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Phase Transitions Between β and γ $(Mg,Fe)_2SiO_4$ in the Earth's Mantle: Mechanisms and Rheological Implications

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The mechanisms of the phase transformations between the spinel (γ) and modified spinel (β) polymorphs of Mg_2SiO_4 have been studied experimentally between 15 and 20 gigapascals and 800° to 950°C. The γ to β transformation occurs by a shear mechanism, whereas the β to γ transformation involves grain-boundary nucleation and interface-controlled growth. These contrasting mechanisms are a consequence of the number of independent slip systems that are available in the respective crystal structures. This result leads to the prediction that in subduction zones and perhaps also rising plumes in the Earth's mantle, the γ to β transformation should be accompanied by a transient reduction in strength.

High-pressure phase transformations are likely to play an important role in several aspects of the dynamic behavior of the Earth's mantle. A detailed understanding of the mechanisms of these transformations is essential for evaluating transformation kinetics in the mantle as well as the effects of phase transitions on mantle rheology and convection. In addition, rheological changes associated with phase transitions may be involved in the triggering of deep focus earthquakes. In the context of rheological changes, the phenomenon known as "transformation plasticity" has considerable potential importance. Metals and ceramics often exhibit a reduction in mechanical strength as they undergo phase transitions (*1, 2*). Unusually high strains can develop under low differential stress, especially when the temperature is varied in order to cycle the conditions back and forth across the phase transition.

Transformation plasticity may be important during mineralogical phase transformations in the Earth's interior (*3–8*). Phase boundaries in the mantle between, for example, the polymorphs of $(Mg,Fe)_2SiO_4$ are obvious locations where this phenomenon might occur. Such phase boundaries are believed to coincide with the seismic discontinuities located at depths of 410 km, possibly 520 km, and 660 km. Theoretical studies have demonstrated that transformation plasticity at such phase boundaries would cause mechanical decoupling and favor layered convection (*8*). In addition, deep focus earthquakes may result from shear instabilities initiated by localized rheological change, associated with phase transformations in subducting slabs (*9–13*). We have studied the mechanisms of the β to γ and γ to β transformations in Mg_2SiO_4 at high pressure to understand how these transformations occur and whether they are likely to cause transformation plasticity in the Earth's mantle.

Different mechanisms of transformation plasticity will presumably operate, depending on the mechanism of the phase transition (*1, 2, 4, 14*). Martensitic or shear transformations are particularly likely to result in a reduction in strength. Such transformations occur by the migration on certain slip planes of partial dislocations, each of which changes the stacking sequence and leaves behind a layer of the product phase. In addition to changing the crystal structure, the migration of the partial dislocations also results in plastic deformation. The driving force for the migration of the partial dislocations and the resulting deformation is the change in Gibbs free energy associated with the phase transformation. Thus, while the phase transformation is in progress, the externally applied stress required to deform the material should be much smaller than normal.

It has been demonstrated experimentally that the olivine to spinel transformation can occur by a martensitic-like mechanism involving the synchroshear of cations (*15, 16*). However, because the differential stress required to produce a small degree of transformation by this mechanism is in excess of 1 GPa (*16, 17*), the martensitic-like mechanism is unlikely to be of major importance in the Earth's mantle. Theoretically, phase transformations between the high-pressure polymorphs of $(Mg,Fe)_2SiO_4$, modified spinel (β) and spinel (γ), may also occur by a shear mechanism (*18, 19*). For the β to γ transformation, the mechanism involves the migration of $1/2[\bar{1}01](010)$ partial dislocations, whereas the γ to β transformation occurs by the migration of $1/4[1\bar{1}2](110)$ partial dislocations (*18*). In either case, partial transformation would result in an increase in stacking disorder [either on $(010)_\beta$ or $(110)_\gamma$, respectively] and, at advanced degrees of transformation, lamellar intergrowths of the two phases, crystallographically oriented with $[001]_\beta // [001]_\gamma$ and $[010]_\beta // [1\bar{1}0]_\gamma$. Similar stacking faults can also form during crystal growth (*20*), making detailed microstructural stud-

