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Microstructure Evolution of Twin-Roll Cast and Hot-Rolled WZ73 Alloy During the Finishing Heat-Treatment

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Abstract. The microstructural evolution of the WZ73 magnesium alloy (Mg-7.4Y-3.8Zn-0.4Zr) was systematically investigated during finishing heat-treatments following twin-roll casting and hot rolling at equivalent strain rates of 17 s^{-1} and 50 s^{-1}. Hot rolling at 500 °C was performed to achieve a logarithmic strain of 0.7 (thickness reduction from 5.3 mm to 3 mm). Higher strain rates during hot rolling enhanced dynamic recrystallization (DRX), resulting in refined microstructures, whereas lower strain rates promoted the formation of lamellar long period stacking ordered (LPSO) phases. Subsequent heat-treatments at 200 °C to 550 °C for up to 24 hours revealed temperature-dependent microstructural transformations. At 500 °C, complete recrystallization occurred with minimal grain growth, while 550 °C caused grain coarsening, partial grain boundary melting, and morphological changes of the LPSO phase from lamellar to spherical and rod-like structures. Notably, at temperatures above 500 °C, prior hot rolling had limited influence on microstructure. The microstructure and phase evolution were characterized using optical and scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD).

Introduction

The increasing demand for environmentally friendly transportation solutions has led to a global focus on lightweight design. In response to the need for CO_2-reduced solutions, current concepts are being retained, but with the clear goal of reducing the weight of machine parts in industries such as automotive and aerospace. In this context, magnesium has gained significant importance as a material, due to its low density of 1.74 g/cm^3, making it the lightest metallic construction material. Compared to traditional construction materials like steel, magnesium offers not only significant weight savings and good mechanical properties, but also excellent long-term stability and high recyclability. [1]

The application of magnesium alloys, particularly with additions such as aluminum, manganese, zirconium, zinc, yttrium, or other rare earth elements, has proven to be a reliable solution. The addition of alloying elements like yttrium and zinc can improve the strength of magnesium alloys without compromising ductility. Previous studies have shown that, for example, a twin-roll cast and hot-rolled Mg-6.8Y-2.5Zn alloy (WZ73) exhibits strengths of up to 292 MPa at a strain of 7.6 %. [2] The Long Period Stacking Ordered Phases (LPSO) present in these alloys play a crucial role in this due to their ability to hinder dislocation movement, them acting as load-bearing structures and their influence on recrystallisation mechanisms. However, despite the progress made in the research of LPSO-containing magnesium alloys, there is still a lack of understanding of the influence of finishing heat-treatment on the microstructure of such alloys. Specifically, there is a need for detailed investigations on the microstructure evolution of twin-roll cast and hot-rolled WZ73 alloy during final heat-treatment as the morphology of the LPSO phases has been shown to differ after twin-roll casting compared to casting or extrusion. [3,4]

This paper aims to investigate the microstructure evolution of twin-roll cast and hot-rolled WZ73 alloy during final heat-treatment, with a focus on the morphological development of the LPSO phase through various heat-treatment concepts. The goal is to gain insights into the influence of these treatments on the microstructural properties after a rolling process.

Material and Methods

In the present investigation, the twin roll casting (TRC) process was carried out at the pilot plant of the Institute of Metal Forming (IMF) at the TU Bergakademie Freiberg. [5] The TRC process was carried out using a twin-roll casting temperature of 730 °C and a twin-roll casting speed of 1.5 m/min. The sheets had a thickness of approx. h_0 = 5.3 mm. Afterwards the sheets were heat-treated at 500 °C for 1 h. The chemical composition of the initial material is summarized in Table 1.

Table 1. Chemical composition of Mg-7.4Y-3.8Zn-0.4Zr [wt.%] alloy.

