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Constitutive behavior of as-cast magnesium alloy Mg-Al3-Zn1 in the semi-solid state

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Keywords: Semi-solid; Magnesium alloy; Tensile properties; Hot tearing; Direct chill casting

Abstract:

The first reported tensile semi-solid stress/strain data for as-cast magnesium alloy Mg-Al3-Zn1 are provided. The results show that the maximum tensile stress at the solidus temperature (460°C) was 13 MPa. Furthermore, this alloy has no ductility above 540°C, and cannot sustain tensile stresses above 560°C. Based on these tests, an equation relating the maximum tensile stress with temperature was derived. The microstructure of the tested specimens was also examined to link the tensile properties to fraction solid and microstructure.

Paper Text:

Having the highest strength-to-weight ratio of common structural metals, magnesium alloys offer many benefits in terms of reduced weight and energy savings for both the automotive and aerospace industries, in spite of their higher cost. These benefits have lead to a significant increase in the demand for cast and wrought magnesium products over the past few years [1]. One of the most common commercial magnesium alloys for wrought products is AZ31, due to its good mechanical properties. As compared to both aluminum and steel, the use of AZ31 provides a significant mass reduction for applications with large surface area. The Direct Chill (DC) casting process, which is utilized to produce the starting material for this alloy, is receiving more attention for the sake of process optimization [2] and defect reduction.

The DC casting process has been used to cast magnesium alloys for approximately 50 years. Although much work has been done to minimize solidification defects, hot tearing, cold cracks and dimensional control remain serious issues. Hot tears are thought to occur as a result of the development of tensile strains in the casting at high fraction solid (f_s) due to temperature gradients and/or mechanical constraints [3], in combination with limited liquid feeding [4] and low ductility [5]. In recent years, a number of mathematical models [6-8] have been developed to quantify the influence of casting parameters on the development of semi-solid thermal stresses and strains, in an effort to reduce solidification defects. These models require prior knowledge of the magnesium constitutive behaviour in the as-cast state over a wide range of temperatures and strain rates. While prior research has been performed to characterize AZ31 under compressive

loading and in the fully solid state (*e.g.* [9, 10]), little is known regarding the semi-solid constitutive behaviour of AZ31 and of magnesium alloys in general.

Due to the technical challenges of thermal control, and stress/strain measurement, it is inherently difficult to measure the semi-solid constitutive behavior of metallic alloys. Methods for such tests can be divided into two categories: (i) cooling from the liquid state to the semi-solid state or (ii) reheating a cast sample from room temperature up to the semi-solid temperature range. A summary of these methods, along with a review of the prior literature has been previously reported [11, 12]. In the present research, reheating tensile tests were carried out on as-cast AZ31 in the solid at high temperature and semi-solid to determine the stresses and strains that can be sustained by magnesium alloys at high fraction solid. These measurements will improve the mathematical simulations of DC casting, and hence aid in reducing solidification defects related to deformation.

The as-cast AZ31 magnesium alloy used in this study, with composition Mg–3.21wt%Al–1.02wt%Zn–0.31wt%Mn, was industrially DC cast by Timminco Ltd., Canada. The material consists of equiaxed-globular grains with an average grain size of 203 ± 12 μm, and fully-solid constitutive behavior as provided in [9]. An optical micrograph of the original as-cast grain structure is shown in Fig. 1. Cylindrical tensile specimens (ℓ = 120 mm, ϕ = 10 mm) were machined from the ingot with their long axis both normal to the casting direction and parallel to the ingot surface. Each specimen also contained a central reduced region (ℓ = 15 mm, ϕ = 5 mm).

The semi-solid tensile tests were performed using a DSI Gleeble 3500 thermomechanical simulator in combination with the two thermocouple technique previously developed by Phillion *et al.* [12] for aluminium alloys. Tests were conducted at temperatures between 400°C and 570°C and at a strain rate of $\sim 10^{-3}$ s^{-1}. The relevant portion of the fraction solid – temperature relationship for AZ31, experimentally derived by Lu [13], is given in Table A. During testing, the force, f, was recorded using a 4500 N load cell, and the current diameter, D, of the gauge region was recorded using a Beta LaserMike laser dilatometer. The true stress σ and true strain ε were derived from the recorded force and diameter, based on the standard equations of $\sigma = 4f / (\pi D^2)$ and $\varepsilon = 2\ln(D_0/D)$, where D_0 is the initial diameter.

