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THE PRODUCTION OF POWDER METALLURGY HOT EXTRUDED Mg-Al-Mn-Ca ALLOY WITH HIGH STRENGTH AND LIMITED ANISOTROPY

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Abstract

Rapidly solidified Mg-Al-Mn-Ca alloy produced by Spinning Water Atomization Process (SWAP) was hot extruded into rectangular bars, from which tensile and compression samples have been cut at 0°, 45° and 90° from the extrusion direction to study their anisotropy. Electron Back Scattered Diffraction (EBSD) has been used to investigate the texture evolution during the hot extrusion process. Both the Schmid factor and the intensity of the basal plane in the pole figure have been evaluated and correlated to the mechanical properties. Results have shown that the extruded rods exhibit high strength and limited anisotropy compared to many previously reported values for magnesium alloys. The reasons for that limited anisotropy were both the fine grained microstructure of the extruded material and the transverse component of the texture evolution.

Introduction

Concerns about power saving and environmental issues encouraged efforts to apply light weight materials, especially in automobiles. Magnesium is one of the most promising materials which show high strength to weight ratio. Wrought magnesium alloys are more likely to get increased markets due to their improved performance compared to cast alloys. However, applications of magnesium alloys are still limited due to their lack of formability at room temperature. This disadvantage arises from the features of the close packed hexagonal crystal structure which limit the deformation mechanisms at room temperature to basal slip [1]. Improvement of the mechanical properties of magnesium alloys could be obtained via various techniques, including, but not limited to, thermo-mechanical processing and chemical alloying [2, 3]. The Hall-Petch relationship, describing the trend of increasing the yield strength with decreasing of the grain size, was shown to prevail. The strengthening factor of Mg alloys in this relationship has shown higher values (0.2 to 0.34 MPa $\sqrt{m}$) compared to that of other materials [4]. This shows the strong effect of grain refinement on the properties of Mg alloys. The present authors have previously shown the considerable impact of rapid solidification powder metallurgy processing to improve the strength of Mg-Al-Mn-Ca alloy through the effects of grain refinement and the dispersion of tiny inter-metallic compound particles [5].

The anisotropy of the mechanical response of wrought Mg alloys caused by the strong texture has been shown as a barrier in the extension of their applications. Hence, the texture analysis became a very important tool for the evaluation of the mechanical response of Mg alloys [6]. The texture evolution during thermo-mechanical processing of Mg alloys has been investigated aiming at understanding their effects on properties of extruded bars [7-8]. It has been shown that the extrusion temperature has no effect on the texture formation [7]. Contrarily, it was also reported that it affects the amount of dynamic recrystallization [8]. However, most references agreed that the fiber texture was usually obtained after hot extrusion of Mg alloys in which the basal plane is aligned parallel to the extrusion direction. Texture evolution during hot deformation, like compression, has also been studied [9, 10], and the effects of both strain rate and processing temperature on the texture were shown through the Zener-Hollomon parameter.

Trials have been made to modify the texture formation in Mg alloys, and ultimately to improve their mechanical response, through the use of alloying elements, especially rare earth elements [11-14]. They have shown a remarkable modification of the texture of wrought Mg alloys owing to the reduced texture strength attained by the formation of randomized crystal orientation. That effect was shown to be mainly obtained through extensive grain refinement, solute atoms interactions, and nucleation of grains at shear bands rather than inside original grains [11-13]. In some other cases particle-stimulated nucleation of recrystallization has been reported to have a minor effect [14]. Alloying with Ca has been shown to reduce the texture strength due to the randomized nucleation of recrystallized grains near intermetallic compound particles [15]. Equal channel angular extrusion has also been reported to improve the ductility of Mg alloys in the longitudinal direction due to the inclined texture formation, but on the expense of reduced strength [16]. The effect of the texture on the mechanical properties of wrought Mg alloys has also been investigated and found to be mainly through favoring the deformation mechanism, namely slipping or twinning, based on the available texture pattern [17, 18]. The tensile and compression properties have shown to be of totally different nature for the same alloy due to the different possibilities of twinning activation [19, 20]. The grain size also has played an important role in determining whether the twinning mechanism becomes active or not, which if activated results in remarkable softening.

