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# Experimental investigation of the creep behavior of MgO at high pressures

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#### ABSTRACT

The high-temperature rheological behavior of polycrystalline periclase, MgO, has been investigated using the deformation-DIA on a synchrotron beamline at pressures up to 10 GPa. Significant experimental scatter in stress measurement illustrates current limitations of this technique. Although temperature and stress sensitivities are not well constrained, there is a clear dependence of creep rate on pressure. Based on our results, the creep rate of MgO depends on confining pressure with an activation volume of  $V^* \approx 2.4 \times 10^{-6} \text{ m}^3/\text{mol}$ . The grain-scale view of deformation processes reveals, as other D-DIA studies have, that subpopulations of grains, grouped by orientation, obey slightly different flow laws. The measurements also reveal that stress heterogeneity in the sample, whether caused by external conditions or processes internal to the sample itself, contribute a significant portion of the overall uncertainty in stress measurement.

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# 1. Introduction

The deformation behavior of MgO has long received attention in both materials science and geosciences. In the former, a wide range of applications of MgO in industry demand a thorough understanding of its mechanical behavior (e.g., Copley and Pask, 1965a, 1965b; Gorum et al., 1960). In the latter, the creep behavior of MgO has been investigated, for example, by Weaver and Paterson (1969), Paterson and Weaver (1970), Meade and Jeanloz (1988), and Sinogeikin and Bass (1999) in order to understand geodynamic processes occurring within Earth's interior, from deep-focus earthquakes to mantle convection. Although the emphasis as well as experimental conditions might be different, both kinds of studies share not only the same trunk but also many of the same branches.

A distinct feature of studies carried out in geosciences is the high-pressure environment since pressure is a critical thermodynamic parameter in describing any physical process occurring deep within Earth's interior. Paterson and Weaver (1970) conducted deformation experiments on polycrystalline MgO at temperatures from 25 to 750 °C at pressures up to 0.5 GPa using a gas-medium press; they quantified mainly the deformation behavior of MgO in the transition from the brittle to the ductile regime. Recently, Merkel et al. (2002) used a diamond anvil cell to deform MgO at room temperature at pressures up to 47 GPa; they focused on investigating texture development within specimens with increasing pressure.

The dependence of creep behavior of MgO on pressure at high temperatures has yet to be investigated, severely limiting our understanding of geological processes based on laboratory studies. To further define the high-temperature, high-pressure creep behavior of MgO, we have conducted a series of experiments using a deformation-DIA (D-DIA) apparatus at a synchrotron beamline. We have explored the rheological properties of our MgO samples at temperatures up to 1573 K and pressures up to 10 GPa. The focus of this study is quantification of the creep behavior of MgO at high temperatures and high pressures in order to provide a basis for applying flow laws obtained in the laboratory to processes occurring within Earth's interior.

## 2. Experimental details

## 2.1. Sample assembly and experiments

Experiments were carried out in the D-DIA apparatus (Wang et al., 2003) at the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory. Though technical features on conducting such deformation experiments with using D-DIA have been illustrated elsewhere (Li et al., 2004a, 2004b; Uchida et al., 2004; Chen et al., 2006), a few details relevant especially to this study are briefly elaborated here for clarity.

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**Fig. 1.** Sketch of sample assembly. Samples (MgO and olivine) were assembled with hard alumina pistons, machinable alumina plugs, a boron nitride sleeve, a graphite resistance heater, and a machinable alumina sleeve into a 6-mm edge length cubic pressure medium. Nickel discs were placed between neighboring pieces in the sample column as strain makers. The temperature during an experiment was in general measured using a W3% Re–W25% Re thermocouple embedded in one of the alumina pistons. Deformation of the olivine is the subject of another study (manuscript in preparation).