One significant technical challenge in testing magnesium alloys at semi-solid temperatures is the risk of fire since these alloys are prone to burning. There is also the potential for an explosion

if water is present since they can produce hydrogen gas. In order to perform the experiments, a number of precautionary measures were taken to minimize these safety hazards. These measures included modifying a fire extinguisher to interface with the Gleeble chamber and reducing the partial pressure of oxygen in the Gleeble chamber using a combination of air removal via vacuum and argon back-fill.

After the tensile tests, a number of the tensile specimens were sectioned parallel to the loading direction for optical metallography and scanning electron microscopy of the fracture surfaces. The specimens for optical metallography were mechanically ground, polished using diamond suspensions of 6 and 1 μm, and then etched for ~20 s with an acetic-picric solution (10ml acetic acid, 10ml water, 4.2g picric acid, 70ml ethanol) to reveal the microstructure.

The solid (high temperature) and semi-solid stress/strain response of as-cast AZ31 as a function of temperature is shown in Fig. 2. The results have been plotted on two separate graphs (Fig. 2(a), 402–494°C; Fig. 2(b), 514–570°C), as there is a substantial decrease in ductility between 494°C (f_s ~0.98) and 514°C (f_s ~0.96). As can be seen in both figures, the yield stress is observed to decrease as a function of temperature for all tests. In Fig. 2(a), the typical material behavior for the solid at high temperature and semi-solid at low fraction liquid is shown – *i.e.* there is an increase in stress with deformation until a yield point is reached, the stress then decreases slightly with increasing strain (the material softens) followed by a period of rapidly decreasing stress (necking). In contrast, the material containing a higher fraction liquid, Fig. 2(b), shows little ductility.

In the solid state, the softening shown in Fig. 2(a) is due to time-dependent creep effects. In the semi-solid state, the rapid drop in stress is thought to be due to the rapid accumulation of internal damage (i.e. voids and cracks) during deformation [14]. Since stress is derived from the ratio of force to cross-sectional area based on the external specimen diameter, its true value is underestimated because of the presence of internal damage. In this figure, it also appears that the observed ductility at 402°C is much lower than the ductility at 425°C. This is because in the test at 402°C the centre of the neck formed outside the laser dilatometer measurement zone. The combination of considerable necking and erroneous dilatometer measurements results in apparent reduced ductility. When the post-deformed specimen diameter was measured using calipers, the ductility at 402°C was found to be much larger than the ductility at 425°C.

In Fig. 2(b), it can be seen from the stress/strain curves that the higher temperature semi-solid AZ31 does not exhibit a well-defined yield point upon tensile loading. This is likely due to the deformation occurring in the liquid within the cross-section. Furthermore, the ductility at all temperatures is quite low, with true strain values less than 0.15%. Thus, it appears that the semi-solid deformation is greatly dependent on the extent of the liquid phase.

The variation in maximum tensile stress and ductility with temperature is shown in Fig. 3. The maximum stresses reported are the peak flow stresses for tests in which the material exhibited ductility above a strain of 0.01, and the maximum stress prior to failure for tests in the absence of ductility. Ductility is defined as strain to failure when the alloy exhibits some ductility, and is assumed to be zero when there is no detectable diameter change during testing. As can be seen in Fig. 3, the maximum stress decreases almost linearly with increasing temperature for the entire temperature range examined. Above 565°C, the flow stress is insignificant. Above 514°C, the ductility is quite small, and the material loses all ductility at a temperature of 540°C. Note that the ductility for the test conducted at 402°C has been excluded from the figure due to the erroneous dilatometer measurement, as previously discussed.

