigated [11–15]. Up to now, enhanced compaction of chips during solid-state recycling has not been studied. Similar to powder metallurgy technology, this recycling method includes chips comminuting, cold pre-compaction, sintering, and subsequently hot deformation [16–19]. In the powder sintering, the smaller particle size, the easier the consolidation. The finer size of the transition metal powders should, therefore, be favored as it will allow a faster rate of formation of the desired compound [20]. It is generally accepted that diffusion bonding plays a significant role in the consolidation of fine powders during sintering [21,22]. The growth of intermetallics is accelerated by the use of finer powders [23]. The coarser metal particles, the longer the time period required to carry out the diffusion to form the intermetallic compound [20]. However, in the solid-state recycling means, unlike that of sintering of fine powders, chips are consolidated by hot deformation. Furthermore, it is not feasible for machined chips, as fine powders from nanometer to micrometer size, to be recycled by simple sintering in vacuum atmosphere due to high cost involved. Thus, in order to develop the solid-state recycling technology, it is necessary to understand consolidation behavior of the chips.

In this work, Mg–10Gd–2Y–0.5Zr alloy chips were recycled by single and multi-passes extrusion at different temperatures,

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and enhanced compaction of chips during solid-state recycling was studied. This will also help us to understand consolidation of powders or particles during hot deformation and to choose the appropriate deformation means in order to obtain the better properties of consolidated materials.

2. Experimental procedures

2.1. Pre-compaction of machined chips

The material used in this study was a GW102K (Mg–9.95 wt%Gd–2.3 wt%Y–0.46 wt%Zr) magnesium alloy. Chips were prepared by machining an original ingot (which was previously solution treated at 490 °C for 8 h) in a lathe. Chips were firstly converted into low-density blocks by pre-compaction method before deformation. Pre-compaction was conducted as follows: the chips were placed in a cylindrical container with a diameter of 29.5 mm, and then compressed at 300 °C with a pressure of 200 MPa in air.

2.2. One-pass and multi-passes extrusion

After chips were converted in blocks by pre-compaction technology, these blocks were put in a cylindrical container with a diameter of 40 mm, and then recycled by one-pass conventional extrusion (CE) with ratio of 2.5:1, 10:1 and 20:1 at different temperatures.

In the present study, a cyclic extrusion compression (CEC) process is applied to investigate multi-passes extrusion on consolidation behavior of chips during solid-state recycling. CEC die used in this investigation and its operation procedure were described elsewhere [2,24,25]. The CEC processing was carried out by pushing a specimen from one cylindrical chamber with a diameter *D*, into the second chamber with the same dimensions, through a die with smaller diameter *d*. For the final extrusion, the opposite Ram was removed. In this study, *D* and *d* are 30 mm and 20 mm, respectively. The number of extrusion passes was defined as the number of the specimen passing through the die. At the final pass, one Ram was removed so that the other Ram could extrude the specimen in a rod shape with 20 mm in diameter.

2.3. Microstructural analysis and mechanical properties test

The flat tensile specimens with a gauge section of 10 mm × 3 mm × 1.5 mm were cut from the recycled samples using an electric-spark wire-cutting machine. Tensile tests were carried out with a strain rate of 5×10^{-4}/s at room temperature using a Zwick T1-Fr020TN.A50. Vickers hardness testing was taken using 49 N load and holding time of 15 s. The densities of the recycled specimens were determined by Archimedes' method, and the relative-density of a block was obtained based on the density of the block and the original ingot. Several deep cracks due to the interfaces de-cohesion were measured by OM and SEM, respectively. Deformation mechanisms and microstructure were analyzed using SEM (JEOL-6460) and TEM (Philips-CM20,TECNAI G2).

3. Results

3.1. The size distribution of the comminuted chips

Specific surface area is a material property of solids which measures the total surface area per unit of mass. Specific surface area is one of the important characters of powders. In vacuum sintering, the smaller the powder size is, the larger specific surface area of the powder is, and the easier consolidation of the powder is. Fig. 1(a) shows the appearance of the comminuted chips. In order to semi-quantitatively describe the specific surface area of the chips, the size distribution of the chips is firstly analyzed.