The aim of this work is to study the anisotropic behavior of hot extruded powder metallurgy Mg-Al-Mn-Ca alloy under both tension and compression loadings. That alloy has previously been shown to have good tensile properties due to the remarkable advantages of rapid solidification powder metallurgy processing of both grain and inter-metallic compound refinement and homogenous distribution [5].

Experimental work

Mg-6wt.%Al-0.26wt.%Mn-2.1wt.%Ca magnesium alloy powder produced using the spinning water atomization process (SWAP) was used. In SWAP process, gas atomization is combined with water atomization to produce very high cooling rates of about 10^6 K/s, which result in powders with fine microstructures. The details of the extensive study on the characteristics of the alloy SWAP powder can be found in the authors' previous reference [5]. For comparison, extruded cast material was also used to confirm the effect of rapid solidification of SWAP powder on the final properties of the extruded alloy. The cast alloy was produced using casting in a steel mold using the melting temperature of 1023 K. Microstructure analyses of the extruded bars were performed using Scanning Electron Microscope (SEM, JEOL: JSM-6500F) because of the fine grain size. X-ray Diffraction (XRD) analysis was used to investigate the phases existing in the alloy using an X-ray Diffractometer (Shimadzu: XRD 6100) over the range of 2θ of 20 to 80°.

As-received SWAP powders were consolidated using cold compaction at room temperature under the pressure of 600 MPa. Both consolidated powder billets and cast billets were then extruded at the temperature of 673 K. Extrusion was performed using a die that produces extrusion rods of 25 x 40 mm cross-section, which is equivalent to an extrusion ratio of 37. The preheating of billets was done just before extrusion using the heating rate of 1 K/s in Ar gas atmosphere. The billets were held at the extrusion temperature in the furnace for 60 min prior to extrusion to ensure homogenous temperature distribution.

The texture evolution in the extruded bars was investigated using the electron back-scattered diffraction (EBSD) analysis. The specimens for EBSD investigation were cut such that the observation plane is parallel to the extrusion direction. The specimen surface was prepared by grinding until 4000 grit emery paper, polishing using 0.25 μm diamond paste, electro-chemical etching using (37.5 vol.% H3Po4 + 62.5 vol.% ethanol) solution at 8 V for 60 s, and then by cleaning with methanol.

Both tensile and compression tests were performed on the extruded bars to evaluate their mechanical response at three different orientations, which have 0, 45 and 90° angles with the extrusion direction. Tests were performed using a universal testing machine (Shimadzu: Autograph AG-X 50 KN) at room temperature on three specimens for each material and processing condition, and the average of the three values was evaluated. The tensile specimens, having the diameter of 3 mm and the gage length of 12 mm, and compression specimens with 6 mm diameter and 15 mm length were evaluated using the strain rate of 5×10^{-4} /s.

Results and Discussion

The extrusion process of Mg-Al-Mn-Ca alloy in both its powder and cast forms resulted in the microstructures shown in Figure 1 observed on a plane normal to the extrusion direction. The extruded SWAP powder specimen shows a fine and homogenous microstructure with the average grain size of about 1.7 μm, as shown in Figure 1a, with grains shown by dotted ovals. The fine and homogenously dispersed inter-metallic compound particles in the structure of the extruded SWAP powder specimens, appearing as white dots shown by arrows, can also be seen. They exist both inside the grains and at the grain boundaries. It should be noted that the grain size, shown herein, is coarser than that previously reported by the authors for the same alloy SWAP powder extrusion in the case of smaller extrusion rods of 7 mm diameter (about 0.5 μm) [5]. This may be due to the grain growth that occurs in the case of bigger extruded bars because of longer heating times and slower cooling rates. In contrast, the microstructure of the extruded cast specimen, shown in Figure 1b, reveals coarser average grain size of about 7 μm with remarkable non-homogenity. However, that size of the extruded cast specimens of the Mg-Al-Mn-Ca alloy is still much finer than many other reported values of grain sizes of extruded Mg alloys [8, 12]. Bimodal microstructure with both coarse deformed and fine recrystallized grains can be observed. The inter-metallic compound particles, also shown by arrows reveal coarser size and lower dispersion compared with that of the extruded SWAP powder specimens. In both cases of extruded SWAP and cast billets, the inter-metallic compound of Al_2Ca can be detected after the dynamic recrystallization process accompanied by hot extrusion, as confirmed by the XRD result shown in Figure 2. However, the morphology of the compound particles varied significantly. In the case of the extruded SWAP powder specimens, the fine compound particles have been formed during the dynamic recrystallization process from the super saturated elements existing in the SWAP powder structure, as the very high cooling rate used in the fabrication of this powder did not permit the precipitation of these elements. On the other hand, the compound particles, originally existing in the structure of the cast billet of the investigated alloy, have either remained after extrusion or fractured during extrusion depending on their sizes and morphologies.