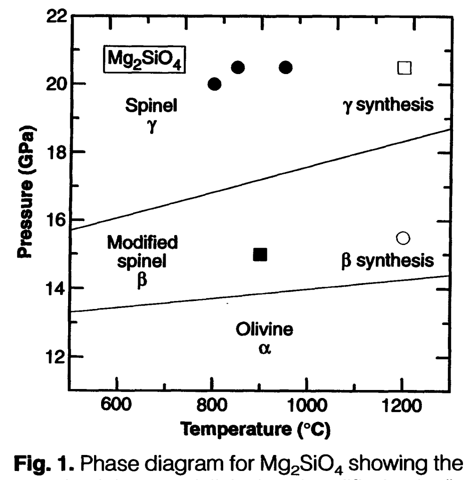

Fig. 1. Phase diagram for Mg_2SiO_4 showing the stability fields of α (olivine), β (modified spinel), and γ (spinel) (*30*). The synthesis conditions for β and γ are shown (open symbols) in addition to the experimental run conditions for the β to γ (filled circles) and γ to β (filled square) transformations.

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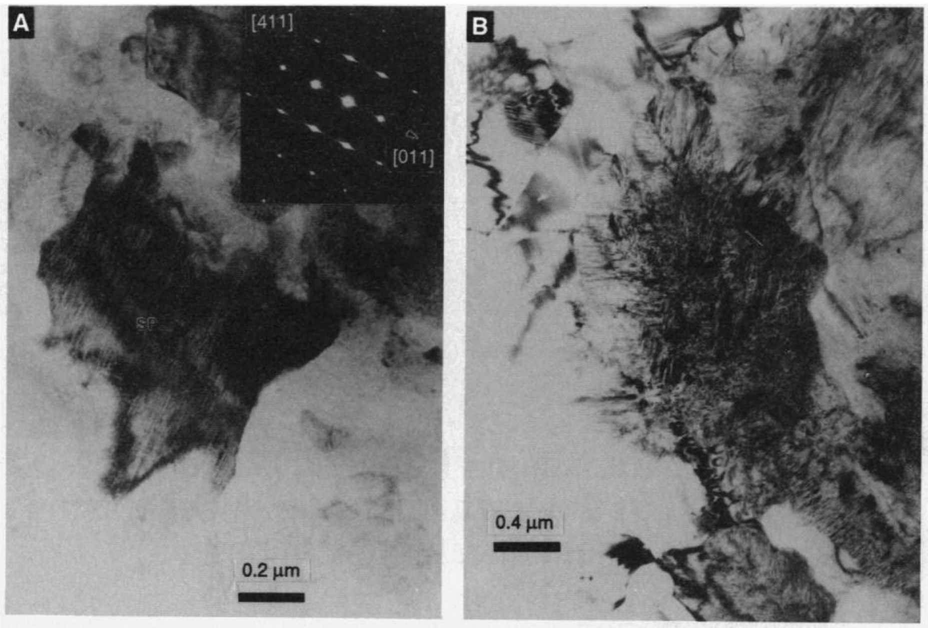
ies of reactant phases in experimental studies essential for the interpretation of transformation microstructures.

Previous work has shown that γ-Mg_2SiO_4 transforms to β by the shear mechanism at 15 GPa and 900°C (*11, 21*). However, there is possible ambiguity in these results because the observed stacking disorder, on which the conclusion was based, could have originated during metastable growth of γ-spinel from forsterite rather than during transformation of γ to β (*21, 22*).

To resolve the question of the exact mechanisms of the γ to β and β to γ transformations in Mg_2SiO_4, we performed high-pressure experiments using a multianvil apparatus (*23*). Starting material for the γ to β transformation was synthesized by reacting forsterite powder to a fully densified γ-Mg_2SiO_4 aggregate at 20.5 GPa and 1200°C for 2.0 hours (Fig. 1). For the reverse transformation, β-Mg_2SiO_4 was synthesized from forsterite powder by reaction at 15.5 GPa and 1200°C for 2.0 hours (Fig. 1). In both cases, the hot-pressed polycrystalline starting materials had grain sizes of 1 to 2 μm and low densities of stacking faults on {110} in γ and (010) in β. These faults appear to have formed by growth processes during synthesis. Following the high-pressure experiments, the samples were characterized by x-ray microdiffraction, optical microscopy, and transmission electron microscopy (TEM) (*24*).