Chips were prepared by machining an ingot in a lathe. To avoid burning in high-speed machining of magnesium alloys, chips with smaller size are made. For the same magnesium alloy, the width or the thickness of chips almost has the same size distribution. For the GW102K magnesium alloy in this study, chips were prepared with average dimensions of 3 mm (width) × 80 μm (thickness). Size distribution of the length for the comminuted chips is shown in Fig. 1(b). Statistical calculation on the length size distribution is made based on 100 pieces of comminuted chips collected. The horizontal axis is the ratio of length to width. For example, as the size of the length for chips is distributed from 6 mm to 9 mm, the value of length to width is correspondingly 2–3. The maximum size of length was distributed from 30 mm to 33 mm, and ~70% of the chips were from 3 mm to 12 mm. Based on the mathematic theory of statistics, 9.6 mm, as the mean value of length, was obtain from 100 pieces of comminuted chips.

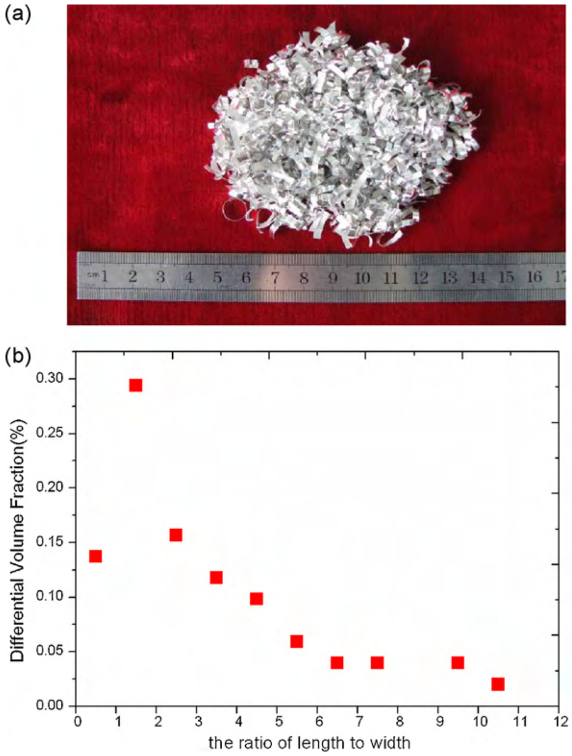


Fig. 1. The comminuted chips: (a) the appearance and (b) the size distribution.

Total number of the comminuted chips per gramme (*N*) and the specific surface area per gramme (S_w) can be written as:

$$N \times L \times W \times T = \frac{1}{\rho} \tag{1}$$

$$S_w = 2N \times (L \times W + L \times T + W \times T) \tag{2}$$

where *N* is total number of the comminuted chips per gramme, *L* is length in mm, *W* is width in mm, *T* is thickness in mm, ρ is density in g/cm^3, S_w is the specific surface area per gramme in cm^2/g.

For the Mg–10Gd–2Y–0.5Zr alloy, $\rho = 1.91 \times 10^3$ kg/m^3. According to the chips with the average geometric dimensions of 9.6 mm (length) × 3 mm (width) × 80 μm (thickness), $S_w = 136.5$ cm^2/g, as the specific surface area per gramme for the comminuted chips, is obtained.

However, for powders from nanometer to micrometer size, $S_w = 10^6$–10^7 cm^2/g, which is much too higher than that of the chips. Correspondingly, machined chips cannot be recycled by simple sintering technology, but by high-temperature deformation.

3.2. Influence of temperature on consolidation of machined chips

In order to investigate consolidation behavior of chips during solid-state recycling, the blocks from machined chips were extruded with extrusion ratio of 10:1 at different temperatures. The diameter of extruded bars was 12 mm. Fig. 2 shows the recycled specimens by CE at 200 °C, 300 °C, 400 °C, 450 °C and 500 °C, respectively. At 200 °C and 300 °C, the blocks from chips was easily destroyed due to shear stress, which was consistent with there being periodic mechanical (shear) instability in the compact as a whole during extrusion. In order to achieve good consolidation, the extrusion *T* of 450 °C to 500 °C was also done. It was shown that

Fig. 2. The recycled specimens extruded from machined chips at 200 °C, 300 °C, 400 °C, 450 °C and 500 °C in sequence.

an increase in the temperature could eliminate the macroscopic cracks. The smooth specimens were obtained from chips by stable shear stress during CE deformation at 450–500 °C.

Fig. 3 shows hardness distribution of the recycled specimens at 200 °C and 400 °C in the radial direction. At 200 °C, higher hardness in the center was obtained than at the edge. However, at 400 °C, hardness values in the center were nearly same to those at the edge. This result is due to limited slip systems and poor cohesion level of chips at 200 °C, which will be introduced in the discussion section. Besides, the average hardness values at 200 °C were higher than those at 400 °C, which could be expected for two reasons:(1) plastic deformation at 200 °C could to a great extent improve physical contact of chips, and (2) deformation at 400 °C induced the dynamic recovery or recrystallization which could weaken the hardness.