Mg [wt.%]	Y [wt.%]	Zn [wt.%]	Zr [wt.%]
Balance	7.4	3.8	0.4

Hot rolling was carried out on the pilot plant at laboratory scale at the IMF. The rolls had a diameter d of 361 mm and were pre-heated to 120 °C in order to minimize temperature losses. Previous studies of Ullmann et al. [2] discovered that the equivalent strain rate has a bigger influence on the microstructure than the equivalent strain. Therefore, in this study two different microstructures were generated by hot rolling in one single pass by reducing the height to h_1 = 2.9 mm and using a rolling speed v_w of 0.5 m/s and 1.5 m/s. To calculate the equivalent strain φ_v (Eq. 2) and equivalent strain rate $\dot{\varphi}_v$ (Eq. 3) firstly the compressed length l_d was determined (Eq. 1).

$$l_d = \sqrt{\frac{d}{2} \cdot (h_1 - h_0)}. \tag{1}$$

$$\varphi_v = \frac{2}{\sqrt{3}} \cdot \ln \frac{h_1}{h_0}. \tag{2}$$

$$\dot{\varphi}_v = \frac{v_w}{l_d} \cdot \varphi_v. \tag{3}$$

After the hot rolling the sheets underwent a final heat-treatment. For this purpose, temperatures from 200 °C to 550 °C for 15 min to 24 h were applied. Additionally, different cooling strategies (quenching in water and air cooling) were tested based on other research. [6–9]

Samples intended for microstructural examination were prepared employing conventional grinding and polishing techniques. Two distinct acids were utilized for the etching process. Initially, a solution comprising distilled water, glacial acetic acid, ethanol, and picric acid was employed, followed by the application of nitric acid in the subsequent phase. The microstructural analysis was executed utilizing optical microscopy with the Keyence VHX 6000 at the IMF in Freiberg, Germany, with the specimens being assessed in the longitudinal orientation. In addition, a scanning electron microscope evaluation was performed using a ZEISS GeminiSEM 450 at the same facility. X-ray diffraction analysis was conducted on a Seifert-FPM URD 6.5 apparatus at the Institute of Materials Science at TU Bergakademie Freiberg, employing CuKα radiation (λ = 1.540598 Å) for phase identification. The diffraction patterns were recorded over a 2θ range of 18° to 150°, with a step interval of 0.02° and a duration of 25 s per step.

Results and Discussion

Initial Material.

Both microstructures consist of darker network like shapes which are probably the LPSO phases precipitated at the grain boundaries. The lighter areas are the α-Mg matrix. Regardless of the applied strain rate, no complete dynamic recrystallisation was achieved. After hot rolling with an equivalent strain rate of 17 s^{-1} the average grain size was estimated as 28 µm ± 13 µm, as the microstructure consists of a mixture of larger grains and big grains which are elongated in rolling direction as shown in Fig. 1a. The fraction of recrystallized grains was measured as approximately 2 %. Additionally, more lamellar structures could be observed within the grains compared to the microstructure of the with 50 s^{-1} hot rolled material (see Fig. 1b). For this microstructure the recrystallized fraction was

estimated as 32 % and an average grain size of 12 µm ± 9 µm. Due to the higher equivalent strain rates it can be estimated, that during hot rolling the temperature losses are lower and therefor dynamic recrystallization is promoted more easily. Previous research identified the continuous dynamic recrystallization and twinning induced dynamic recrystallisation as the main mechanisms. [2] Those were identified in this work as well.

