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RADIATION EFFECTS ON Si:P AND SiGe:P ALLOYS: ELECTROPHYSICAL AND MECHANICAL CHARACTERISTICS

The scope of the study is to investigate the microstructure, electrophysical properties, and amplitude-dependent behavior of internal friction and shear modulus in phosphorus-doped monocrystalline n-Si and n-Si+0.5 at.% Ge alloy, both in their initial state and after irradiation with ^{60}Co-gamma photons. Additionally, the regularities of changes in critical strain amplitude will be examined. Metallographic study showed that the dislocation density on the (111) crystallographic planes of the samples range from 10^4 to 5×10^4 cm^{-2}. Irradiation by ^{60}Co gamma photons does not cause significant changes in the density and distribution of dislocations. It was established that in test n-Si sample gamma photons irradiation increases the electrical resistivity by 15-20%, reduces the concentration of current carriers by 8-10 times, and increases their mobility by1,5-times. Such changes were relatively weakly detected in the tested SiGe alloy. Under conditions of torsional oscillation frequencies of 0.5-5.0 Hz and strain amplitude of 10^{-5} to 5×10^{-3} at room temperature, an increase in critical strain amplitude by 15-20% was determined in irradiated samples. The effect of radiation hardening was more clearly observed in the n-Si+0.5 at.% Ge alloy. The possible mechanisms of changes of physical-mechanical characteristics have been analyzed. Established regularities in the changes of electrical and physical-mechanical characteristics in Si-Ge alloys irradiated by gamma photons can be applied in the development of radiation-stable, high-efficiency semiconductor devices.

Keywords: Gamma-photon irradiation; radiation hardening; dislocation density; critical strain amplitude; torsional oscillations

1. Introduction

To date, the accumulated scientific and technological information in the field of semiconductor SiGe alloys production technologies and physical properties research clearly indicates their high potential for applications in radiation technologies and microelectronics products. Unlike pure Si, SiGe alloys are expected to exhibit significant and specific changes in defect structures and structure-sensitive physical-mechanical properties. These changes are primarily due to the strain fields localized around isovalent Ge atoms in the crystalline lattice of SiGe alloys, which practically guide the conditions for the generation, interaction, and mobility of thermal, radiation-induced, and deformation defects across a wide range of Ge concentrations.

Local compositional fluctuations in the SiGe alloy induce a long-range strain field and the dynamic formation of a solute atmosphere around dislocations, thereby suppressing dislocation activity and revealing the material's strengthening [1]. On the other hand, germanium doping leads to the formation of small size oxygen precipitates in the structure of SiGe alloys, in the dislocation core areas. These precipitates act as strong obstacles to dislocation motion. Under such conditions, the velocity of dislocations and their displacement are reduced, thereby improving the substrate's warpage during processing and device manufacturing [2].

In paper [3], the alloy hardening with shallow indents into the thinner SiGe epitaxial layers is discussed. The authors suggest that geometrically necessary dislocations, formed at the dc/β-tin phase separation boundary, may play a significant role in the observed hardening effect. Additionally, it is expected that bond strengthening will become evident during the indentation process. Relatively strong Si-Ge bonds within the structure of SiGe alloys primarily contribute to the enhancement of microhardness and elastic modulus, both in thin epitaxial layers and bulk samples. Nanoindentation of substrates of Si and SiGe alloys, grown by the Czochralski method, has demonstrated a significant increase in the alloy's microhardness and elastic

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modulus [4]. The authors suggest that this effect is driven by a germanium-induced metallic phase transformation (Si-I to Si-II phase) during the indentation process.