Fig. 4 shows SEM images of the pre-compaction from chips and of the recycled specimens extruded from chips at 200 °C. Chips in the pre-compaction demonstrated a horizontal and lamellar distribution microstructure. However, after extruded at 200 °C, chips were turned to 90° by shear stress. The relative-density of the recycled specimens at 200 °C was higher than that of the pre-compaction. It meant that, even at low temperature, the shear forces during deformation were sufficient to cause local chips rearrangements, deformation and comminuting of individual chips. In result, a substantial degree of conformation between local chips could cause to achieve significant increase in density. There was little evidence of chips bonding manifested in either direct macroscopic observation (as shown in Fig. 2), microscopic examination (as shown in Fig. 4), or in resultant room temperature mechanical properties (<100 MPa). The implication is that there was sufficient contact between chips not to realize high level of diffusion bonding at 200 °C. This lack of integrity in the compact was ascribed to the limited slip systems and poor metallurgical bonding between chips at 200 °C.

When the deformation temperature was increased to 300 °C from 200 °C, cracks between chips almost vanished due to diffusion bonding as shown in Fig. 5. However, many triple cracks were relatively difficult to vanish due to the unattained interfaces contact between chips.

Microstructures and the tensile fractograph of the recycled specimens extruded from chips at 400 °C are also shown in Fig. 6. Although the fracture was relatively flat, dimples and tearing edges were nearly nothing, it is found that diffusion bonding of chips in the recycled specimens was mainly obtained, which resulted in the moderate tensile strength and elongation of the recycled specimens at room temperature (UTS – 277.14 MPa, Elongation – 1.9%), just as shown in Table 1.

Table 1
Room temperature mechanical properties of the recycled specimens at 400 °C.

Mechanical properties	Pre-compact	Conventional Ex.		6-passes CEC
		E2.25	E10	
Relatively density (%)	93	97	98	~100
HV	49.5	52.3	57.7	58.2
Elongation (%)	<0.2	0.83	1.9	13.18
The tensile strength (MPa)	<50	222.38	277.14	322.96

3.3. *Influence of extrusion ratio on consolidation of machined chips*

Fig. 7 shows the microstructures of the recycled specimens extruded from chips at 400 °C with extrusion ratios of 2.25:1 and 20:1. Compared with the microstructures in the recycled specimens extruded from chips with extrusion ration of 10:1 at 400 °C in Fig. 6, interfaces between chips could be observed with extrusion ratio of 2.25:1. When extrusion ratio was increased to 10:1 from 2.25:1, interfaces were partly dissolved due to diffusion bonding at high temperature. However, in the recycled specimens with extrusion ratio of 20:1 at 400 °C, interfaces between chips were almost dissolved and the residual pores were hardly remained. For magnesium alloys, more shear strains are caused with high ratio extrusion than with low ratio one, which could increase the diffusion bonding of chips. Besides, compared with extrusion ratio of 10:1, deformation with extrusion ratio of 20:1 caused more heat energy, which could be proved by the coarser grain size. Hence, shear strain and heat energy could cause the good bonding of chips at high temperature.

3.4. *Influence of multi-passes on consolidation of machined chips*

On the whole, provided that the chips are completely consolidated by hot deformation, the lower the temperature is, the higher the strength of the recycled specimens is. For one-pass extrusion, in order to improve consolidation of chips, the temperature or extrusion ratio is increased, which could weak the strength or decrease the size of the recycled specimens. Thus, effects of multi-passes on consolidation of machined chips at 400 °C were investigated. Table 1 shows room temperature mechanical properties of the recycled specimens at 400 °C. Multi-passes could obviously improve room temperature mechanical properties.

Fig. 8 shows SEM images of the recycled specimen by 6-passes CEC at 400 °C. The triple cracks due to the interfaces de-cohesion were vanished, and the residual pores were not observed. TEM micrographs of oxide contaminants in the recycled specimen are shown in Fig. 9. Oxide contaminants were distributed at the interfaces of chips in the specimen by one-pass extrusion. As CEC was carried out, multi-passes shear deformation breaks the oxide films easier into small particles which are dispersed within the grains or at grain boundaries. Besides, due to multi-pass shear deformation, the pores were also closed by high pressure easier. Hence, the ductility was restored to a high level after multi-passes CEC processing. The restoration of ductility mainly depended on the elimination of the interfaces between chips [2].