Each sample is assembled within a 6-mm edge length cubic pressure medium of either boron–epoxy resin or mullite. In general, experiments conducted in this study are aimed at quantifying creep behavior of two different minerals: olivine and MgO. Specifically, a cold-pressed MgO sample (0.8 mm length  $\times$  1.1 mm diameter) and a hot-pressed olivine sample (1.1 mm length  $\times$  1.1 mm diameter) were assembled with Al<sub>2</sub>O<sub>3</sub> pistons, a boron nitride sleeve, and a graphite resistance heater into the cubic pressure medium. Nickel discs located at the ends of the samples acted as strain makers, since most of the assembly parts are X-ray transparent. A sketch of the sample assembly is presented in Fig. 1. Starting powder size of cold-pressed MgO is  $\sim 4 \,\mu$ m and grain size of deformed MgO is  $\sim 8 \,\mu$ m. The microstructure of a deformed sample is shown in an optical micrograph in Fig. 2. The temperature during the experiments was either measured directly using a W3% Re–W25% Re thermocouple



Fig. 2. Transmitted plane light photomicrograph of deformed sample (San 96). The micrograph is from polished section cut normal to the long axes of the sample.



**Fig. 3.** Radiograph of sample column as taken periodically during deformation, viewed through the transparent anvils of the D-DIA. The lighter vertical stripe is a portion of the sample column exposed in the gap between anvils. Nickel disks are placed between column pieces as strain markers. The nickel capsule surrounding the olivine sample.

embedded in one of the  $Al_2O_3$  pistons or was estimated based on calibrated heater power versus temperature curves in assemblies prepared without a thermocouple.

At the start of an experiment, the cell was first pressurized hydrostatically to the desired level and then heated to run temperature. Next the sample was deformed in triaxial compression at constant pressure. Deformation resulted from pushing top and bottom anvils toward each other. During an experiment, a beam of white X-rays ( $50 \,\mu m \times 50 \,\mu m$  in size) passed through deforming sample, and diffraction patterns as well as X-ray radiographs of the deformation column were taken periodically. This information is used, as described next, in computing pressure, differential stress, and strain for each run.

# 2.2. Data acquisition and processing

## 2.2.1. Strain

In the synchrotron beamline, X-ray radiographs taken every  $\sim$ 15 min of the sample column are used to measure the change in length of a sample during a deformation experiment. A radiograph of a typical deforming sample is shown in Fig. 3. Such images make it possible to measure the change in sample length to within a few microns. A typical length versus elapsed time plot is shown in Fig. 4.

# 2.2.2. Pressure and differential stress

The state of elastic lattice strain in the sample is determined via X-ray diffraction measurements, and the state of stress is calculated based on non-isotropic elastic lattice strains within the sample (Singh, 1993; Singh et al., 1998; Weidner et al., 1994, 1998; Merkel, 2006). The four elements of the solid-state X-ray detector system are oriented such that diffracted X-rays from (hkl) planes whose normals lie either nearly parallel to (vertical in lab coordinates) or nearly perpendicular to (horizontal) the applied stress axis are detected simultaneously. We thus refer to the four elements as "vertical" and "horizontal" detectors. Diffraction patterns collected simultaneously on detectors aligned parallel to the compression axis (vertical detectors) and perpendicular to the compression axis (horizontal detectors) are compared in Fig. 5 for a sample of MgO. If we call  $d_V$  the spacing of planes whose Bragg reflections are detected by the vertical detectors and  $d_{\rm H}$  detected by the horizontal detectors, then the *d*-spacing corresponding to the mean stress



**Fig. 4.** Typical plot of sample length vs. elapsed time from a deformation experiment on one sample. In general, a run involves several steps including a change in temperature at a fixed pressure or a change in pressure at a given temperature.

(i.e., mean pressure P) is

$$d_{\rm P} = \frac{d_{\rm V} + 2d_{\rm H}}{3}.\tag{1}$$

Based on the analysis of *d*-spacings described above, mean stress as well as differential stress can be computed.

For a specific lattice plane (*hkl*), *P* was deduced from unit-cell volume:

$$V = \left[d_{\rm P}(h^2 + k^2 + l^2)^{1/2}\right]^3 \tag{2}$$

and Murnaghan's EOS (1937):

$$P = \frac{K_{0T}}{K'_{0}} \left[ \left( \frac{V_{0T}}{V} \right)^{K'_{0}} - 1 \right]$$
(3)

where  $K_0$  is bulk modulus at zero pressure and temperature,  $K'_0$  is the first pressure derivative of the bulk modulus,  $K_{0T}$  and  $V_{0T}$  are bulk modulus and unit-cell volume, respectively, at zero pressure