Fig. 4 shows optical micrographs of the specimens tested at (a) 474°C, (b) 526°C, and (c) 566°C. These specific micrographs were chosen to compare the fracture surfaces of tests containing different fraction liquid. Note that the loading direction was horizontal. A number of salient observations may be made. Firstly, the lowest-temperature micrograph, Fig. 4a (474°C), contains a number of large cracks at the grain boundaries immediately behind the fracture surface. Some small voids also exist along the grain boundaries further away from the fracture surface. With increasing test temperature, (Fig. 4b and 4c), the voids become smaller and more evenly distributed. Thus the damage that lead to the crack would have been much more localized. Secondly, the edges along the intergranular openings in Fig. 4a are quite rough as compared to Fig. 4b and 4c. These rough edges suggest that, at 474°C, the liquid film at the grain boundary is thinner and may be discontinuous whereas at 526°C and 566°C, the liquid is thicker and continuous. Thirdly, the fracture surfaces Fig. 4b and 4c appear to be covered by a layer with a much finer microstructure. It is believed that this layer is liquid formed when the sample fails and an arc is generated briefly between the two surfaces. The rapid heating of the surface causes localized melting and this material is then rapidly quenched resulting in a structure distinct from

the as-cast microstructure. In Fig 4c, this liquid appears to have flowed down the side (upward facing side) of the sample.

The fracture surfaces of two semi-solid test specimens, tested at 474°C and 514°C, were examined using a Hitachi S-3000N scanning electron microscope to relate the surface morphology of the fractured as-cast AZ31 to the deformation temperature and the presence of liquid in the alloy. In Fig. 5a, the fracture surface of a specimen tested at 474°C ($f_s \sim 0.99$) is shown. It can be seen from this figure that most of the surface is dominated by complex topography showing typical ductile fracture features. However, some isolated regions appear smooth, indicating partial melting of the surface. In Fig. 5b, the fracture surface of a specimen tested at 514°C ($f_s \sim 0.97$) is shown. As can be seen in the figure, the further increase in test temperature increases the extent to which liquid plays a role in the deformation since the surface of each grain is covered almost entirely by a smooth thin liquid film. The grain boundaries are also clearly evident on the surface of the images.

The results of the tensile testing and microstructural examination show that as-cast AZ31 behaves in a predominantly ductile manner between 400°C and 514°C. At temperatures between 514°C and 542°C, AZ31 shows very low ductility (<0.0015). Above ~542°C, the alloy shows no significant ductility, and cannot sustain tensile stress above ~ 560°C. These critical temperature values can be translated into fraction solid, although care must be taken in the evaluation since the fraction solid on reheating may not be the same as that experienced during solidification due to kinetic effects. Based on the fraction solid curve given in Table A, the sharp transition from moderate ductility to low levels of ductility takes place at $f_s \cong 0.96$, and the alloy loses all ductility at f_s <0.95. Furthermore, the alloy begins to demonstrate tensile strength at $f_s \cong 0.87$. Between 0.87 and 0.95, the fraction solid has almost no effect on tensile stress, probably because at this stage there is sufficient liquid to allow for the grains to slide over one another, fully lubricated by the liquid films, as proposed by Lahaie and Bouchard [15]. Above $f_s \cong 0.95$, the tensile strength increases rapidly.

Based on the experiments performed in this study, a simple linear relationship can be proposed between temperature and maximum stress using a least-squares fit:

$$\sigma = -0.115T + 65.9 \qquad (1)$$

where σ is the maximum stress in *MPa*, and T is the temperature in *°C*. This equation is valid for the range of temperatures examined in this study (400 – 570°C), and for the strain rate of ~10^{-3}.

It can now be included in industrial thermal-mechanical simulations of magnesium alloys to improve the model predictions in the high temperature and semi-solid regimes. Furthermore, hot tearing predictions using current hot tearing criteria [11] would be improved since the tensile constitutive behavior in the temperature range critical to hot tearing is now known. Although the maximum semi-solid stress will certainly be a function of strain rate, the semi-solid strain rates during DC casting are very low. Thus, the error associated with using this relationship in DC casting models is small in comparison to the error associated with other variables, most importantly the fraction solid – temperature relationship, which industrially will vary in different regions of the casting.

Tensile properties of magnesium alloy AZ31 close to solidus temperature have been measured at a strain rate of $10^{-3}s^{-1}$. Based on these measurements, along with a microstructure and fracture analysis of tested specimens, the following conclusions can be drawn:

1. Both the maximum tensile strength and ductility of as-cast magnesium alloy AZ31 decrease steadily with temperature in the range of 400°C and 570°C.
2. The mechanical properties of AZ31 are predominantly ductile in the temperature range between 400°C and 514°C, and transitions towards exhibiting no significant ductility between 540°C and 570°C.
3. The microstructure and fracture surfaces examined showed evidence of increasing amounts of liquid present along the as-cast grain boundaries with increasing test temperature.
4. The magnesium alloy AZ31 has no ductility above 540°C, corresponding to $f_s \cong 0.95$, and cannot sustain tensile stresses above 560°C, corresponding to $f_s \cong 0.87$.

