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**INVESTIGATION ON THE LATTICE SITE LOCATION OF THE EXCESS
ARSENIC ATOMS IN GaAs LAYERS GROWN BY LOW TEMPERATURE
MOLECULAR BEAM EPITAXY**

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AN INVESTIGATION ON THE LATTICE SITE LOCATION OF THE EXCESS ARSENIC ATOMS IN GaAs LAYERS GROWN BY LOW TEMPERATURE MOLECULAR BEAM EPITAXY.

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ABSTRACT

We have measured the excess As atoms present in GaAs layers grown by molecular beam epitaxy at low substrate temperatures using particle induced x-ray emission technique. The amount of excess As atoms in layers grown by MBE at 200°C were found to be $\sim 4 \times 10^{20} \text{ cm}^{-2}$. Subsequent annealing of the layers under As overpressure at 600°C did not result in any substantial As loss. However, transmission electron microscopy revealed that As precipitates (2-5 nm in diameter) were present in the annealed layers. The lattice location of the excess As atoms in the as grown layers was investigated by ion channeling methods. Angular scans were performed in the $\langle 110 \rangle$ axis of the crystal. Our results strongly suggest that a large fraction of these excess As atoms are located in an interstitial position close to an As row. These As "interstitials" are located at a site slightly displaced from the tetrahedral site in a diamond cubic lattice. No interstitial As signal is observed in the annealed layers.

INTRODUCTION

GaAs thin films grown by Molecular Beam Epitaxy (MBE) at low growth temperature, T_g ($< 400^\circ\text{C}$) have been subject to many investigations recently [1-10]. Murotani *et al.* [11] first reported the development of this material in 1978 and predicted it to be a useful buffer materials for GaAs field effect transistors (FET). Smith *et al.* [1] have successfully used the low temperature MBE (LTMBE) GaAs as buffer layers for the fabrication of GaAs FET's and showed that the LTMBE-GaAs layer eliminates backgating between the devices. In addition to its great technological potential, the LTMBE-GaAs layers also have many interesting material properties such as high defect concentration, high electrical resistivity and are therefore of scientific interest as well. Previous reports revealed that LTMBE-GaAs layers grown at substrate temperatures in the range of 200-250°C are high quality single crystals with > 1 atomic % excess As [1-3]. These layers have lattice parameters larger than that of a normal GaAs crystal by $\sim 0.1\%$ as measured by x-ray rocking curve experiments. The breakdown of the crystallinity in these layers occurs at growth temperature below 200°C [6]. At $T_g < 200^\circ\text{C}$, only a thin layer ($< 2 \mu\text{m}$) of good quality GaAs can be grown before a high density of "pyramidal" defects nucleates and columnar polycrystalline growth starts.

Electron paramagnetic resonance measurements on LTMBE-GaAs layers showed that these layers have $\sim 5 \times 10^{18} \text{ cm}^{-3}$ As antisite defects [2]. In addition, they are also found to be highly resistive and possess no measurable photoluminescence signal [1]. Subsequent annealing of these layers above 400°C in As overpressure results in a uniform semi-insulating property and the lowering of their lattice parameter to the value of stoichiometric GaAs grown at normal temperatures ($\sim 600^\circ\text{C}$). Transmission electron microscopy (TEM) investigation revealed that As precipitates with diameter ranging from 2 to 5 nm are formed in the layers after annealing [2-5]. The As precipitates formation in the LTMBE layers has been studied in detail by many investigators [4,5,8]. Recently, it has also been speculated that the semi-insulating nature of the annealed LTMBE-GaAs layers is the result these As precipitates [5].

In this paper we report on an investigation of the LTMBE-GaAs layers grown at $T_g = 200^\circ\text{C}$ using ion beam techniques, namely, particle induced x-ray emission (PIXE), Rutherford backscattering spectrometry (RBS), and the combination of these techniques with ion channeling. Our study addresses on the issues of stoichiometry, defects arising from the off-stoichiometry, and the lattice location of excess As in the layers.

EXPERIMENTAL PROCEDURES

The LTMBE-GaAs layers were grown on semi-insulating liquid-encapsulated Czochralski (100) GaAs substrates. The samples were grown using a Varian GEN II MBE system at $T_g = 200^\circ\text{C}$ with a growth rate $\sim 1\mu\text{m/hr}$. The thickness of the layers grown is $\sim 2\text{--}3\mu\text{m}$. Some of the layers were annealed at 600°C in the MBE chamber with an As overpressure. The stoichiometry of LTMBE-GaAs layers was measured by PIXE using a 1.2 MeV H^+ beam with samples tilted at $60\text{--}70^\circ$ with respect to the ion beam. The beam energy and the tilt angle were chosen so that the x-ray emitted by the substrate GaAs is $< 10\%$ of the total collected x-ray signals. The x-rays emitted were detected by a Si(Li) detector located at 30° with respect to the ion beam.