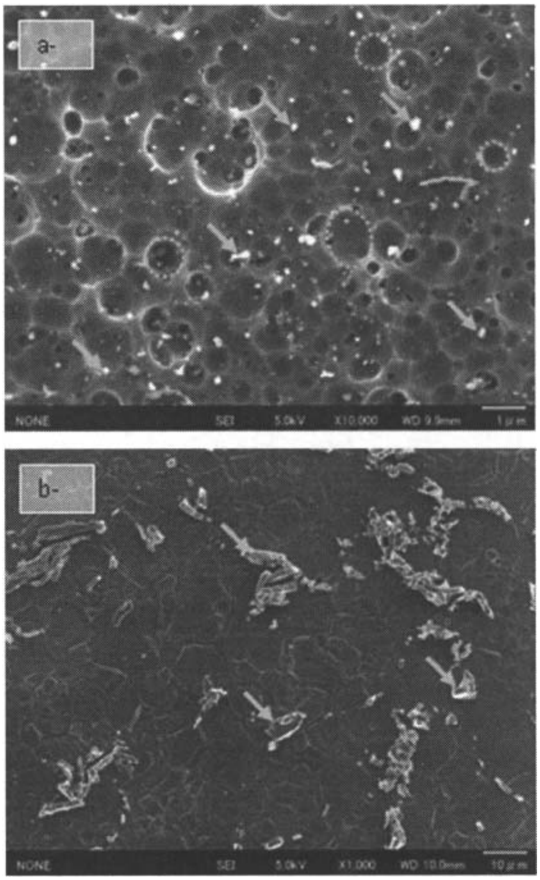

Figure 1. The SEM observation of (a) extruded SWAP powder and (b) extruded cast billet

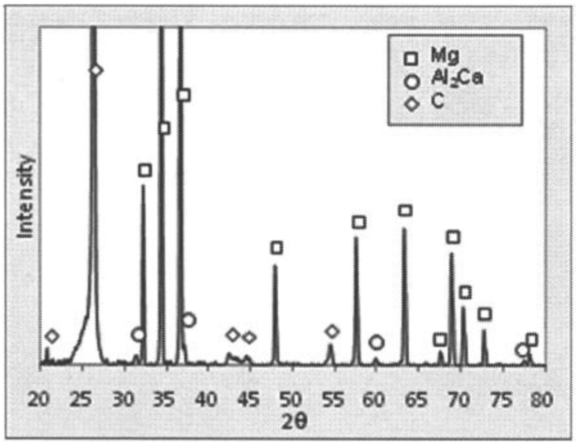

Figure 2. The XRD pattern of extruded SWAP powder specimen

The above shown microstructure of the extruded SWAP powder specimen has resulted in an improved mechanical response of this alloy compared to that of the extruded cast specimen, as shown in Figure 3, and in the authors' reference [5]. The tensile and compression test results at 0, 45 and 90° angles from the extrusion direction are shown in terms of the stress-strain plots for both extruded SWAP powder and cast billet. It can be shown that the extruded SWAP powder specimen possesses high values of yield and ultimate stresses while maintaining reasonable levels of ductility. It is evident that the strength of extruded SWAP powder is drastically increased compared to the extruded cast billet from the 50 % increase in tensile yield strength, and even 100 % increase in compressive yield strength. They are superior to previously reported values of the strength of Mg alloys via conventional routes. However, these results are lower than those presented earlier in the current authors' references for smaller extrusions of 7 mm extruded bars of the same alloy [5]. That is due to the grain growth that accompanied the larger size extrusions which is performed at longer times of homogenization, 60 min compared to 5 min for smaller size extrusions. Generally, both yield and ultimate strengths have decreased as the angle of the loading has increased from 0 to 45 and 90°. This behavior has occurred in both the tensile and compression cases. In contrast to the previously reported pattern of increasing the compression/tensile yield strengths' ratio with the angle, the investigated alloy SWAP powder has shown almost the same values of both tensile and compression yield strengths at all directions, with the ratio equals unity, as shown in Figures 3 a and b. However, the alloy has shown more strain hardening in the case of compression than that of tension. The elongation has also decreased simultaneously. However, the difference of both ultimate and yield strengths are much lower in the case of extruded SWAP powders than those for extruded cast billets. It could be shown from the figure that the extruded cast alloy shows more anisotropy than that of extruded SWAP powders in terms of wider difference in both yield strength and elongation at various directions, which is more noticeable for elongation levels, as shown in Figures 3 c and d.