4. Discussion

4.1. *Physical mechanisms on the consolidation of chips*

Consolidation of Mg chips during solid-state recycling was introduced in the present study. High-temperature or multi-passes extrusion deformation could realize intimate interfaces contact between chips, accelerate the interface bonding of chips, and successfully make chips consolidation.

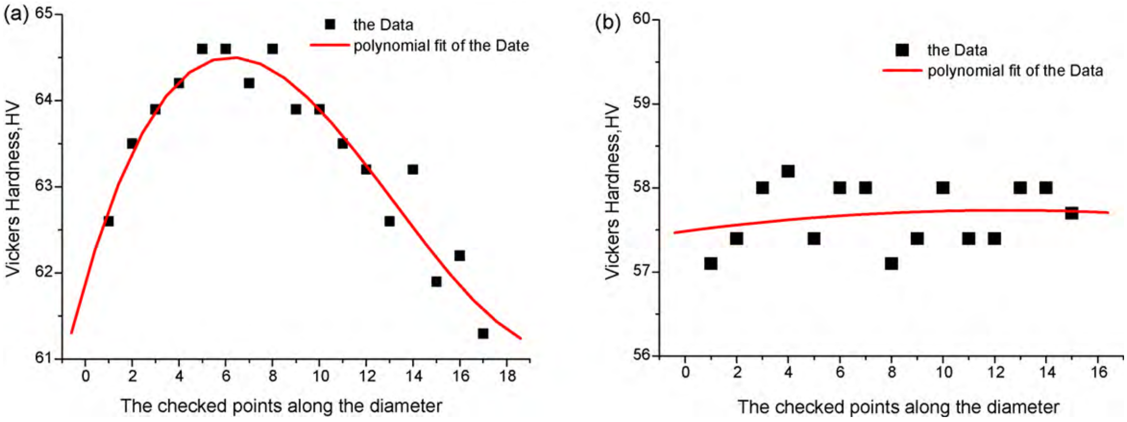


Fig. 3. Hardness distribution of the recycled specimens extruded from chips at: (a) 200 °C and (b) 400 °C in the radial direction.

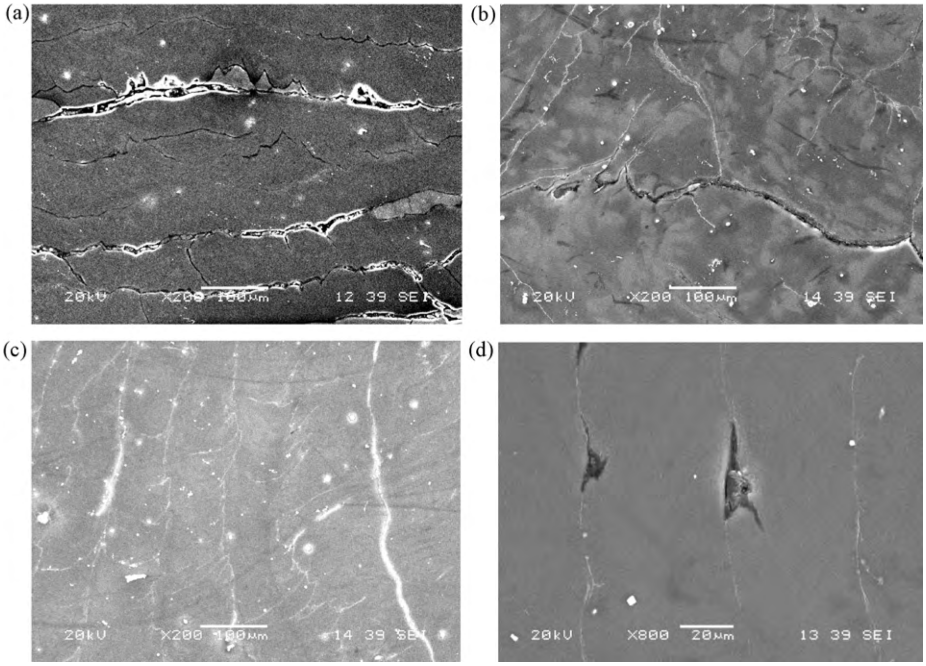


Fig. 4. SEM images of the pre-compaction from chips: (a) work press direction is vertical, (b) work press direction is horizontal, and of the recycled specimens extruded from chips at 200 °C: (c and d) the extrusion direction is vertical.