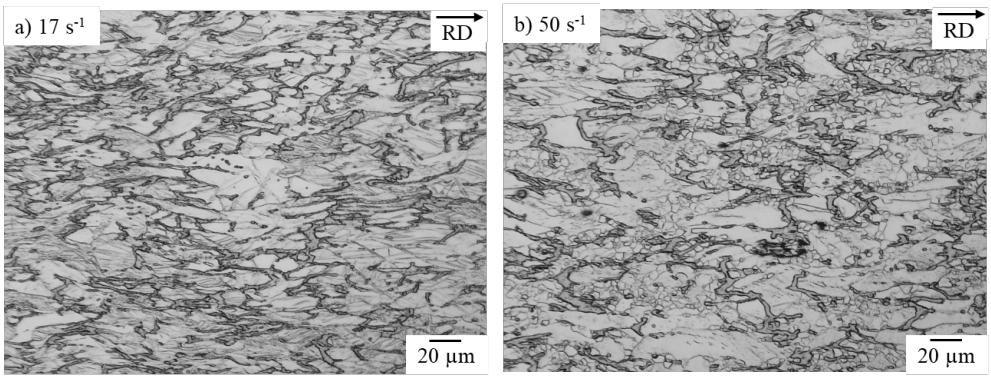


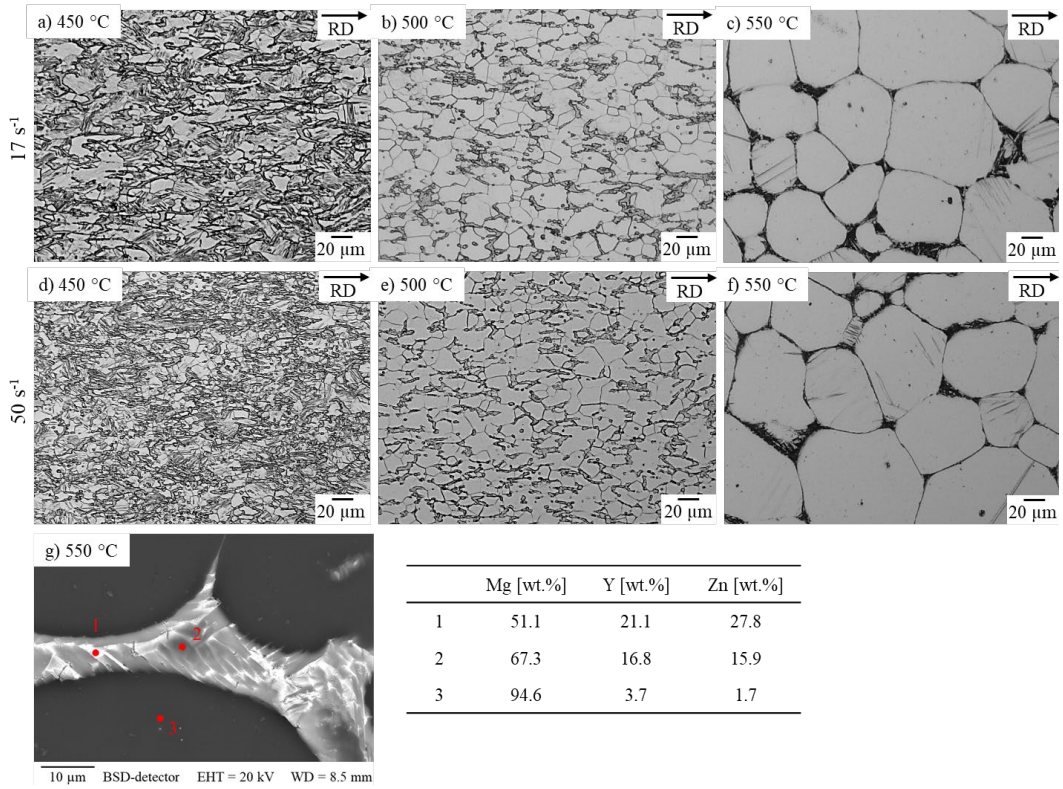
Fig. 1. Optical images of the initial material (twin-roll cast, heat-treated) hot-rolled with different equivalent strain rates a) 17 s^{-1} (~ 2 % recrystallized); b) 50 s^{-1} (~ 32 % recrystallized).

Influence of Temperature.

Regarding the influence of the temperature of the final heat-treatment there is a severe change noticeable when exceeding 500 °C. Figure 2 shows the influence of the final heat-treatment at different temperatures after 1 h. It is visible, that at temperatures below 500 °C the microstructure is still significantly influenced by the previous forming process. The microstructures show both formed and recrystallized grains although the recrystallized fraction increases with higher temperatures. The microstructures are fully recrystallized at temperatures of 500 °C and above, when heat-treated for 1 h. If the holding time is increased, lower temperatures of 450 °C already lead to a fully recrystallized microstructure. These results are in alignment with research from Xiao et al. [7], who showed that at 420 °C even heat-treatments for 120 h do not favor a fully recrystallized microstructure as the LPSO phases can hinder the recrystallisation and grain growth. Especially the lamellar 14H phase can hinder recrystallisation whereas the 18R phase can promote it. [7,10] Within the microstructure both a lamellar LPSO phase within the grains (probably 14H phase) and a block-shaped at the grain boundaries (probably 18R) could be detected.