The crystallinity and the depth profile of crystalline defects in the LTMBE-GaAs layers were characterized by RBS in the channeling orientation using a 1.95 MeV $^4\text{He}^+$ beam. Back-scattered particles were analyzed by a Si surface barrier detector located at 165° with respect to the ion beam. The samples were mounted on a two-axis goniometer for alignment. The lattice location of the excess As atoms in the LTMBE-GaAs layers grown at 200°C was studied by PIXE/channeling using a 0.5 MeV H^+ beam. The ion beam was aligned along the $\langle 110 \rangle$ axis of the layer. Angular scans were obtained by tilting the sample parallel to a $\{110\}$ plane across the $\langle 110 \rangle$ axis and measuring the $\text{GaK}\alpha$ and $\text{AsK}\beta$ x-rays. The x-ray yields at each tilt angle were normalized by the values obtained when the ion beam was not aligned with any axis of the sample, i. e., in a random direction.

RESULTS AND DISCUSSION

Figure 1 shows the $\text{GaK}\beta$ and $\text{AsK}\alpha$ x-rays obtained by PIXE for a LTMBE GaAs layer ($T_g = 200^\circ\text{C}$) and the bulk GaAs standard normalized by the $\text{GaK}\alpha$ x-ray (not shown in the figure). The higher $\text{AsK}\alpha$ x-ray yield from the LTMBE layer indicates the presence of excess As in the layer. Over 10^6 counts of $\text{GaK}\alpha$ and $\text{AsK}\alpha$ x-rays were accumulated. The relative composition of the layer $[\text{As}]/[\text{Ga}]$ is calculated by taking the ratio of the total measured $\text{AsK}\alpha$ and the $\text{GaK}\alpha$ x-rays and comparing this ratio to that obtained for a bulk GaAs standard. Since the $\text{GaK}\beta$ and the $\text{AsK}\alpha$ x-rays are not resolved, it is necessary to subtract the $\text{GaK}\beta$ x-ray from the total yield in order to obtain a more accurate $[\text{As}]/[\text{Ga}]$ ratio. This is carried out by assuming a fixed $\text{GaK}\beta/\text{GaK}\alpha$ yield ratio. The overall statistical error associated with this measurement is estimated to be $\sim 0.2\%$. The amount of excess As, $\Delta[\text{As}] = ([\text{As}] - [\text{Ga}]) / ([\text{As}] + [\text{Ga}])$, in this case is ~ 0.01 ($\sim 4 \times 10^{20}$ atoms/cm³). The effect of annealing on the stoichiometry of the LTMBE layers is also studied. PIXE results show that when the layers were annealed under an As overpressure, there is no significant change in $\Delta[\text{As}]$ up to an annealing temperature of 600°C .

X-ray rocking curve measurements on the unannealed LTMBE layers show that the lattice parameter, a , of the layers is considerably larger than that of a normal GaAs crystal, a_b [2,6,7]. The deviation in the lattice parameter, $\Delta a = (a - a_b)/a$, is found to be $\sim 0.2\%$. Therefore, the PIXE and x-ray results show that more excess As is incorporated into the layer at $T_g = 200^\circ\text{C}$ and that the dilation of the lattice parameter in the layers can be attributed to the presence of excess As atoms in the layer.

Rocking curve measurements [7] on samples annealed in As overpressure show that the lattice parameter of the layers gradually decreases as the layers are annealed at temperatures higher than 300°C and finally resume the bulk value when the annealing temperature reaches 450°C . Since no As loss can be detected by PIXE, the excess As atoms are believed to coalesce forming As precipitates in the layer and thus returning the lattice parameter to a_b . This is

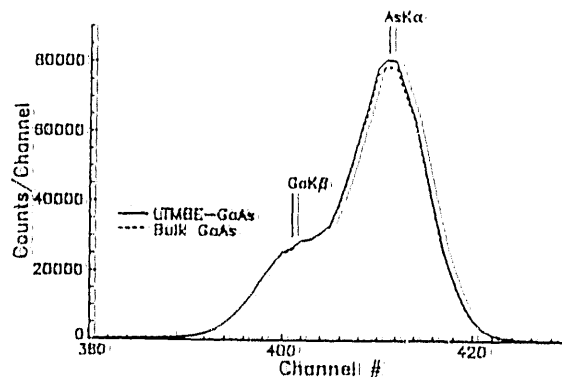


Fig.1 1.2 MeV H^+ PIXE spectra from the bulk GaAs standard and a LTMBE GaAs layer grown at $T_g = 200^\circ\text{C}$. Only the $\text{GaK}\beta$ and $\text{AsK}\alpha$ peaks are shown in the figure.

confirmed by TEM experiments which observed small As precipitates (2-3 nm in diameter) in a LTMBE layer ($T_g=200^\circ\text{C}$) annealed at 450°C under As overpressure [8].