For the GW102K Mg alloy ingots, the lowest extrusion temperature was 350 °C [26]. When the temperature was low than 350 °C, the ingots were not successfully extruded or their surfaces were destroyed due to low plastic deformation ability. This phenomenon was consistent with the recycled alloy at different temperatures. Although, at low temperature, plastic deformation could to some extent increase the compaction, the blocks from chips were easily destroyed by shear stress. As the temperature was lower than

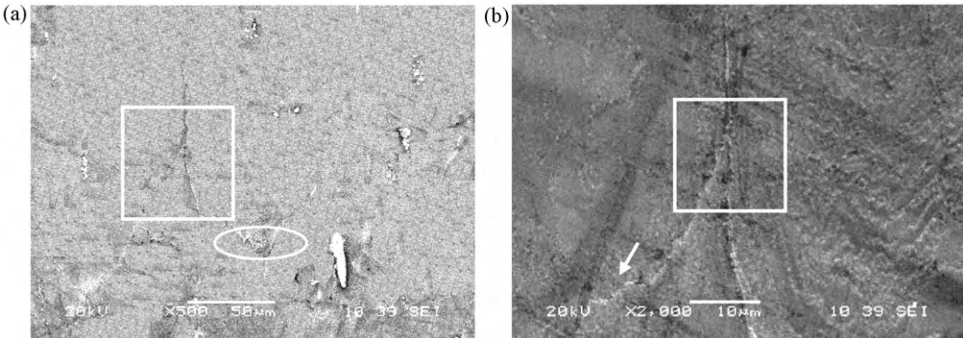


Fig. 5. SEM images of the recycled specimens extruded from chips with extrusion ratio of 10:1 at 300 °C: (a) low magnification and (b) high magnification image. The ellipse shows the triple cracks, the rectangle indicates the bonding of the triple cracks.

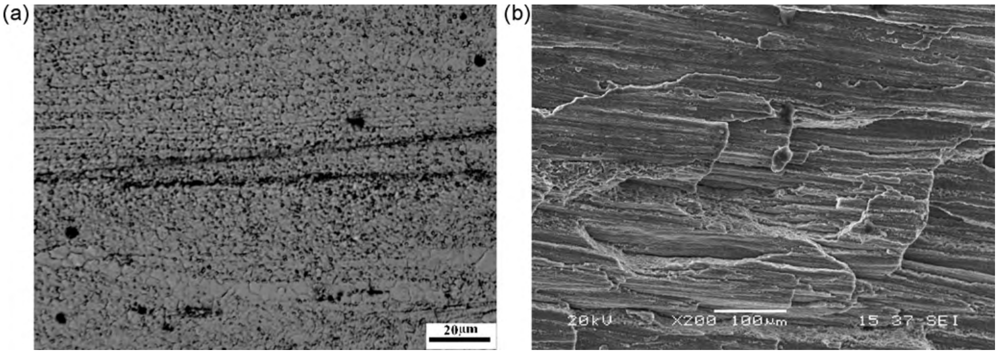


Fig. 6. (a) Microstructures and (b) the tensile fractograph of the recycled specimens extruded from chips with extrusion ratio of 10:1 at 400 °C, the extrusion direction is horizontal.

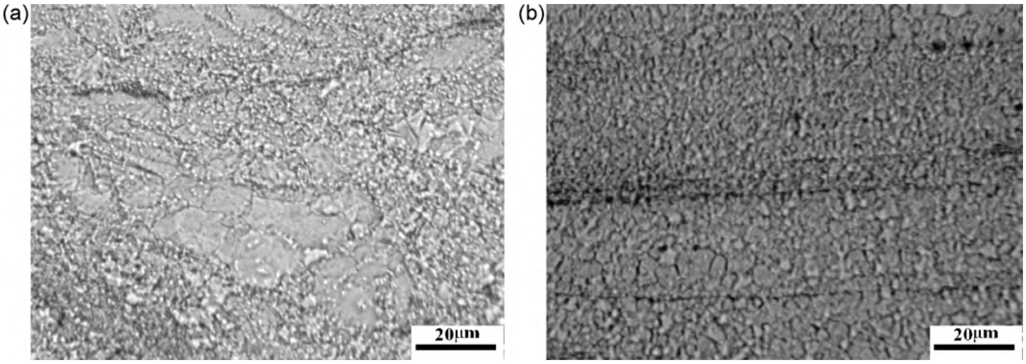


Fig. 7. Microstructures of the recycled specimens extruded from chips at 400 °C with: (a) extrusion ratio of 2.25:1 and (b) extrusion ratio of 20:1, the extrusion direction is horizontal.