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Figure and Table Headings

Fig. 1: Optical micrograph of the initial grain structure of the as-cast AZ31 material used in this study.

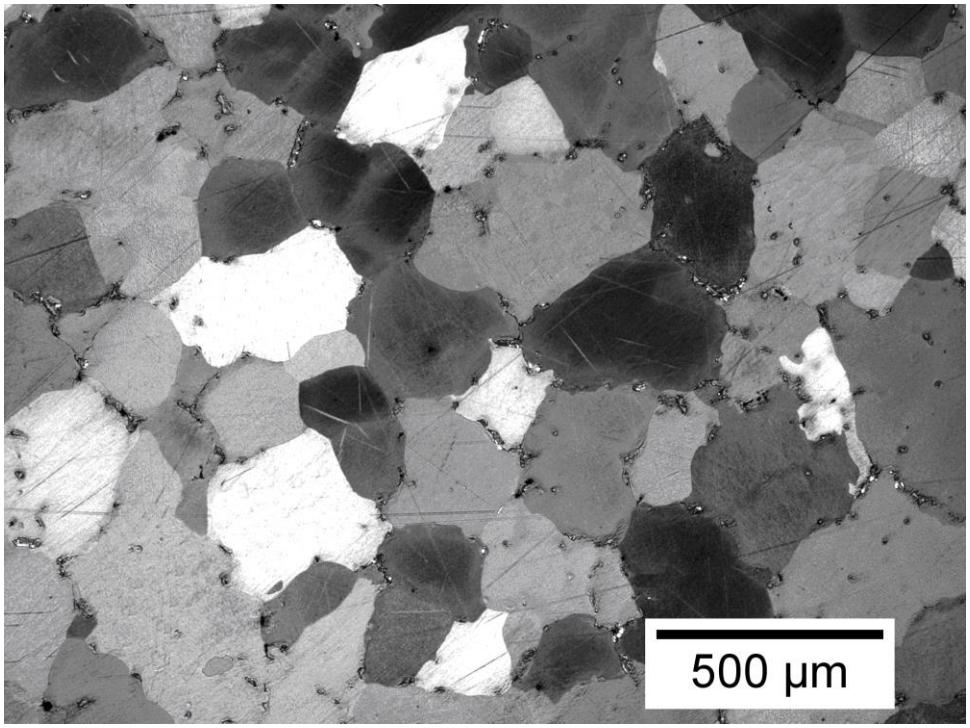
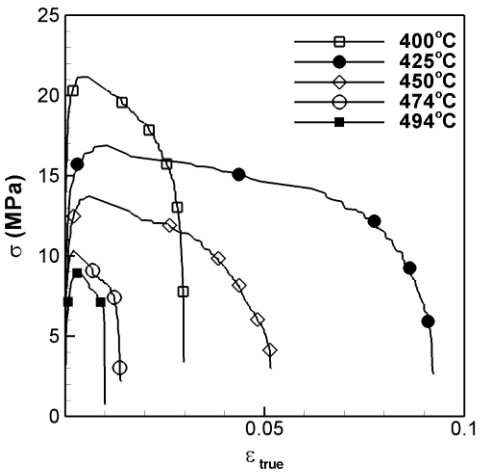
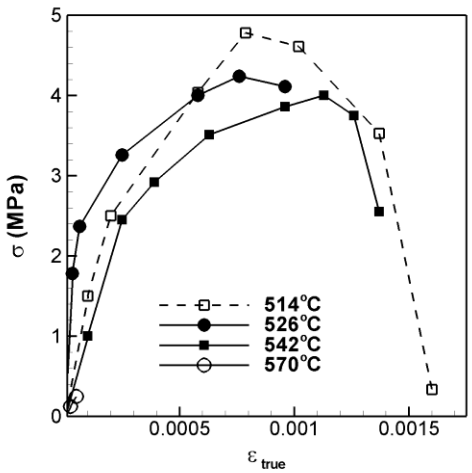


Fig. 2: True-stress versus true-strain results for as-cast AZ31 at (a) 402-494°C and (b) 514-570°C.



a)



b)

Fig. 3: Variation in tensile strength, and ductility as a function of temperature.