For LTMBE layers grown at $T_g \geq 200^\circ\text{C}$, the RBS/channeling spectra are very similar to that of the bulk GaAs sample with slightly higher dechanneling in the spectra from the layer as the ions penetrate deeper into the layer. Fig. 2 shows the $\langle 110 \rangle$ aligned RBS spectra from a bulk GaAs and a LTMBE layer grown at $T_g=200^\circ\text{C}$ as grown and annealed at 600°C under As overpressure. Note that except for the higher dechanneling rate in the spectra from the layers, all the spectra are very similar to the bulk one indicating that the layers have good crystalline quality. Since TEM experiments did not reveal any extended defects in these layers, we can assume that the higher dechanneling rates in the layers are due to point defects. The concentration of point defects can be calculated from the RBS/channeling spectra as outlined in Feldman et al. [12]. The point defect concentrations in the as-grown and annealed layers calculated from the data in Fig. 2 are shown in Fig. 3. A uniform distribution with $n_D \sim 1.7 \times 10^{20} \text{cm}^{-3}$ is observed in the as-grown layer. This value of n_D is lower than the total excess As concentration measured by PIXE in this layer ($\sim 4.0 \times 10^{20} \text{cm}^{-3}$). The n_D for the annealed sample increases from $\sim 1 \times 10^{20}$ at the surface to a constant value of $\sim 4.6 \times 10^{20} \text{cm}^{-3}$ at $0.4 \mu\text{m}$ depth. This saturated value of point defect density agrees very well with the excess As concentration in the layer, indicating that most of the excess As in the annealed layer exist in the form of random point defects contributing to the dechanneling of the ion beam. TEM on this layer revealed that small As precipitates (2-3 nm in diameter) are present in this layer. These precipitates are either amorphous or "pseudocubic" constrained by the GaAs host crystal [8]. These observations are in good agreement with the RBS/channeling results. The fact that the n_D in the as-grown layer is much lower than the excess As concentration suggests that the excess As in the as-grown layer may be in the Ga sites or sitting in specific sites close to the normal host sites and therefore with a dechanneling factor, σ_D smaller than that of a random point defect. To investigate the lattice location of the excess As atoms in the layer, PIXE/channeling experiments were also carried out.

Figure 4 shows the angular scans of the $\text{GaK}\alpha$ and $\text{AsK}\beta$ x-rays across the $\langle 110 \rangle$ axis along a $\{110\}$ plane from (a) the bulk GaAs standard and (b) the as-grown LTMBE layer. The $\langle 110 \rangle$ axis is chosen since the Ga and As atoms form separate rows in this direction [12,13] defining two separate channels, one bound by three Ga rows and the other by three As rows. Since the critical angle for channeling ψ is proportional to the square root of the atomic number Z_2 of the target atoms forming the channel, i.e. $\psi \propto Z_2^{1/2}$ [12] the values of the ψ for the scans arising from the $\langle 110 \rangle$ channel as defined by the Ga and As strings should be different. In this case, $\psi(\text{Ga})/\psi(\text{As}) \propto \sqrt{(31/33)} = 0.97$. The critical half angle $\psi_{1/2}$ of the scans is defined as the angular deviation between the channeled direction and the angle at which the x-ray yield is equal to half of the value when the alignment of the beam is far from the axial channel. Since $\psi_{1/2} \propto \psi$, the measured $\psi_{1/2}$ values are also $\propto Z_2^{1/2}$. Table I summarizes the average $\psi_{1/2}$'s of the $\text{GaK}\alpha$

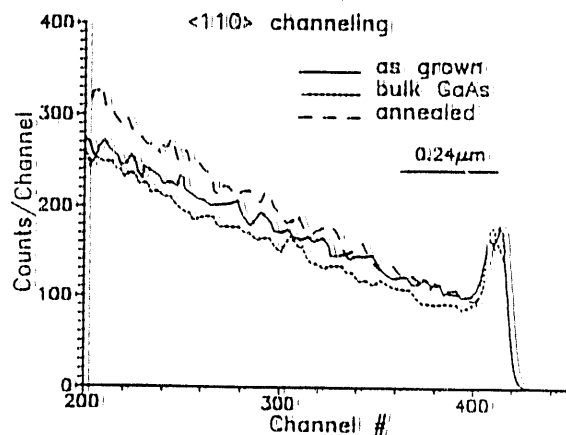


Fig.2 1.95 MeV $\langle 110 \rangle$ aligned RBS spectra from a LTMBE layer ($T_g=200^\circ\text{C}$) as grown, annealed in As overpressure at 600°C , and a bulk GaAs standard.