225 °C, Mg alloys were only deformed by the basal plane slip $\left\{0001\right\}\left\langle 1\bar{1}20\right\rangle$ and the pyramidal plane twins $\left\{10\bar{1}2\right\}\left\langle 10\bar{1}1\right\rangle$ [27]. Correspondingly, Mg alloys chips had poor plastic deformation ability at 200 °C. Even before shear stress during deformation induced pyramidal plane $\left\{10\bar{1}2\right\}\left\langle 10\bar{1}1\right\rangle$ twins, shear deformation would cause the poor bonding interfaces or physical contact between chips to be torn out again in order to release the redundant stress. As a result, for the limited slip system at low temperature, more shear strain during the deformation would difficultly cause chips compaction. In the recycled specimen at 200 °C, more shear strain at the edge was induced than in the center, which more easily made crack happen at the edge. Correspondingly, hardness value in the center was higher than that at the edge, as shown in Fig. 3a. When the temperature was increased to 400 °C, besides the basal plane slip $\left\{0001\right\}\left\langle 1\bar{1}20\right\rangle$ and pyramidal plane twins $\{10\bar{1}2\}\left\langle 10\bar{1}1\right\rangle$, the pyramidal plane $\{10\bar{1}1\}$ and cylindrical plane $\{10\bar{1}0\}\left\langle 1\bar{1}20\right\rangle$ were also activated [27]. Due to the excellent plastic deformation ability of Mg alloys at 400 °C, high strain easily made chips compaction. On the other hand, more shear strain at the edge was easy to increase intimate interfaces contact between chips. In result, hardness value in the center was almost same as that at the edge in Fig. 3b.

4.2. Metallurgical mechanisms on the consolidation behavior of chips

Enhanced compaction of chips during solid-state recycling is not only ascribed to the physical mechanisms caused by plastic deformation, but to atom diffusion between chips triggered by severe plastic deformation at elevated temperatures. Physical mechanisms during the deformation have been discussed as above. For the powder from nanometer to micrometer size, $S_w = 10^6–10^7\,\text{cm}^2/\text{g}$, metallurgical bonding of particle is easily obtained due to surface diffusion mechanism during vacuum sintering. However, it is

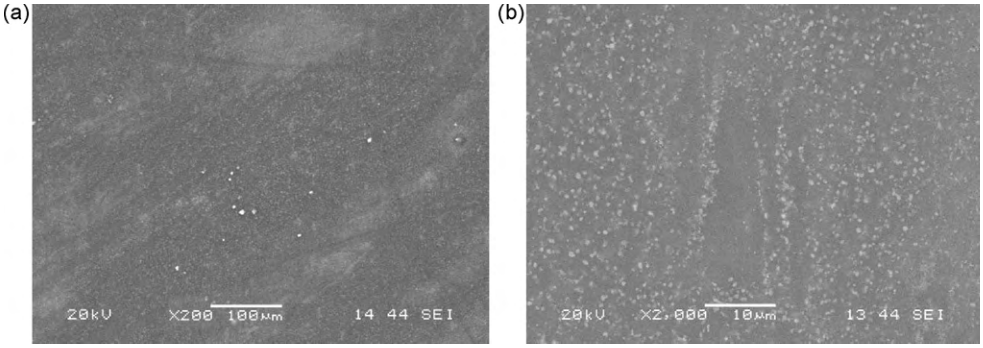
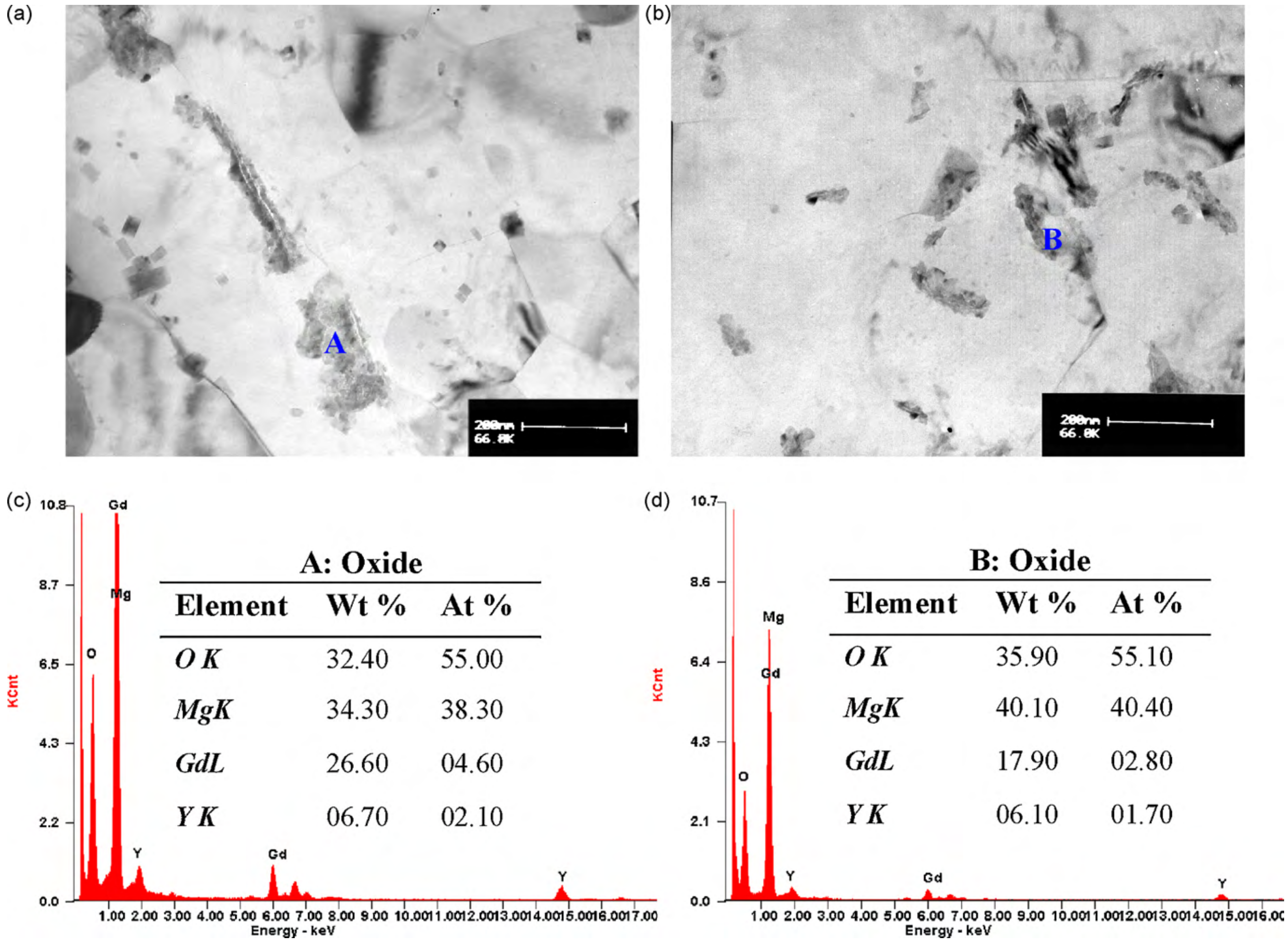