To study the γ to β transformation, we partially reacted γ-Mg_2SiO_4 within the β stability field at 15 GPa and 900°C for 45 min (Fig. 1). Pressure was increased to 15 GPa at room temperature before the sample was heated (*25*). After the experiment, x-ray microdiffraction showed that the partially reacted sample still consisted primarily of γ. However, TEM studies showed that many γ grains had developed a striated microstructure, and pronounced streaking was present in electron diffraction patterns parallel to <110>* (Fig. 2A). These phenomena are indicative of a significant increase in the degree of stacking disorder in the γ-spinel in comparison with the starting material. This stacking disorder results from the development of lamellae, a few unit cells wide, of β within γ, as observed by high-resolution TEM (*21*). The lamellae have the orientation relation expected for a martensitic-like transformation, that is, $[001]_\beta//[001]_\gamma$ and $[010]_\beta//[110]_\gamma$. Some γ grains exhibit a complex microstructure (Fig. 2B), and diffraction patterns show streaking of the diffraction maxima in all <110> directions (Fig. 2C), which indicates that stacking faults have developed on all equivalent (110) planes. The striated microstructure is identical to that reported previously (*11*) within grains of γ-Mg_2SiO_4.



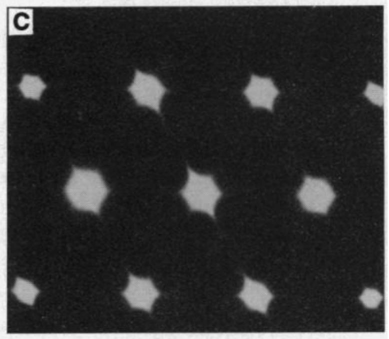


Fig. 2. (**A**) Bright field TEM image of a γ-Mg_2SiO_4 grain showing a striated microstructure parallel to (110) after reaction at 15 GPa and 900°C for 45 min. (**Inset**) Streaking of diffraction maxima, which is indicative of stacking disorder, is more pronounced than in γ from the starting material. The stacking disorder is caused by the presence of stacking faults with the local stacking sequence of β-Mg_2SiO_4, produced during the transformation. (**B**) Bright field TEM image of γ from the same experiment showing the development of an extremely complex microstructure. The mottled contrast is the result of the development of a large number of stacking faults on {110} planes as a result of partial shear transformation to β. (**C**) Electron diffraction pattern from the [111] zone axis of a γ grain from (B) showing strong streaking of diffraction maxima parallel to <110>*, which indicates the presence of stacking disorder on all sets of (110) planes.

The increase in stacking fault density in γ can only have resulted from partial transformation to β by the shear mechanism. These results demonstrate conclusively that the shear mechanism dominates during the γ to β transformation under these conditions, in agreement with our earlier results (*11, 21*). The shear mechanism also appears to operate during the γ to β transformation in $Mg_{1.8}Fe_{0.2}SiO_4$ at 13 GPa and 1000°C (*26*).

To study the β to γ transformation, we synthesized β-Mg_2SiO_4 and subsequently partially transformed it to γ in a single experiment. After synthesizing β at 1200°C and 15.5 GPa, we reduced the temperature to 600°C over a period of several minutes and then increased the pressure to the desired value in the γ stability field (20.0 to 20.5 GPa) (Fig. 1). The temperature was then increased to between 800° and 950°C for 60 to 300 min to produce partial transformation to γ-spinel.

Experiments at 800° to 950°C at about 20 GPa all produced some transformation to γ. In general, there was no increase in the density of the (010) stacking faults in β. This result shows that the transformation to γ did not occur by the shear mechanism. Instead, grains of γ nucleated at grain boundaries and grain boundary triple junctions, and many of the γ grains were crystallographically oriented with respect to adjacent β grains (Fig. 3A). The most common orientation observed is $[001]_\beta//[001]_\gamma$ and $[010]_\beta//[110]_\gamma$ (Fig. 3B), which is that expected for the shear transformation. However, the sharp, but irregular, nonglissile interphase boundaries and the lack of lamellar intergrowths between the two phases show that the transformation has occurred by topotactic, coherent nucleation of γ on β followed by interface-controlled growth, which involves short-range diffusion of atoms across the interphase boundary. Some grains of γ show no special orientation with respect to β and have nucleated incoherently.