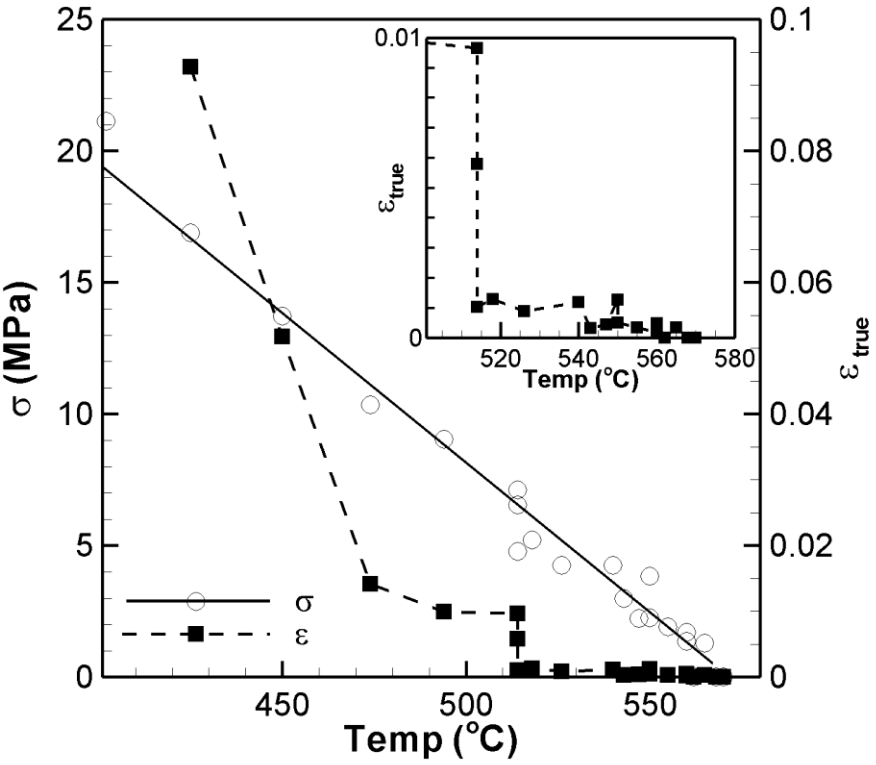


Fig. 4: Optical micrographs of as-cast AZ31 tensile specimens tested at (a) 474°C, (b) 526°C, and (c) 566°C.