**Fig. 5.** Typical diffraction patterns from MgO collected simultaneously on the detectors aligned parallel to the compression axis (vertical detectors) and perpendicular to the compression laxis (horizontal detectors). For both,  $2\theta = 6.8^{\circ}$ . The relative shift of diffraction peaks between vertical and horizontal detectors is a measure of the differential stress applied to the specimen.

and temperature of *T*. Both  $K_{0T}$  and  $V_{0T}$  are functions of *T* with

$$K_{0T} = K_0 + \left(\frac{\partial K_0}{\partial T}\right)_{\rm P} \Delta T \tag{4}$$

and

$$V_{0T} = V_0 \, \exp\left(\alpha_0(T - T_0) + \frac{1}{2}\alpha_1(T^2 - T_0^2)\right)$$
(5)

where  $(\partial K_0/\partial T)_P$  is the temperature derivative at constant pressure and  $V_0$  is unit-cell volume at room temperature  $(T_0)$ ,  $\alpha_0$  and  $\alpha_1$  are volume coefficients of thermal expansion at zero pressure. All parameters used in calculations are taken from reported works (Anderson et al., 1991; Xia and Xiao, 1993; Kushwah and Shanker, 1996).

Deformation of a sample is composed of both elastic and plastic components. The elastic part in the deforming sample results in lattice strain that can be used to determine the stress state in the sample with use of Hoose's law as

$$\sigma_i = c_{ij}\varepsilon_j \tag{6}$$

where  $\sigma_i$  and  $\varepsilon_j$  are the principal stress and strain components and  $c_{ij}$  is the elastic stiffness tensor. In our analysis, the influence of temperature and pressure on the stiffness tensor at experimental conditions is corrected using following formula:

$$c_{ij}(P,T) = c_{ij}(P=0,T=T_0) + \left(\frac{\partial c_{ij}}{\partial P}\right)\Delta P + \left(\frac{\partial c_{ij}}{\partial T}\right)\Delta T$$
(7)

In computation, elastic parameters  $c_{ij}$ ,  $\partial c_{ij}/\partial P$ , and  $\partial c_{ij}/\partial T$  are taken from Spetzler (1970).

Further reasoning by Singh (1993) and Singh et al. (1998) can be used to compute differential stress ( $\sigma_{\rm diff}$ ) in a deforming sample from lattice strain:

$$\sigma_{\rm diff} = \left(\frac{\alpha}{2G_{\rm R}} + \frac{1-\alpha}{2G_{\rm V}}\right)^{-1} \frac{d_{\rm H} - d_{\rm V}}{d_{\rm P}} \tag{8}$$

where  $G_R$  is the aggregate shear modulus calculated under the *Reuss* condition (iso-stress),  $G_V$  is the aggregate shear modulus under the *Voigt* condition (iso-strain), and  $\alpha$  is a weighting parameter  $(0 \le \alpha \le 1; \alpha = 1 \text{ and } 0 \text{ represent the$ *Reuss*and*Voigt*conditions, respectively).

For present purposes, we compute differential stresses assuming that the material is in the iso-stress (*Reuss*) state by letting  $\alpha = 1$  in Eq. (8). For MgO as cubic material,  $G_R$  can be further expressed with the elastic compliance,  $S_{ij}$ , as

$$G_{\rm R} = \frac{1}{2} \left[ S_{11} - S_{12} - 3\left(S_{11} - S_{12} - \frac{1}{2}S_{44}\right) \Gamma_{h\ kl} \right]^{-1}$$
(9)

with

$$\Gamma_{hkl} = \frac{h^2 k^2 + k^2 l^2 + l^2 h^2}{\left(h^2 + k^2 + l^2\right)^2} \tag{10}$$