At a frequency of 1 Hz for torsional oscillations over a wide temperature range, monocrystalline Si-Ge alloys grown by the Czochralski method showed a slow decrease in both the activation energy of dislocation-related relaxation processes and the dynamic shear modulus as the germanium content was increased from 0 to 2.5 at.% [5]. The possibility of enhancement of the shear modulus and the activation energy of relaxation processes by approximately 5% through boron doping and thermal annealing is shown in papers [6,7]. The amplitude dependence of internal friction and shear modulus in Si-Ge alloys with 0-2 at.% germanium has been studied at temperatures of 20°C and 600°C [8]. The reduction in the first and second critical strain amplitudes and the shear modulus has been observed with increasing germanium concentration. In Si and Si+0.5 at.% Ge alloy irradiated by 12 MeV electrons of fluence of 10^{13} cm^{-2}, a relaxation process stipulated by the motion of radiation-induced VO defects was observed. A slight decrease in the activation energy of the process in the Si-Ge alloy and complete suppression of the process after 1 hr. thermal annealing at 450°C have been observed [9]. The effect of irradiation by ^{60}Co gamma photons on the temperature spectra of internal friction and shear modulus in monocrystalline SiGe alloys, as well as their changes in the strain amplitude range of 10^{-5} to 10^{-3} at room temperature, has been studied [10]. An increase in dislocation-related internal friction characteristics and critical strain amplitude has been observed in samples irradiated by ^{60}Co gamma photons. Strengthening effects were observed in the internal friction spectra of phosphorus-doped Si-Ge alloys irradiated by ^{60}Co gamma photons [11].

The objective of this study is to examine the microstructure, electrophysical properties, and amplitude-dependent behavior of internal friction and shear modulus in phosphorus-doped monocrystalline n-Si and n-Si+0.5 at.% Ge alloys in both initial and ^{60}Co gamma-photons irradiated states. Gamma photons are used in our study due to their high penetrating ability, which is essential for accurately diagnosing real structural changes throughout the volume of the samples.

2. Materials and methods

Bulk monocrystalline samples were grown by the Czochralski method on the EQ-SKJ-50CZ system device in flowing Ar gas atmosphere. Double-sided polished plates were prepared by standard methods of cutting on a diamond disc, mechanical grinding, and polishing. For metallographic and electrophysical studies, samples with dimensions of 5×5×0.5 mm^3 were used. Internal friction measurements were conducted on rectangular parallelepiped samples with dimensions of 1×1×30 mm^3. The number of repetitions for each measurement was 10, with the experimental errors reported as 5% for internal friction measurements and 1% for shear modulus measurements. The microstructure was examined using an NMM-80RF/TRF optical microscope. Electrophysical characteristics were determined in the constant magnetic field of 0.5 Tesla induction on the Ecopia HMS-3000 device by Hall effect measurements. Measurements of the logarithmic decrement and frequency of torsional oscillations were conducted on the vacuum laboratory device (Fig. 1), based on the direct pendulum, with strain amplitude intervals ranging from 5×10^{-5} to 5×10^{-3} and oscillation frequencies from 0.5 to 5.0 Hz. Loading on the sample is 50 g/mm^2.

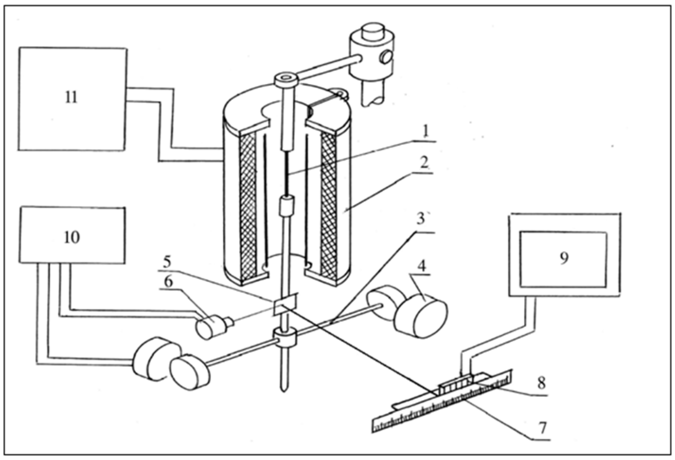

Fig. 1. The Scheme of measuring device of the internal friction of torsional oscillations. 1. sample; 2. furnace; 3. horizontal component of the direct pendulum with a variable load; 4. pair of Electromagnets; 5. reflective Mirror; 6. light source; 7. transparent scale; 8. photodiode converter; 9.counter; 10. controller; 11. Thermoregulator

During torsional oscillations, the rotation angle of the samples is considered as the torsional deformation, from which the strain amplitude is calculated using the following relationship:

$$\varepsilon = \frac{r \cdot L}{R \cdot l} \qquad [1]$$

where r is the radius of the circle inscribed on the cross-section of the sample, l – the length of the sample, R – the distance from the sample to the registration scale, L – the deviation of the light beam on the registration scale reflected from the mirror fixed on the axis of pendulum.