At temperatures above 500 °C already 15 minutes are enough to promote a fully recrystallized microstructure and no structures from the deformation process remain. Additionally, a slight grain growth can be detected (19 µm ± 10 µm). The LPSO phase mainly precipitated at the grain boundaries, only a few spherical structures can be found within the grains. Both are probably of the 18R stacking sequence. This agrees with a previous study by Yuan et al. [11], who observed the formation of a stable LPSO-phase during the solid solution treatment and cooling of a Mg-Dy-Ni alloy. The rod-shaped LPSO-phase is probably characterized by a 14H-type structure. The formation of the rod-shaped LPSO-phase is a result of the growth of the spherical LPSO-phase during the holding time. Firstly, the blocky LPSO phase transforms into the spherical structure which then grows into the rod-shaped structure. Whereas the blocky and spherical structures probably belong to the 18R phase, the rod-shaped structures are said to be the 14H phase. If the final heat-treatment temperature is further increased to e.g. 550 °C the morphology of the grains changes drastically. The grains become very spherical and grow to an average size of 60 µm ± 28 µm. Along the grain boundaries black areas with a network-like shape can be detected (see Fig. 2c and f). When those structures are investigated via the SEM (see Fig. 2g) then there are additional structures visible. The white fish-bone like structures (measuring point 1) show an even higher Y and Zn content than the grey structures (measuring point 2). According to the literature [11] the white structure can be identified as the W phase ($Mg_3Zn_3Y_2$) and the grey structures belong to the 18R LPSO phase. The W phase appears as the eutectic phase because at temperatures above 540 °C a partial melting of the grain

boundaries and areas with high alloying element content can appear. [11,12] Therefore the grains show a round shape and the appearance of the W phase can be explained.



	Mg [wt.%]	Y [wt.%]	Zn [wt.%]
1	51.1	21.1	27.8
2	67.3	16.8	15.9
3	94.6	3.7	1.7

Fig. 2. Optical images showing the influence of the finishing heat-treatment temperature (a,d) 450 °C; b,e) 500 °C; c,f) 550 °C) applied for 1 h for the sheets hot rolled at 17 s^{-1} (a-c) and 50 s^{-1} (d-f); g) SEM image including EDS measuring points of the at 50 s^{-1} hot rolled and at 500 °C heat-treated sheet.

Influence of the Holding Time.

The progression of microstructural changes in the alloy is notably affected by the duration of the finishing heat-treatment. Extended holding times at elevated temperatures lead to distinct transformations in the morphology of the LPSO phase.

At 500 °C, a short holding time of 15 minutes results in a microstructure where block-like LPSO phases dominate, accompanied by some fine dispersions of LPSO structures. However, after 1 hour at the same temperature, the LPSO phase appears more refined, and a wider variety of morphologies - including spherical and rod-like structures - are visible, as illustrated in Fig. 3b. As the holding duration increases further, the block-shaped phases gradually vanish, and the LPSO phase predominantly appears in spherical and rod-shaped forms (see Fig. 3c).

These observations align with findings by Yuan et al. [13], who documented the stabilization and transformation of LPSO phases during solution treatment and subsequent cooling in a Mg-Dy-Ni alloy. X-ray diffraction (XRD) analysis (see Fig. 3d) performed on samples treated at 500 °C for 24 hours supports the coexistence of both 18R and 14H LPSO structures. The XRD pattern displays several peaks corresponding to the 18R-type phase and a prominent peak near 28°, which is indicative of the 14H-type structure, thus confirming the simultaneous presence of both LPSO variants. [13]