Crystalline materials, including MgO, typically exhibit plastic anisotropy, meaning that the flow stress (akin to  $\sigma_{diff}$  in Eq. (8)) depends on the orientation of the crystallographic axes relative to the deviatoric stress tensor. For a polycrystalline material with grains randomly oriented, not all grains yield at the same differential stress. The result is that local stresses begin to redistribute from the moment of plastic yield, so that whole solid departs, often in a very short amount of strain, from the *Reuss* state (Weidner et al., 2004; Chen et al., 2006; Merkel and Yagi, 2006; Weidner and Li, 2006). Although the issue mentioned is a concern, it is still a good approximation to use the *Reuss* bound in analyzing stress in deformation experiments. Chen et al. (2006) analyzed the weighting parameter ( $\alpha$ ) in Eq. (8) for both un-deformed and deformed

| Table 1                             |  |
|-------------------------------------|--|
| Experimental conditions and results |  |

| Exp                   | P(GPa) | T(K) | $\dot{\varepsilon}$ (s <sup>-1</sup> ) | ε (%) | $\sigma_{(111)}$ (MPa) | $\sigma_{(200)}({ m MPa}$ |
|-----------------------|--------|------|--|-------|------------------------|---------------------------|
| 67 <sup>a,b</sup>     | 3.1    | 1373 | $2.5\times10^{-5}$                     | 0.27  | 300                    | 133                       |
| 68(1) <sup>a, c</sup> | 2.4    | 1373 | $3.0	imes10^{-5}$                      | 0.35  | 380                    | 168                       |
| 68(2) <sup>a, c</sup> | 2.2    | 1473 | $3.1 	imes 10^{-5}$                    | 0.20  | 300                    | 149                       |
| 68(3) <sup>a, c</sup> | 1.5    | 1573 | $3.2 	imes 10^{-5}$                    | 0.17  | 160                    | 96                        |
| 69(1) <sup>a,b</sup>  | 4.4    | 1473 | $4.2 	imes 10^{-5}$                    | 0.29  | -                      | 204                       |
| 69(2) <sup>a,b</sup>  | 4.0    | 1573 | $6.5 	imes 10^{-5}$                    | 0.41  | 320                    | 168                       |
| 76 <sup>c, d</sup>    | 10.0   | 1350 | $4.0	imes10^{-5}$                      | 0.38  | 440                    | 344                       |
| 96 <sup>b,d</sup>     | 8.8    | 1373 | $1.4 	imes 10^{-5}$                    | 0.32  | 300                    | 149                       |

Pressure medium was boron epoxy resin.

<sup>b</sup> Temperature for the run was estimated based on pre-calibrated heater power vs. temperature curves.

<sup>d</sup> Pressure medium was mullite.

MgO polycrystalline samples. For samples without notable plastic deformation,  $\alpha = 1$  predicts exactly the *Reuss* state. While with significant plastic deformation,  $\alpha \approx 1.1$  indicates that the stress state is still near the boundary of *Reuss* state. Thus, for the present purposes of identifying the effect of pressure on the flow of polycrystalline MgO, the approach we took for analyzing stress in our deformation experiments is well-justified.

# 3. Results and discussion

Five samples of polycrystalline MgO were tested in the D-DIA at constant displacement rates ranging from 1.4 to  $6.5 \times 10^{-5}$  s<sup>-1</sup> over true axial strains of 17-41% at temperatures of 1350-1573 K and pressures of 1.5-10 GPa. Two of the experiments consisted of multiple temperature steps, with deformation halted between steps. Although the stress measurements were rather noisy, as will be seen shortly, after a brief initial transient, they did not exhibit clear strain hardening or strain weakening; thus, we characterized each deformation step with a single value of differential stress. Results are listed in Table 1. Creep curves from one run that involved temperature stepping are shown in Fig. 6. For most of the runs, we were able to discern with confidence diffraction peaks only at the MgO (111) and (200) reflections; for runs san68 and san69, we were also able to measure peaks for (222) and (400). Differential stresses  $\sigma(hkl)$  in Table 1 are given for only the two strongest reflections.



Fig. 6. Differential stress and pressure vs. strain for run san68. Differential stresses were determined from four different (hkl) reflections as shown. Three separate deformation steps were taken on sample, labeled (1)-(3). Between each step deformation was stopped, temperature raised to the new value, and deformation restarted.