Fig. 8. SEM images of the recycled specimens by 6-passes CEC at 400 °C: (a) low magnification and (b) high magnification.



A: Oxide

Element	Wt %	At %
O K	32.40	55.00
MgK	34.30	38.30
GdL	26.60	04.60
Y K	06.70	02.10

B: Oxide

Element	Wt %	At %
O K	35.90	55.10
MgK	40.10	40.40
GdL	17.90	02.80
Y K	06.10	01.70

Fig. 9. TEM image of oxide contaminants (a) in the recycled specimens by CE at 400 °C, in the recycled specimens by 6-passes CEC at 400 °C, and (c and d) corresponding energy dispersive X-ray spectra of the points indicated in the image.

different that chips are metallurgically bonded during solid-state recycling from the powder bonding in vacuum sintering. An essential factor of solid-state recycling is a destruction of oxide layer by severe plastic deformation just as shown in Fig. 9. Thus, it is suggested that utilization of Mg powder with smaller specific surface area (i.e. coarser powder) can enhance solid-state bonding because of the decrease in specific surface area which suppress oxide contamination in the recycled specimen. Chino et al. [11] indicated that the mechanical properties of the specimen from cylindrical scraps were found to be almost the same as those of the rolled specimen from a virgin ingot. This is because the oxide contamination in the specimen from cylindrical scraps was lower compared with that from the chips. Therefore, unlike that of sintering of fine powder, coarse chips are easier for metallurgical bonding during the recycling.

Metallurgical bonding of chips is instantaneously obtained by severe plastic deformation at elevated temperatures. It means that, in short processing time, the shear formation of chips is responsible for the better consolidation by extrusion. Shear deformation at elevated temperatures causes more crystal defects such as particles surface, grain boundary and dislocation acting as additional diffusion paths which trigger atom diffusion. In the low-temperature compaction of Ti powder using ECAP with back pressure, Lapovok et al. [28,29] reported that it was that the macroscopic shear strain experienced during compaction would promote local plastic deformation that was to be relatively severe in the vicinity of the intimate contact particle surfaces. High-temperature shear deformation will not only increase the area and proximity of contact, but also lead to local defect structures that enhance atom diffusion and promote bonding of chips across increased areas of intimate contact. Hence, high deformation will lead to an increase in contact area between the neighboring chips, thus increasing potential areas for diffusion bonding. Elevated temperature increases thermal activation for transport processes that will affect bonding at the interfaces.