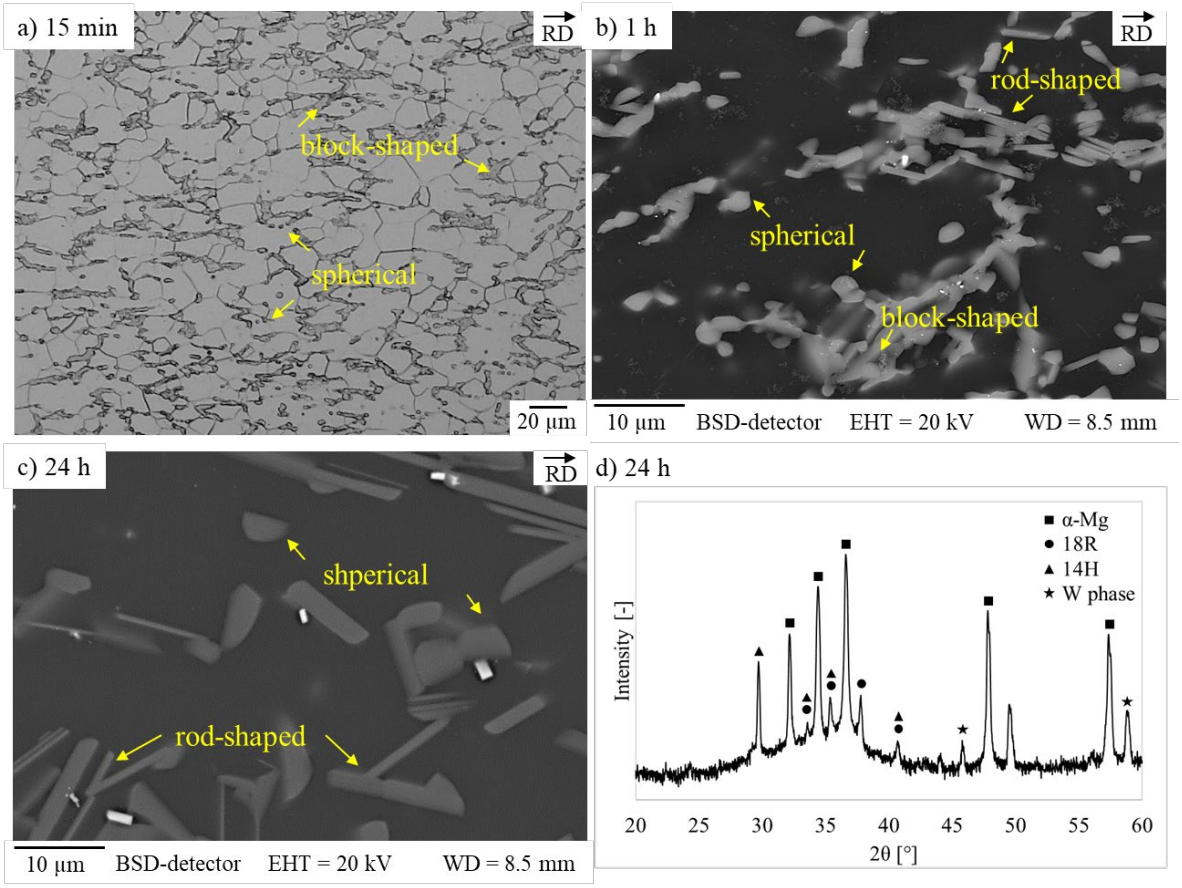


Fig. 3. a) Optical image and b,c) SEM images of the material with a final heat-treatment at 500 °C for a) 15 min; b) 1h and c) 24 h and d) XRD analysis of the final heat-treatment at 500 °C for 24 h.

Summary

The microstructural evolution of the studied alloy is strongly influenced by deformation conditions and finishing heat-treatment. Initial hot rolling at varying strain rates resulted in partial dynamic recrystallization, with higher strain rates (> 50 s^{-1}) promoting finer grains and increased recrystallized fractions due to reduced thermal loss. The as-rolled microstructures featured α-Mg grains and LPSO phases at grain boundaries and within grains.

Finishing heat-treatments revealed a critical temperature threshold around 500 °C. Below this, recrystallization remained incomplete after 1 h, while full recrystallization occurred at or above 500 °C, with higher temperatures accelerating the process and promoting grain growth. Holding time further influenced the LPSO morphology: block-shaped phases at short durations transformed into spherical and rod-like forms with longer exposure. XRD confirmed the coexistence of 18R and 14H LPSO structures and the eutectic W phase. Overall, both temperature and holding time critically affect recrystallization and LPSO-phase development.

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References

[1] J. Freudenberger, M. Heilmaier, Materialkunde der Nichteisenmetalle und -legierungen, Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, 2020.