The above results demonstrate that contrasting mechanisms operate during the β-γ

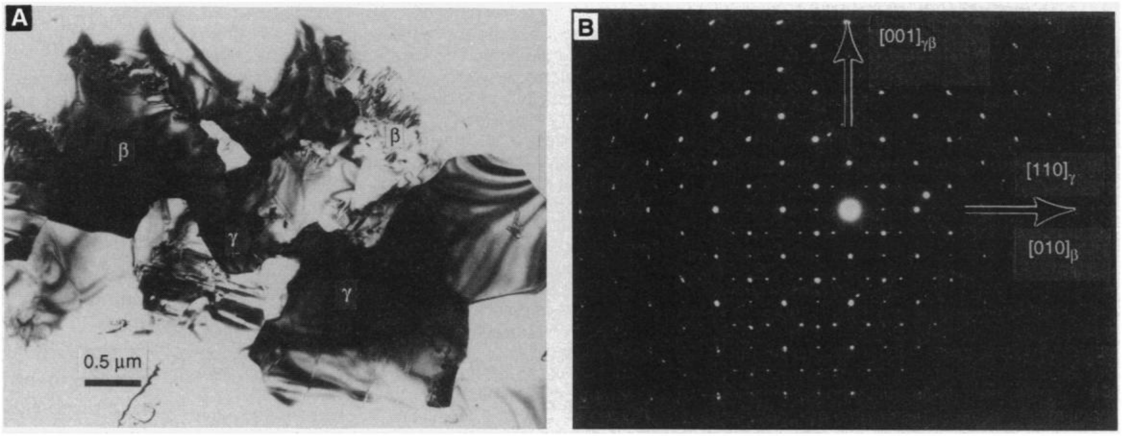


Fig. 3. (**A**) Bright field TEM image of a mixed region of γ- and β-Mg_2SiO_4 after partial transformation of β at 20.5 GPa and 950°C for 60 min. The γ has nucleated topotactically on β and the interface between the two phases is sharp. There is no evidence of a striated microstructure in either phase, indicating that the transformation has occurred by coherent nucleation and interface-controlled growth. (**B**) Electron diffraction pattern showing the crystallographic orientation relation $[001]_\beta//[001]_\gamma$ and $[010]_\beta//[110]_\gamma$ between grains of β and γ shown in (A). This is the same orientation relation as expected for the shear-like transformation, but the absence of streaking in the diffraction pattern shows that the two phases are not intergrown on a fine scale.

and γ-β transformations at similar temperatures. Although γ transforms to β by the shear mechanism at 900°C and 15 GPa, the transformation of β to γ at 850° to 900°C and 20 GPa primarily involves interface-controlled growth. During the latter transformation, both coherent and incoherent nucleation have been observed.

The difference in transformation mechanisms can be explained by von Mises criterion. A result of the shear transformation mechanism is that individual grains must deform and undergo significant shear strain as a result of the migration of the partial dislocations. According to von Mises criterion, such homogeneous strain can only be accommodated in a polycrystalline aggregate if the constituent crystals can deform on five independent slip systems (*27*). In β, there is only one slip system, $[\bar{1}01](010)$, which results in transformation to γ; thus, the deformation of a particular grain of β-phase cannot be accommodated by the deformation of the surrounding grains because there are insufficient independent slip systems. This explains the absence of transformation of β to γ by the shear mechanism. In contrast, the slip system in γ that results in transformation to β, $[1\bar{1}2](110)$, constitutes five independent slip systems (partly as a result of the cubic symmetry of this phase) (*27*). Thus, von Mises criterion is fulfilled, and the transformation of γ to β by the shear mechanism can occur readily in a polycrystalline aggregate, in accord with our experimental results. It can often be observed directly that several slip systems were activated within single γ grains, as described above (Fig. 2, B and C).