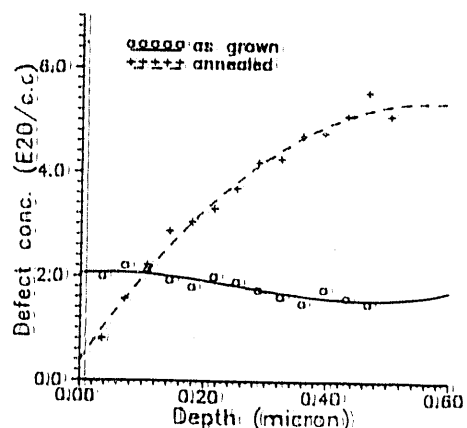


Fig.3 Point defect concentration, n_D as a function of depth derived from the channeling spectra in Fig. 2 for the as grown and annealed LTMBE layer.

Table I. A summary of the average critical half angles, $\psi_{1/2}$, the minimum yield, χ_{\min} , and the x ratios obtained for the $\text{Ga}_{\text{K}\alpha}$ and $\text{As}_{\text{K}\beta}$ scans of the bulk GaAs standard and the as-grown and annealed LTMBE GaAs layers.

	Average $\psi_{1/2} <110>$ ($\pm 0.03^\circ$)		$x = \frac{\psi_{1/2}(\text{Ga}_{\text{K}\alpha})}{\psi_{1/2}(\text{As}_{\text{K}\beta})}$	χ_{\min}	
	$\text{Ga}_{\text{K}\alpha}$	$\text{As}_{\text{K}\beta}$		$\text{Ga}_{\text{K}\alpha}$	$\text{As}_{\text{K}\beta}$
Bulk GaAs	0.79	0.85	0.93	0.20	0.17
As-grown LTMBE	0.78	0.74	1.05	0.23	0.22
Annealed LTMBE	0.78	0.80	0.97	0.21	0.20

and $\text{As}_{\text{K}\beta}$ scans and χ_{\min} for the bulk standard, the as-grown and the annealed LTMBE layers.

The ratios: $x = \psi_{1/2}(\text{Ga})/\psi_{1/2}(\text{As})$ for the three samples are also tabulated.

In Table I we notice that $x=0.93$ for bulk GaAs, slightly lower than the calculated value 0.97. Since the ratio of the x-ray absorption coefficient of $\text{As}_{\text{K}\beta}$ to that of the $\text{Ga}_{\text{K}\alpha}$ in GaAs is = 2, the mean emission depth of the $\text{Ga}_{\text{K}\alpha}$ is greater than that of the $\text{As}_{\text{K}\beta}$. The net effect of this is that the $\psi_{1/2}(\text{Ga})$ becomes narrower due to dechanneling of the beam at greater depth in the sample. Therefore $x < 0.97$ in our measurement is consistent with the calculation. For the as-grown LTMBE layer, $x=1.05$, much greater than that for bulk GaAs. Notice also that the absolute values of the $\psi_{1/2}(\text{Ga})$ for the three samples are equal within a measurement error ($\pm 0.04^\circ$). The significant narrowing of the $\psi_{1/2}(\text{As})$ for the as grown LTMBE layer indicates that the $<110>$ channel as defined by the As strings is smaller. The most probable cause for this is the presence of As atoms slightly displaced from the normal As position into the $<110>$ channel. Since the PIXE results reveal that $\approx 4 \times 10^{20}$ excess As atoms/cm³ are present in the layer, the narrowing of the $\psi_{1/2}(\text{As})$ can be interpreted as the result of the presence of excess As atoms located close to the As atom rows.