3. Results of experimental research

The metallographic study of the experimental n-Si and n-SiGe samples revealed that dislocation etch pits on the (111) planes are uniformly distributed, with their density ranging from 10^4 to 10^5 cm^{-2}. Irradiation with gamma photons practically does not change the character and density of dislocations distribution on (111) planes. The level of phosphorus doping and the increased dislocation density up to 10^5 cm^{-2} , formed during crystal growth, significantly reduce the mobility of electric current carriers and result in a decrease in total electrical resistivity (TABLE 1).

TABLE 1

Electrophysical Characteristics of Test Samples at Room Temperature

Test samples	Electrical resistivity, Ohm·cm	Carriers' concentration, cm^{-3}	Carriers mobility, $cm^2/V \cdot s$	Dislocation density, cm^{-2}
n-Si:P initial state	9.0	5×10^{14}	1380	1×10^{4}
n-Si:P after irradiation	15.5	3×10^{14}	1360	$(1\text{-}3)\times10^{4}$
n-Si+0.5 at.% Ge initial state	15.0	3×10^{14}	1400	$(3\text{-}5)\times10^{4}$
n-Si+0.5 at.% Ge after irradiation	23.0	2×10^{14}	1370	5×10^{4}

In the gamma-irradiated state, the electrical resistivity of both samples increases, and the concentration of electric current carriers decreases. The mobility of electrons in n-Si sharply improves, while in the n-SiGe sample, the increase in electron mobility is weaker after gamma photons irradiation. This difference is possibly due to the formation of additional scattering centers for electric current carriers on the radiation defects associated with germanium atoms and the localized strain field near Ge impurity atoms in the n-SiGe alloy. It should be noted that the metallographic and electrophysical characteristics for both the direct and opposite planes of the test irradiated samples are practically identical, which can be attributed to their small thickness (0.5-0.8 mm). The results of the measurements of the electrophysical characteristics is presented in TABLE 1.

In the initial state, the amplitude dependence of internal friction $Q^{-1}(\varepsilon)$ in the monocrystalline Si sample is characterized by a strain amplitude at which the linear increase in internal friction intensity begins (Fig. 2,1). Under conditions of reduced values of high strain amplitude, no background elevation is observed, as confirmed by the coincidence of the ascending and descending branches of the $Q^{-1}(\varepsilon)$ dependence. Gamma photons-irradiation significantly increases the critical strain amplitude (Fig. 2,1'). In the irradiated state, a decrease in the background intensity of oscillation energy is observed across a wide range of strain amplitude, indicating an increase in the concentration of dislocation anchoring centers within the radiation defect-impurity atmosphere.

At amplitudes higher than the critical strain amplitude, a noticeable decrease in the slope of the $Q^{-1}(\varepsilon)$ dependence is clearly evident. This is possible due to the formation of nonuniformly distributed anchoring centers on dislocations during the irradiation process, which reduces the contribution of relatively long oscillating dislocation segments to the linearly increasing internal friction intensity. Under these conditions, the ascending and descending branches of the internal friction curve are nearly identical. This indicates that in samples irradiated by gamma photons, no evidence of microplastic deformation is observed in the strain amplitude range of 10^{-5} to 10^{-3} at room temperature.

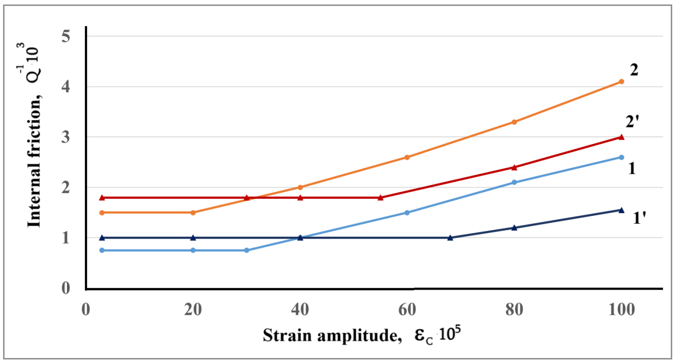

Fig. 2. Dependence of the internal friction on the strain amplitude the test samples: 1 – Si, initial state; 2 – Si+0.5 at.% Ge initial state; 1' – Si, after irradiation with ^{60}Co gamma photons; 2' – Si+0.5 at.% Ge, after irradiation with ^{60}Co gamma photons