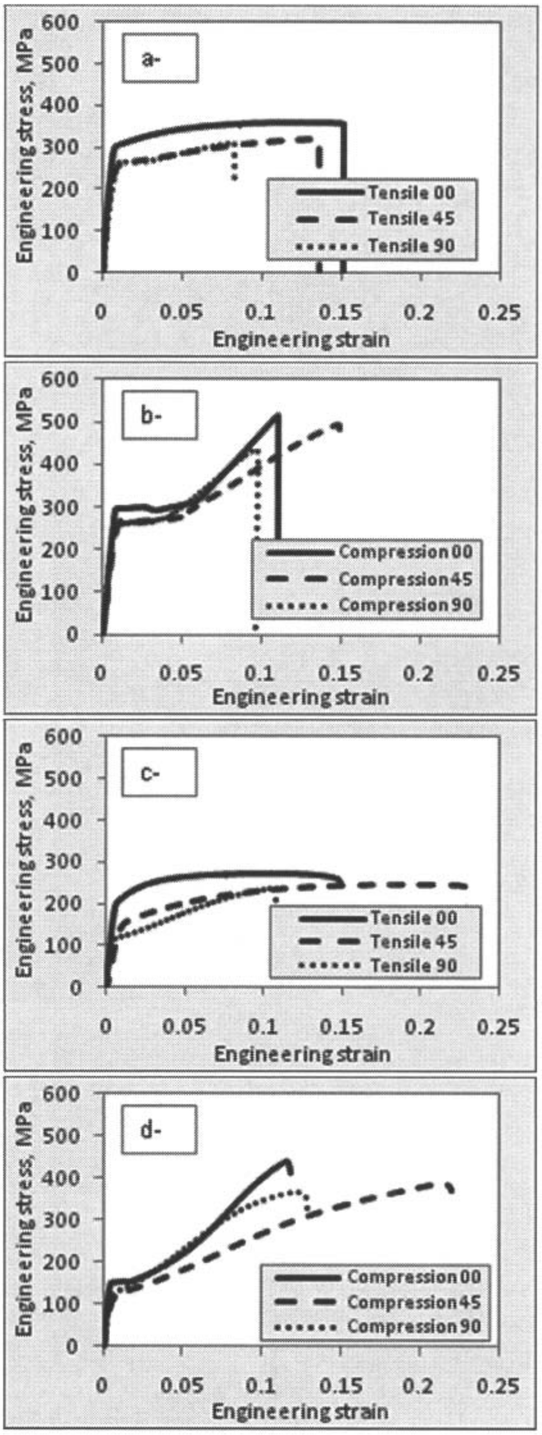

Figure 3. Stress-strain plots of tensile (a) and compression (b) of extruded SWAP powder, and tensile (c) and compression (b) of extruded cast billets