Just shown in Table 1, multi-passes shear deformation could obviously improve room mechanical properties of the recycled specimens at 400 °C and lower the deformation temperature. Even, in the specimens recycled by one-pass deformation with extrusion ratio of 20:1 at 400 °C, the residual pores were still remained. Multi-passes shear deformation can cause more crystal defects such as particles surface, grain boundaries and dislocations. Fig. 10 showed TEM micrographs in the recycled specimen by one-pass and multi-passes extrusion at 400 °C. Multi-pass shear deformation can cause more fine grains, twins and higher dislocation density which act as additional diffusion paths. In order to obtain good bonding at the interfaces, it is necessary that shear deformation is introduced during high-temperature deformation.

5. Conclusions

1. Unlike that of sintering of fine powders, utilization of Mg chips with smaller specific surface area (i.e. coarser powder) contributes to easier solid-state bonding because of the decrease in specific surface area which promotes suppression of oxide contamination in the recycled specimen. Coarse chips

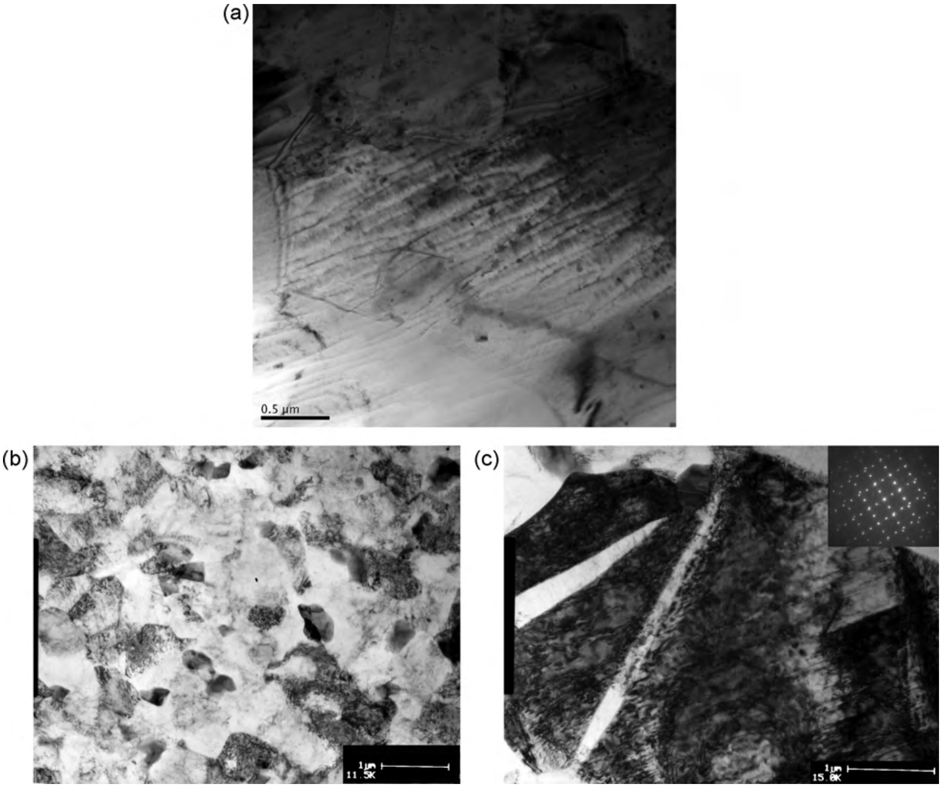


Fig. 10. TEM micrographs of (a) grains and dislocation in the recycled specimen by CE at 400 °C, and (b and c) grains, and {1 0 −1 2} twins in the recycled specimens by 6-pass CEC at 400 °C.

are easier for metallurgical bonding at the interfaces during the recycling.

2. Enhanced consolidation of chips is not only ascribed to the physical mechanisms caused by plastic deformation, but also to atom diffusion between chips triggered by shear plastic deformation at elevated temperatures.
3. It is postulated that the shear deformation of chips is responsible for the better consolidation of chips by high-temperature deformation. Multi-passes shear deformation breaks the oxide films easier into small particles which are dispersed within the grains or at the grain boundaries.

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