The $Q^{-1}(\varepsilon)$ dependence of the initial monocrystalline Si+0.5 at.% Ge alloy is also characterized by a single critical amplitude within the deformation range of 10^{-5} to 10^{-3} (Fig. 2,2). At low strain amplitude values, the internal friction background intensity is higher compared to silicon, mainly due to the localized strain field around the germanium atoms. They result in increased mobility of point defects that interact with dislocations. Such changes lead to a reduction in the critical strain amplitude in the tested SiGe alloy. The weakening of dislocation anchoring is clearly shown in the more pronounced increase of the internal friction background intensity at higher oscillation amplitudes, compared to the $Q^{-1}(\varepsilon)$ behavior of silicon in the same range of oscillation amplitudes. On the $Q^{-1}(\varepsilon)$ dependence of the SiGe sample irradiated by gamma photons, there is a pronounced increase in the critical strain amplitude (by 2.8) and a weak increase in the background internal friction intensity at higher strain amplitudes. We propose that irradiation with gamma photons resulted in an increased concentration of radiation-induced defects in the dislocations atmosphere and the dislocations cores, leading to a decrease in dislocation mobility. The observed changes in the $Q^{-1}(\varepsilon)$ dependence for both Si and Si+0.5 at.% Ge samples confirm the occurrence of radiation-induced strengthening of their structure. It should be noted that the ascending and descending branches of the $Q^{-1}(\varepsilon)$ graphs for both materials irradiated by gamma photons are identical, indicating that no microplasticity is observed in the 10^{-5} to 10^{-3} strain amplitude interval.

The impact of germanium doping and gamma photon irradiation is clearly evident in the amplitude dependence of the frequency squared, proportional to the dynamic shear modulus (Fig. 3).

It shows a nearly linear decrease in the function $f^2(\varepsilon)$ from the critical point of oscillatory deformation, with a more pronounced effect observed in the initial Si:P sample (Fig. 3.1). Relatively low critical strain amplitude and a significant reduction in the frequency squared (f^2) are observed in the Si+0.5 at.% Ge:P sample (Fig. 3.2). This effect is likely due to the increased mobility of defects within the alloy structure, influenced by the localized strain field near the germanium atoms. In both cases,

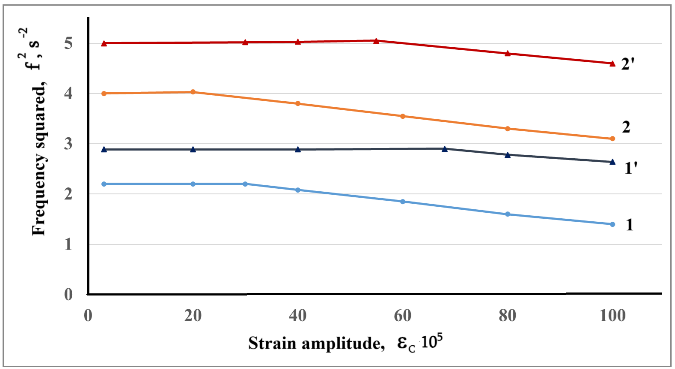

Fig. 3. Dependence of the shear modulus on the strain amplitude of the test samples: 1 – Si, initial state; 2 – Si+0.5 at.% Ge initial state; 1' – Si, after irradiation with ^{60}Co gamma photons; 2' – Si+0.5 at.% Ge, after irradiation with ^{60}Co gamma photons

changes in the $f^2(\varepsilon)$ dependencies are repeated within the 10^{-5} to 10^{-3} strain amplitude interval. Gamma photon irradiation induces radiation hardening in both test materials. The frequency squared shows a multiple increase across the entire strain amplitude range. Additionally, there is a significant increase in the critical strain amplitude for both irradiated materials. This effect is attributed to the enhanced bonding of the oscillating dislocation segments, under impact of radiation-induced defects distributed in the dislocation cores and defect-impurity atmosphere surrounding them. The majority of radiation defects are located within the volume of the materials. These defects may contribute to a reduction in the shear modulus; however, they have minimal impact on the critical strain amplitude.