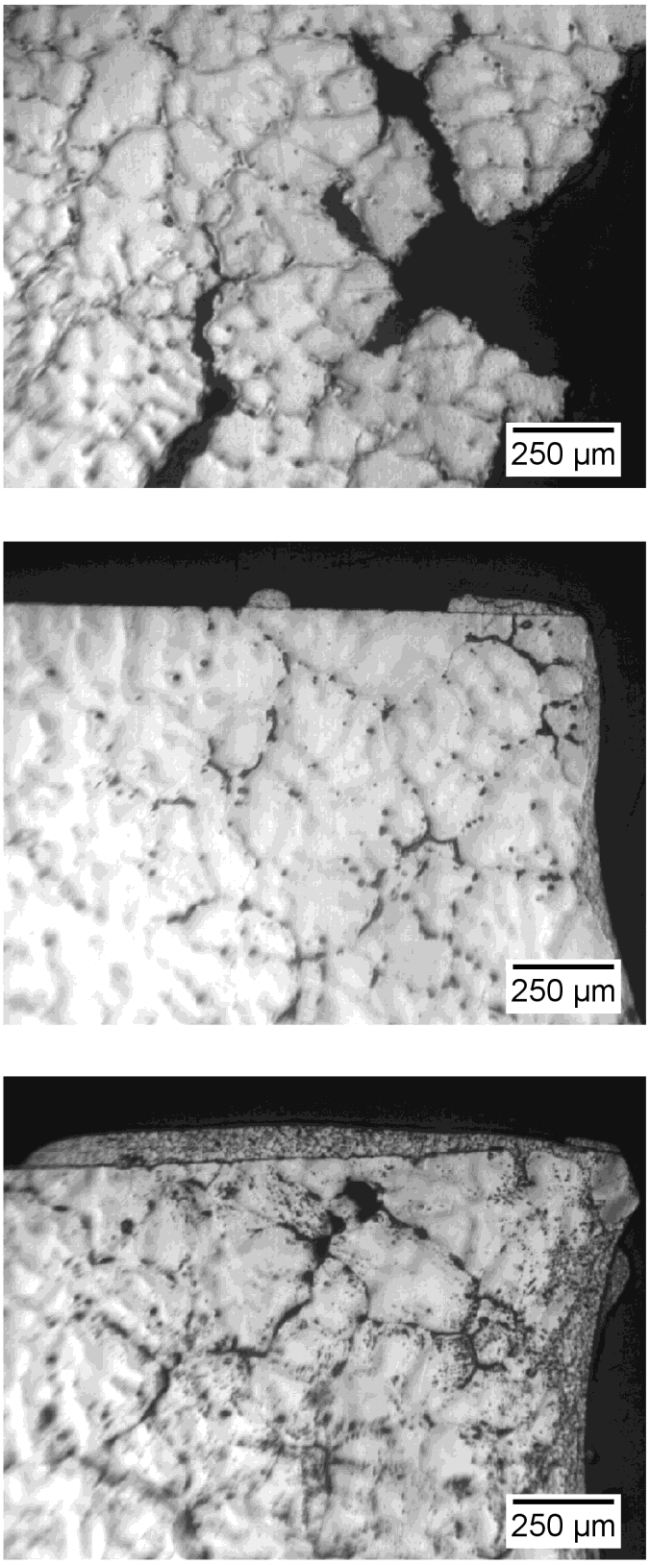


Fig. 5: SEM micrographs showing the fracture surface morphologies of specimens tested at (a) 474°C and (b) 514°C.

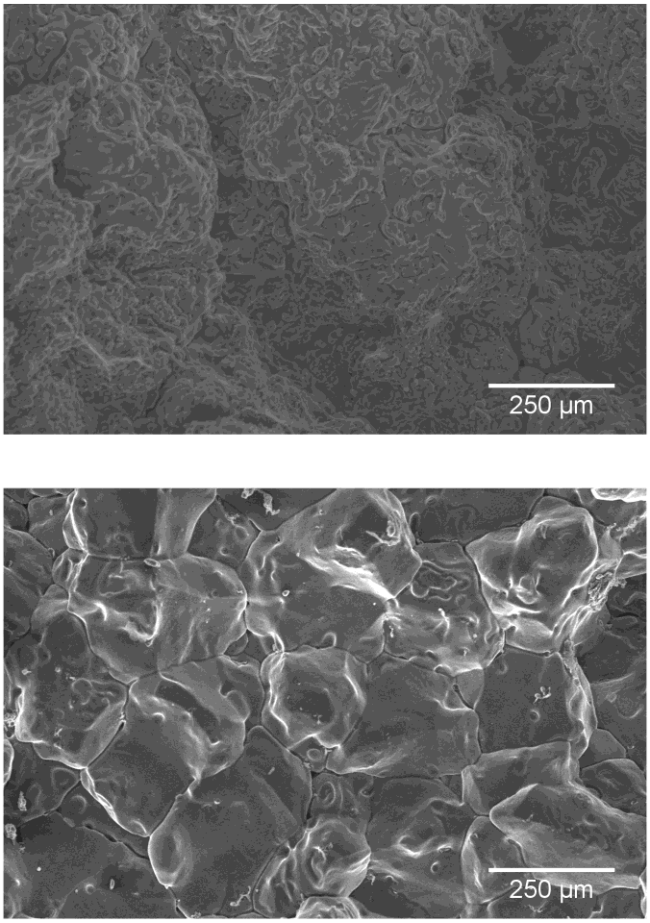


Table A: Variation in f_s with temperature for AZ31 after Lu [13].

f_s	Temp (°C)	f_s	Temp (°C)
1.0	460	0.90	562
0.98	504	0.85	572
0.95	545	0	630

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