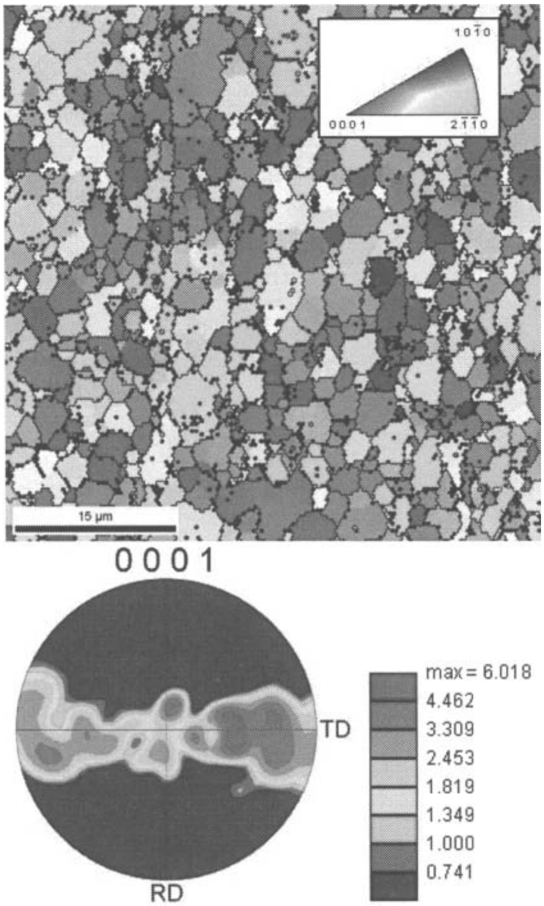

Figure 4. The EBSD analysis results of extruded SWAP powder specimen showing both the crystal orientation mapping and the basal plane orientation texture

In order to comprehend the aforementioned tensile and compression behaviors, the analysis of EBSD patterns has been investigated for both extruded SWAP powder specimen and extruded cast one, as shown in Figures 4 and 5. The inverse pole figure showing the crystal orientation mapping, with the extrusion direction vertically aligned, and the corresponding pole figure of the basal plane texture of the extruded SWAP powder specimen is shown in Figure 4. The homogenous distribution of grain sizes can easily be observed. The inter-metallic compound particles are not shown in this map, with the black dots representing their positions. The basal plane texture shows a mix of the normal and transverse components, which is consistent with the findings by other references [11]. The basal planes in most grains are aligned along the direction that makes an angle of about 60 to 80° with the normal direction towards the transverse side. The maximum intensity of the basal texture shows the value of 6.02, while the Schmid factor shows the average value of 0.18. On the other hand, the extruded cast specimen shows almost the same pattern of basal plane texture component, but with its maximum intensity increased to about 9.62, as shown in Figure 5. The Schmid factor has shown a very close value of 0.17 to that of the extruded SWAP powder specimen. However, the grain sizes have shown non-homogenous distribution, an observation that is consistent with that of SEM images. The observed area includes some examples of the fractured inter-metallic compound particles, as previously mentioned, in contrast with the ones shown in Figure 1b. These fractured particles appear as clusters of vertically aligned black dots in the inverse pole figure as they are accumulated during extrusion.

Separating both deformed and recrystallized grains in separate maps and pole figures provides deeper understanding of the effect of recrystallization during hot extrusion of the investigated alloy, as shown in Figure 6. Deformed grains show higher maximum intensity of the basal plane texture than that of recrystallized ones. However, as the volume fraction of both types of grains has similar values, the maximum intensity ultimately takes an intermediate value. It should also be noted that both types of grains show similar orientations of the basal plane compared to each other, and to that of the extruded SWAP powder specimen.

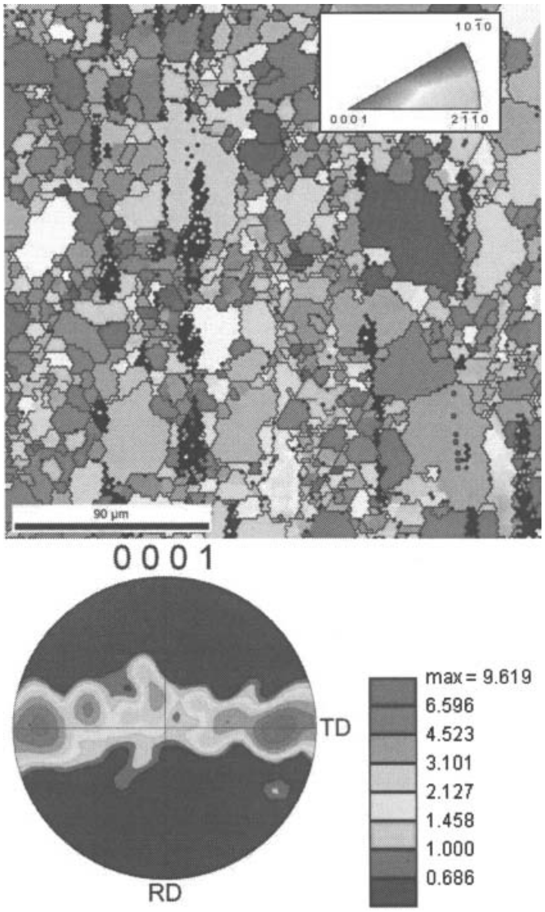

Figure 5. The EBSD analysis results of extruded cast specimen showing both the crystal orientation mapping and the basal plane orientation texture