[2] M. Ullmann, K. Kittner, T. Henseler, C. Krbetschek, D. Rafaja, R. Kawalla, U. Prahl, Dynamic recrystallization and texture evolution of Mg-6.8Y-2.5Zn-0.3Zr alloy during hot rolling, Procedia Manuf. 50 (2020) 809–816. https://doi.org/10.1016/j.promfg.2020.08.146.

[3] K. Kittner, M. Ullmann, U. Prahl, A Comparative Study on the Hot Deformation Behavior of As-Cast and Twin-Roll Cast Mg-6.8Y-2.5Zn-0.4Zr Alloy, Materials 14 (2021) 3628. https://doi.org/10.3390/ma14133628.

[4] B. Chen, X. Li, C. Lu, D. Lin, Recrystallization and microstructural evolution during hot extrusion of Mg97Y2Zn1 alloy, Met. Mater. Int. 20 (2014) 489–497. https://doi.org/10.1007/s12540-014-3012-7.

[5] K. Kittner, M. Ullmann, F. Arndt, R. Kawalla, U. Prahl, Microstructure and Texture Evolution during Twin-Roll Casting and Annealing of a Mg–6.8Y2.5Zn–0.4Zr Alloy (WZ73), Crystals 10 (2020) 513. https://doi.org/10.3390/cryst10060513.

[6] X. Zhou, C. Liu, Y. Gao, S. Jiang, X. Han, Z. Chen, Evolution of LPSO Phases and Their Effect on Dynamic Recrystallization in a Mg-Gd-Y-Zn-Zr Alloy, Metall. Mater. Trans. A 48 (2017) 3060–3072. https://doi.org/10.1007/s11661-017-4081-2.

[7] J. Xiao, Z. Chen, J. Shao, T. Chen, X. Lin, C. Liu, Evolution of long-period stacking ordered phases and their effect on recrystallization in extruded Mg-Gd-Y-Zn-Zr alloy during annealing, Mater. Charact. 167 (2020) 110515. https://doi.org/10.1016/j.matchar.2020.110515.

[8] R. Lu, J. Wang, Y. Chen, D. Qin, W. Yang, Z. Wu, Effects of heat treatment on the morphology of long-period stacking ordered phase, the corresponding damping capacities and mechanical properties of Mg–Zn–Y alloys, J. Alloys Compd. 639 (2015) 541–546. https://doi.org/10.1016/j.jallcom.2015.03.208.

[9] X.R. Dong, H. Yu, S.M. Kang, W. Yu, B.A. Jiang, D. Chen, Z.F. Wang, F.X. Yin, K.S. Shin, A comparable study of Mg98.15Y1Zn0.85 sheets fabricated by twin-roll casting and direct-chill casting and related annealing behavior, Mater. Sci. Eng. A 815 (2021) 141316. https://doi.org/10.1016/j.msea.2021.141316.

[10] T. Itoi, T. Suzuki, Y. Kawamura, M. Hirohashi, Microstructure and Mechanical Properties of Mg-Zn-Y Rolled Sheet with a Mg₁₂ZnY Phase, Mater. Trans. 51 (2010) 1536–1542. https://doi.org/10.2320/matertrans.L-M2010816.

[11] Y. Meng, Q. Chen, S. Sugiyama, J. Yanagimoto, Effects of reheating and subsequent rapid cooling on microstructural evolution and semisolid forming behaviors of extruded Mg–8.20Gd–4.48Y–3.34Zn–0.36Zr alloy, J. Mater. Process. Technol. 247 (2017) 192–203. https://doi.org/10.1016/j.jmatprotec.2017.04.001.

[12] C. Zheng, S. Chen, M. Cheng, S. Zhang, Y. Li, Y. Yang, Controlling dynamic recrystallization via modified LPSO phase morphology and distribution in Mg-Gd-Y-Zn-Zr alloy, J. Magnes. Alloys 11 (2023) 4218–4234. https://doi.org/10.1016/j.jma.2023.01.006.

[13] L. Yuan, G. Bi, Y. Li, J. Jiang, Y. Han, D. Fang, Y. Ma, Effects of solid solution treatment and cooling on morphology of LPSO phase and precipitation hardening behavior of Mg–Dy–Ni alloy, Trans. Nonferrous Met. Soc. China 27 (2017) 2381–2389. https://doi.org/10.1016/S1003-6326(17)60264-4.

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