Thermal instability of primary radiation defects, their tendency to formation of complexes and transformations in the structure of complexes affect the distribution and concentration of anchoring centers on dislocations. This factor plays a significant role in determining the changes in dislocation mobility within the wide range of isochronal annealing (Fig. 4). As expected, in the initial state, the Si:P and Si+0.5 at.% Ge:P samples exhibit only a slight linear increase in the internal friction background during isochronal annealing between 20°C and 600°C. Compared to n-Si, the critical strain amplitude values of SiGe alloys are lower, primarily due to elastic stresses localized around the germanium atoms. These stresses result in a local weakening of interatomic bonding forces.

The n-Si sample irradiated by gamma photons exhibits a 2.5 times increase in the critical strain amplitude. A gradual increase in isochronal annealing temperature up to 200°C results in an increase in ε_c, which is then followed by its sharp decrease up to 400°C. Further increases in the isochronal annealing temperature result in only a slight increase in the critical strain amplitude. A gradual increase in ε_c within the temperature range of 20-200°C, indicating a rise in the concentration of anchoring centers on dislocations, may result from the dissociation of radiation centers (PV), Ci-Cs, and Si_i complexes formed in the volume of the material, as well as the redistribution of their constituent components in the dislocation cores and in the impurity-defect atmosphere around dislocations.

The critical strain amplitude (ε_c) of the n-SiGe alloy irradiated by gamma photons increases by three times. A noticeable rise in ε_c is observed up to 150°C during isochronal annealing, with only a slight increase observed up to 200°C. Beyond this point, further temperature increase leads to a gradual decrease in ε_c up to 400°C. In the temperature range of 400-600°C, ε_c shows only a sight increasing trend. In the n-SiGe alloy, ε_c increases linearly up to isochronous annealing temperatures of 150-200°C. This behavior is attributed to the reduction in the annealing temperature of radiation-induced VO defects (A-centers), influenced by the presence of germanium atoms in nearest neighbor coordination to these defects. It is known [12] that germanium impurity atoms are effective traps for mobile VO defects in SiGe crystals. The decrease in the values of ε_c observed in the test samples within the temperature range of 250-300°C during isochronous annealing is stipulated by the reduction in the concentration of VO defects both in the vicinity of the dislocations and in the surrounding atmosphere. A further increase in the isochronous annealing temperature leads to an increase in the concentration of electrically neutral VO_2 complexes, which can brake dislocations and tend to increase the critical strain amplitude in the isochronal annealing temperature range of 400-600°C.

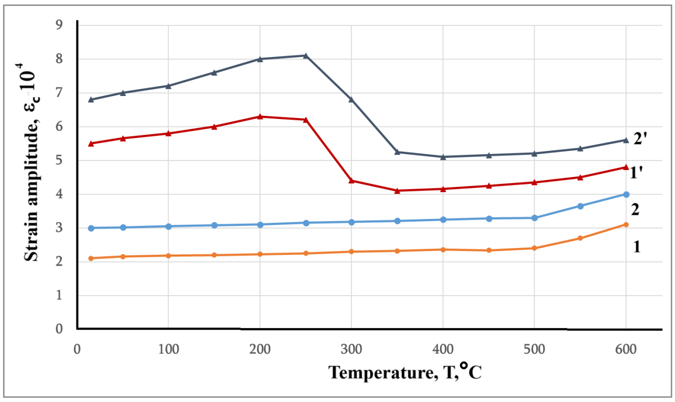

Fig. 4. Critical strain amplitude vs the isochronal annealing temperature: 1 – Si, initial state; 2 – Si+0.5 at.% Ge initial state; 1' – Si, after irradiation with ^{60}Co gamma photons; 2' – Si+0.5 at.% Ge, after irradiation with ^{60}Co gamma photons

The effect of isochronous annealing in vacuum (10^{-4} Torr) on the square of the torsional oscillation frequency in Si:P and Si+0.5 at.% Ge:P monocrystals was investigated. In the initial state, the frequency squared of both test samples remains nearly constant. A linear but weak increase is observed only within the temperature range of 400-600°C, similar to the behavior of the critical strain amplitude in the non-irradiated state. The samples irradiated by gamma photons exhibit non-monotonic variations in the frequency squared over the temperature range of 20-600°C during isochronous annealing (Fig. 5).