It has been observed that polycrystalline olivine (α) only transforms to γ by a martensitic-like mechanism involving synchroshear when the differential stress is very high (>1 GPa) and only to a limited extent (*16*). This result can also be explained by a lack of independent slip systems in olivine, because the transformation of α to γ by the synchroshear mechanism can only be achieved by glide on one particular slip system. Although glide on this single slip system should be very easy in the spinel stability field, the resulting deformation must be accommodated by glide on other slip systems, which are not related to the shear transformation. Glide on these other slip systems does not occur readily, thus explaining the high shear stress required to partially transform olivine by the martensitic mechanism (*16, 17*).

The above results suggest that transformation plasticity associated with the shear transformation between the polymorphs of $Mg_{1.8}Fe_{0.2}SiO_4$ is only likely to occur during the γ to β transformation. With increasing depth in the interior of subducting slabs, the expected sequence of $Mg_{1.8}Fe_{0.2}SiO_4$ polymorph stability fields is $\alpha \rightarrow \alpha + \gamma \rightarrow \alpha + \beta \rightarrow \beta \rightarrow \beta + \gamma \rightarrow \gamma$ (*28*). The third stage of this sequence involves the transformation of γ to β, but metastable γ might form in the β stability field and subsequently transform back to β (*11, 21, 29*). Although it has been argued that transformational faulting occurs during the α to β transformation in $(Mg,Fe)_2SiO_4$ (*10*), the occurrence of deep focus earthquakes by this mechanism should be even more likely as a result of the α to γ transformation because of its relatively large volume change and high latent heat production (*13*). Our results suggest that if early formed γ transforms to β by the shear mechanism during transformation faulting, transformation plasticity could enhance the faulting process. Our data also indicate that the mechanism of the β to γ transformation (that is, nucleation and growth) is unlikely to cause deep focus earthquakes by an enhancement of plasticity.

In the transition zone of the Earth's mantle, the γ to β transformation will occur primarily in rising plumes. Transformation between the two phases may also occur if material fluctuates periodically across the β-γ phase boundary as a result of instability in mantle flow (*5*). The applicability of our experimental results to reaction mechanisms at the β-γ phase boundary in regions of the mantle remote from cold subduction zones, where transformation probably occurs under near equilibrium conditions at temperatures of 1500° to 1600°C (*30*), is currently uncertain. If the shear mechanism operates during the γ to β phase transformation under such conditions, there could be important consequences for the rheology of rising plumes and perhaps for mechanical decoupling across the β-γ phase boundary.

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23. The pressure assembly for the experiments was a MgO octahedron, with a 10-mm edge length, containing a $LaCrO_3$ heater (D. Canil, *Phys. Earth Planet. Inter.*, in press). It was compressed with Toshiba F-grade WC anvils with 5-mm truncations. Sample pressure was calibrated at 1200°C by reversing phase transitions in SiO_2 (quartz-coesite and coesite-stishovite) and Mg_2SiO_4 (α-β and β-γ). The uncertainty in the quoted pressures is ±1 GPa. Temperature was monitored with a W3%Re-W25%Re thermocouple without correction for the effect of pressure on the thermocouple. The Mg_2SiO_4 samples were contained in 1.6-mm-diameter Pt capsules, and before the experiments, samples were dried for several hours at 230°C in a vacuum oven.
24. Doubly polished petrographic thin sections suitable for TEM studies were prepared from the experimental charges. Selected areas of the sample were demounted from the thin section and prepared for TEM examination by conventional ion beam milling. After carbon coating, all the samples were studied in a JEOL 2000FX analytical transmission electron microscope operating at 200 kV.
25. Ideally, the synthesis of the γ-Mg_2SiO_4 starting material and the subsequent partial transformation to β would have been accomplished in a single experiment, as described for the β to γ transformation. This procedure was not adopted because it would have involved reducing the sample pressure during the experiment; the sample pressure during decompression in the multi-anvil apparatus has not been calibrated, and it is unlikely that this could be done reliably. Pressurizing the γ-Mg_2SiO_4 at high temperature to reach the β stability field (as was done for the β to γ transformation) would have risked transformation of the sample into forsterite during compression through the α-Mg_2SiO_4 stability field.
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Arabidopsis ABA Response Gene *ABI1*: Features of a Calcium-Modulated Protein Phosphatase

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The *Arabidopsis* *ABI1* locus is essential for a wide spectrum of abscisic acid (ABA) responses throughout plant development. Here, *ABI1* was shown to regulate stomatal aperture in leaves and mitotic activity in root meristems. The *ABI1* gene was cloned and predicted to encode a signaling protein. Although its carboxyl-terminal domain is related to serine-threonine phosphatase 2C, the ABI1 protein has a unique amino-terminal extension containing an EF hand calcium-binding site. These results suggest that the ABI1 protein is a Ca^{2+}-modulated phosphatase and functions to integrate ABA and Ca^{2+} signals with phosphorylation-dependent response pathways.