#### Table 2

Best fit parameters for (200) reflection to Eq. (11)

| $\log A ({\rm MPa^{-3}s^{-1}})$               | $-8.1\pm1.7$ |
|---|--------------|
| n   | ≡3           |
| Q (kJ/mol)                                    | $72\pm50$    |
| $V^*$ (×10 <sup>-6</sup> m <sup>3</sup> /mol) | $2.4\pm0.9$  |

Measurement uncertainties are significant and a serious concern to us. Absolute temperatures are known to roughly  $\pm 50$  to  $\pm$ 75 K in the hot zone along the length of the sample, which is a typical pathology of development work (such as this) involving small sample volumes and cell configurations that are altered slightly run to run (as ours are) in search of an ideal design. Uncertainties in differential stress are even more substantial. Understanding the causes of stress uncertainty is a subject of ongoing development by our group and others; an analysis of the problem, except for some observations reported below regarding stress heterogeneity, is beyond the scope of this paper. For the present, we call stress uncertainty the range of stress covering a loosely defined "substantial portion" of observed scatter. The (200) reflection invariably produces the strongest peak; over the course of three to four measurements of a deformation step, the stress uncertainty is roughly  $\pm 0.04$  GPa. For the (111) reflection, whose diffraction peak has a much lower intensity, the uncertainty is typically  $\pm 0.15$  GPa (Fig. 5). For comparison, the uncertainty in measured pressure is small, on the order of  $\sim$ 10%.

MgO can be easily deformed at high temperatures by crystalline slip since both of its sets of slip systems,  $\{110\}$   $\langle 110 \rangle$  and  $\{001\}$ (110), become active at T > 873 K, thus providing five independent slip systems required by the von Mises-Taylor criterion for homogeneous crystalline ductility (Copley and Pask, 1965a; Paterson and Weaver, 1970). We therefore analyze our results in terms of a power law equation of the form:

$$\dot{\varepsilon}(\sigma, T, P) = A\sigma^n \exp\left(-\frac{Q + PV_*}{RT}\right) \tag{11}$$

where  $\dot{\varepsilon}$  is axial strain rate,  $\sigma$  is differential stress. T is temperature. and *P* is confining pressure. Our objective is to identify the four material parameters: *A*, the pre-exponential; *n*, the stress exponent; Q, the activation energy; and  $V^*$ , the activation volume.

The narrow range of experimental strain rates combined with measurement noise (especially for the (111) reflection) does not allow reasonable constraint on the values of all four of these parameters. We therefore assert that n = 3, a reasonable value for high-temperature dislocation glide or climb-controlled dislocation creep of MgO (Wilshire, 1995). Since data from the (111) reflection carry comparably bigger uncertainty, only the data from the (200) reflection are fitted to obtain the values for A, Q, and  $V^*$  that are reported in Table 2.

Experiments were carried out at several temperatures. In Fig. 7, the effect of temperature on strain rate is explored in a plot of strain rate versus inverse temperature. The best fit value for the activation energy of Q = 72 kJ/mol is identical to that obtained by Weidner et al. (1994). However, this value for activation energy is much lower than those expected for creep mechanisms involving self-diffusion, which are usually around 200 kJ/mol (Frost and Ashby, 1982). Thus, we suggest that deformation of our samples is dominated by a process such as dislocation glide or possibly dislocation-accommodated grain boundary sliding.

The effect of pressure on creep behavior of MgO is presented in Fig. 8 as a semi-log plot of differential stress versus pressure, normalized to a common temperature and strain rate using those best fit parameters and Eq. (11). The total uncertainty in stress (i.e., the scatter as described above) and temperature has been combined into a single error bar for each point by using Eq. (11) to convert an

Sample was assembled with thermocouple for monitoring temperature.



**Fig. 7.** Arrhenius plot of strain rate vs. inverse temperature for all runs. Data obtained at different pressures and stresses were normalized to a pressure of 5 GPa and differential stress of 60 MPa using Eq. (11) and the flow parameters in Table 2. The straight line is the least-square best fit to data points.

uncertainty in temperature to an uncertainty in stress. Despite the magnitude of the overall uncertainty, the positive trend of differential stress with pressure is visible. The best fit for activation volume with n = 3 is  $\sim 2.4 \times 10^{-6}$  m<sup>3</sup>/mol. This value is similar to but somewhat smaller than that for deformation reported by Weidner et al. (1994) of  $4-7 \times 10^{-6}$  m<sup>3</sup>/mol based on a comparison of their results on the high pressure strength of MgO to those of Hulse et al. (1963) obtained at lower pressures.