In particular, in the case of Si:P, there is a non-linear increase in the frequency squared, which reaches a maximum at a temperature of 300°C, after that it sharply decreases to a temperature of 450°C, where its magnitude is 1.5 times lower than the value at room temperature (Fig. 5). At elevated temperatures, a weak

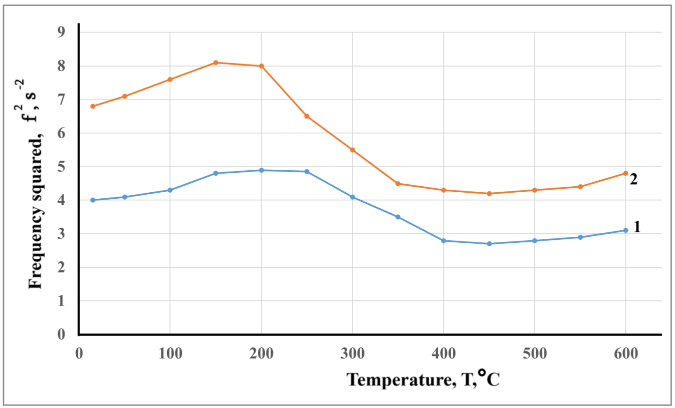

Fig. 5. Effect of isochronal annealing temperature on the frequency squared of samples irradiated by gamma photons. 1 – Si:P, f_o = 2 Hz; 2 – Si+0.5 at.%Ge:P , f_o = 2.6 Hz

increase in the frequency squared is observed, which is possibly caused by the diffusive redistribution of defects of thermal and radiation origin in the volume of the samples in the vicinity of dislocations and their additional blocking.

The frequency squared in the Si+0.5 at.% Ge:P alloy irradiated by gamma photons is characterized by a practically similar temperature change (Fig. 5). In the alloy, the frequency squared reaches a maximum value at 250°C, after which it undergoes a significant decrease, continuing until the temperature reaches up to 400-450°C. At these temperatures, the frequency squared is 1.3 times lower than its value measured at room temperature. Non-monotonic variations of the frequency squared in both materials are attributed to structural transformations of radiation-induced defects. The starting temperatures of the reduction of the maximum values of the frequency squared coincide with the starting temperatures of the dissociation in the radiation-induced VO defects in n-Si and n-SiGe alloys [13]. In the range of 20-250°C, the non-linear increase in the frequency squared is likely due to the rise in the concentration of VO defects in defect – impurity atmosphere around the dislocations. One of the sources of the increased concentration of VO defects might be the association of vacancy (V), released due to the dissociation of radiation-induced E-centers (PV), with interstitial oxygen atoms (Oi).

The weak increase in the frequency squared at elevated temperatures may be attributed to the formation of VO_2 and more complex V_mV_n defects, which contribute to enhanced dislocation blocking in the 400-600°C range during isochronal annealing.

The results of the study clearly show that the internal friction method provides valuable insights into the structural state of semiconductor silicon profiled products formed during the mechanical, chemical, and thermal processing. Specifically, it enables the determination of the energetic characteristics of motion of dislocations formed during processing, the identification of phase transformations, and the evaluation of activation parameters for defects of thermal, mechanical, technological, and radiation origin. This is important for the fabrication of Si and SiGe profiled samples with controlled structures and defect profiles, ensuring their suitability for application in semiconductor devices.

4. Conclusion

A significant increase in the critical strain amplitude and shear modulus, proportional to the frequency squared, was observed in n-Si and n-SiGe alloys under gamma-photon irradiation with a fluence of 10^{13} cm^{-2}. This radiation-induced hardening is assumed to result from the enhanced braking of dislocations within an atmosphere enriched with radiation defects.

Non-monotonic changes in the critical strain amplitude and dynamic shear modulus of gamma-photon-irradiated n-Si:P and n-Si+0.5 at.% Ge:P monocrystals are stipulated by gradual transformations in the structure of radiation defects during the isochronal annealing process.

For an in-depth analysis of the effects of gamma-photon irradiation, it is essential to study n-Si and n-SiGe samples under conditions of varying germanium and phosphorus concentrations, as well as different gamma-photon fluences, across a wide range of isochronal annealing.

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