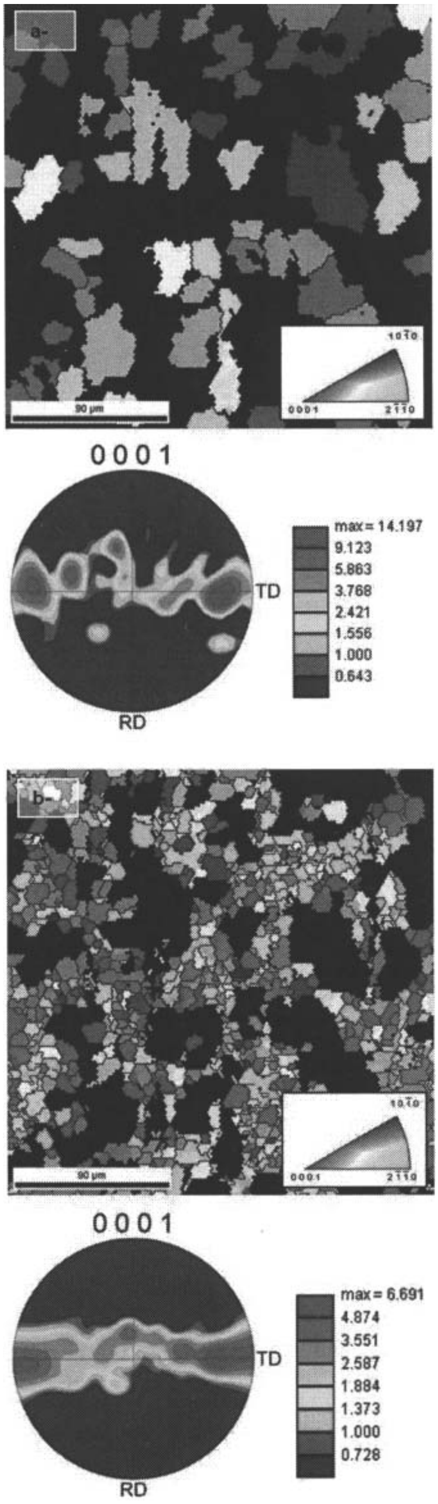

Figure 6. The EBSD results of extruded cast specimen showing (a) separated deformed grains and (b) separated recrystallized grains

It has been shown in previous reports that the Ca element has the effect of weakening of the texture of Mg alloys through the random nucleation of recrystallized grains due to the formation of inter-metallic compound particles [15]. This effect can also be observed in this study in terms of the decreased maximum intensity of the basal texture of the extruded SWAP powder specimen compared to that of extruded cast due to the fine distribution of intermetallic compounds, which did not occur in the case of extruded cast. Hence the decreased asymmetry of both tensile and compression results in various directions can be explained through the effect of randomized nucleation of recrystallized grains stimulated by compound particles. This claim may also be supported by the observation of the decreased tensile and compression strengths from 0 to 45°, and the similarity of both of 45 and 90° angle results, which can be correlated to orientation of the basal plane being randomized in both the normal and transverse directions. On the other hand, the tensile-compression isotropy shown in this alloy can be attributed to the remarkably refined grain sizes. It has been previously shown that the fine grain size results in the difficulty of the activation of the twinning mechanism, which if activated results in the remarkable softening during deformation in either tensile or compression directions based on the available texture [17, 18].

Conclusion

Owing to the advantages of the powder metallurgy processing of Mg-Al-Mn-Ca alloy, the following conclusions can be drawn:

- The alloy has shown very promising tensile and compression results.
- Both the tensile and compression results have shown very low asymmetry in various loading angles with respect to the extrusion direction.
- Isotropic tension-compression behavior could be obtained at various loading directions.

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