Our experimental results yield stresses determined from the (111) reflection that are about two times higher than those obtained from the (200) reflection (see Table 1). The cause of the discrepancy in flow stress obtained from analyses of different sets of diffraction peaks remains an open issue, which could possibly have several sources such as elastic anisotropy at experimental conditions, the validity of Singh's formula for analyzing stress in the



**Fig. 8.** Semi-log plot of differential stress vs. confining pressure based on eight deformation steps on five samples based on measurements made with (200) reflection. Actual temperatures and strain rates are given in Table 1; for plotting purposes, stresses have been normalized to 1473 K and  $3 \times 10^{-5}$  s<sup>-1</sup> using Eq. (11) and the flow parameters in Table 2. The straight line is the least-square best fit to the (200) data with *n* constrained to 3.

plastic deformation regime, the accuracy of measured elastic moduli at high pressures and temperatures, as well as the geometric relationship among active slip planes and analyzed planes.

Values obtained for differential stress from different diffraction planes (e.g., (200) and (111)) are generally scattered (see Fig. 6), yet values of stress determined from parallel lattice reflections (e.g., (200) and (400)) appear to be reasonably well correlated. In the current configuration, diffraction peaks other than (111) and (200) were too weak to measure except in runs san68 and san69. Stress measurements on two pairs of parallel reflections - (200) versus (400) and (111) versus (222) - are plotted in Fig. 6 for San68. The correlation between (200) and (400) is quite clear; all but 1 of the 12 pairs of points are within 0.1 GPa of one another, while the range of scatter was estimated above to be 0.08 GPa (i.e.,  $\pm 0.04$  GPa). For the pair of reflections (111) and (222), the scatter of individual reflections is  $0.3 \text{ GPa} (\pm 0.15 \text{ GPa})$ , with only 4 of the 12 pairs of points differing by more than this amount. Determinations of stress from parallel sets of reflections are independent measurements of the state of the same volume of material at the same moment in time. Discrepancies between measurements must owe their existence to uncertainties introduced by the measurement system (geometry of sample, beam, collimator, and detectors, as well as details associated with the multi-channel analyzer and peak-fitting software). Therefore, in Fig. 8, we infer that a substantial portion of the scatter in the values of stress resulting from physical changes to the diffracted volume between measurements. Temperature fluctuations have a small effect on elastic constant of MgO (Spetzler, 1970) and thus may account for some of the scatter in Fig. 8. Temperature fluctuations also directly affect the strength of a sample, and temperature gradients give rise to spatial gradients in strength. In addition, the differential stress will vary spatially within the sample due to the finite strength of the solid confining medium. In addition, due to the relatively small length-to-diameter ratio of a sample, relatively large dead zones develop due to friction between the sample and the pistons. The volume sampled by the X-rays undoubtedly changes from one exposure to the next due to dynamic effects that occur during deformation and due to slight shifts in the position of the sample with respect to the X-ray beam.

#### 4. Conclusions

These experiments mark some of the first attempts to carry out high-strain compressional deformation experiments in a D-DIA apparatus. Significant uncertainties in the values of stress and of temperature impair our ability to measure flow parameters with sufficient accuracy to discern flow mechanism. Nevertheless, the measurements indicate a positive pressure dependence of the flow stress with  $V^* \approx 2.4 \times 10^{-6} \text{ m}^3/\text{mol}$ . This work involves not only the development of a piece of hardware, the D-DIA, but also the formulation of an approach to handling a new type of input to deformation science, namely, the measurement of stress and strain rate at the grain scale. As has been observed previously in similar experiments on plastic flow of polycrystalline samples, a grain scale redistribution of differential stress occurs among different orientation populations of grains. Interpretation of the different flow laws obtained from analysis of different sets of diffraction peaks is a topic of ongoing study. We also see with rather good resolution that, apart from the stress redistribution just mentioned, the stress distribution is spatially significantly non-uniform within a sample. This observation is quite independent of the system (e.g., slits, collimators, and detectors) that measures the intensities of diffracted X-rays. It is not clear at this point if the cause is extrinsic (i.e., imposed by non-uniform tractions on the outer surfaces of the sample from pistons and pressure medium) or if it is inherent to deformation processes within the polycrystalline sample itself.

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