The plant hormone ABA regulates diverse physiological processes including seed maturation and dormancy, as well as the adaptation of vegetative tissues to environmental stresses (*1, 2*). The responses of this hormone range from the rapid alteration of ion fluxes in stomata (*3*) to the induction of specific changes in gene expression (*1, 4*). However, the knowledge of the molecular network that couples ABA perception to an integrated set of physiological responses remains fragmentary. Several ABA-regulated genes (*4*) and corresponding transcription factors (*5*) have been identified. Saturable ABA-binding sites have also been reported (*6*), but these proteins have not been further characterized. On the other hand, ABA response mutants offer a propitious means to isolate signaling elements and, furthermore, provide insight into their biological functions on the basis of their associated mutant phenotypes. This approach has led to the cloning of the maize *VP1* (*7*) and *Arabidopsis ABI3* (*8*) loci, whose molecular actions appear to target seed-specific developmental processes. Critical to our understanding of the early events in hormone perception and transduction, however, are probably mutants that exhibit pleiotropic phenotypes. In this regard, mutations in the *Arabidopsis* abscisic acid insensitive 1 (*ABI1*) gene (*9*) appear to have unraveled such an early signaling component. The *abi1* mutations severely impair a wide spectrum of ABA responses including reduced seed dormancy, excessive water loss, and abnormal drought rhizogenesis (*9, 10*). Moreover, the expression of several ABA- or stress-induced genes has been found to be greatly diminished in the mutants (*11*). To elucidate the regulatory role of the *ABI1* gene product in these diverse developmental, tissue-specific, and adaptive response pathways, we cloned the locus by chromosome walking.

As an aid to localize the gene by plant transformation and to interpret the molecular function of the protein later, we first analyzed two phenotypic traits of the *abi1-1* mutant. One characteristic phenotype of *abi1-1* mutant (*9*) seedlings is their ability to achieve root development in the presence of inhibitory concentrations of applied ABA (Fig. 1, A and B). Indeed, *abi1-1* root meristems exhibited active cell division in the presence of added ABA, as revealed by the nuclear incorporation of bromodeoxyuridine (BrdU) (*12*), which traces passage through the S phase of the mitotic cycle (Fig. 1, D and F), and the observation of occasional mitotic figures (*13*). In contrast, no BrdU incorporation was detectable in the wild type treated with ABA (Fig. 1, C and E). These results indicate the involvement of the *ABI1* gene product in mediating the ABA inhibition of mitotic activity in the root meristem. Another trait of the *abi1-1* mutant is that the mean aperture of the stomatal pores present on the abaxial (lower) surface of freshly detached rosette leaves in *abi1-1* (8.6 ± 3.3 μm, $n = 115$) is twice as large as that in the wild type (4.1 ± 2.1 μm, $n = 110$) (*14*) (Fig. 1, G and H). Altered stomatal regulation is consistent with the wilty phenotype displayed by the *abi1-1* mutant (*9*).

To clone the *ABI1* locus, restriction fragment length polymorphism (RFLP) linkage analysis was used to delineate a small genomic region that contains the target gene. Plants selected for recombination between *abi1-1* and flanking morphological markers *cer2* and *cer4* on chromosome 4 were used to analyze the linkage of the *abi1-1* mutation with existing RFLP markers assigned to this region (*15*). One of these (PG11), found to map about 0.5 to 1.0 centimorgan from the *ABI1* locus and to represent the closest available RFLP marker (*16*), was used to initiate a chromosome walk. Genomic clones identified from two yeast artificial chromosome (YAC) libraries of *Arabidopsis* (*17*) were used to assemble in successive steps an overlapping set of 13 YACs extending approximately

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