In Fig. 4(b) we also observe two "kinks" in the $\text{As}_{\text{K}\beta}$ scan at tilt angle $\approx 0.35^\circ$ in both directions of the scan for the as-grown LTMBE GaAs as indicated by the arrows in the figure. It should be pointed out that although these "kinks" are only slightly larger than the measurement error, they are reproducible in the as-grown sample but are not observed in either the annealed or the bulk standard sample. However, due to the relatively large statistical error, the following quantitative analysis on these "kinks" can only provide a rough estimation. Using the continuum model formulated by Lindhard [14], the atomic displacement r_x can be related to the $\psi_{1/2}$ by the following expression:

$$\psi_{1/2} \propto \sqrt{\ln\left[\left(\frac{Ca}{r_x}\right)^2 + 1\right]}$$

where C is a constant and a is the Thomas-Fermi screening distance. For a normal crystal, r_x is just the transverse rms thermal vibration amplitude, ρ ($\approx 0.1202 \text{ \AA}$ for GaAs). A rough estimate using 0.35° as the half angle for the displaced As atoms yields a projected displacement $r_x \approx 0.3 \text{ \AA}$ in the as-grown LTMBE sample.

In the $<110>$ axis, as the sample is tilted parallel to a $\{110\}$ plane, the ion beam will interact with an As string in one direction and a Ga string in the other direction. This asymmetric effect has been used to identify the lattice location of impurity atoms in many III-V semiconductors [15,16]. In our channeling results, the direction toward the negative tilt angles corresponds to the direction toward the As string. This is confirmed by the Rutherford

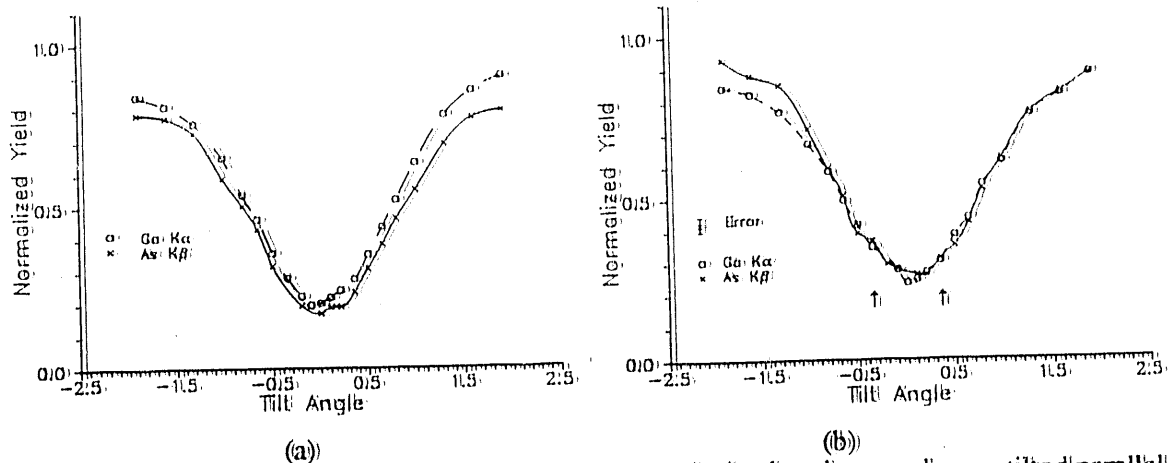


Fig.4 Angular scans of the $GaK\alpha$ and $AsK\beta$ x-rays obtained as the samples are tilted parallel a (110) plane about a $\langle 110 \rangle$ axis for (a) a bulk GaAs standard, and (b) the LTMBE GaAs layer. Note the "kinks" in (b) in the As scan.

backscattering results recorded simultaneously with PIXE. The $\psi_{1/2}$'s and x ratios for $GaK\alpha$ and $AsK\beta$ from both the bulk and the as-grown LTMBE GaAs as the sample is tilted toward the As row parallel to a (110) plane are tabulated in Table II. The pronounced narrowing in the $AsK\beta$ scan from the LTMBE GaAs sample suggests that the excess As atoms in the sample are preferentially bonded to the As atoms in the normal As sites. The stronger "kink" toward the As string direction in the As scan in Fig. 4(b) also confirms this suggestion.

Experiments performed on as-grown LTMBE-GaAs layers using convergent beam electron diffraction (CBED) and large-angle diffraction pattern (LACBED) [10] suggested that the excess As atoms in these layers do not reside on tetrahedral interstitial sites. The CBED and LACBED results can be explained by assuming that the As atoms form lower symmetry split interstitials along the $\langle 111 \rangle$ axis, i.e., assuming that an As atom at $1/4, 1/4, 1/4$ in the unit cell is shifted to a new position $1/8, 1/8, 1/8$ and an interstitial is inserted at $3/8, 3/8, 3/8$ [10]. Our PIXE/channeling results are compatible with this suggestion.

Ion channeling experiments on the annealed LTMBE sample show that the narrowing on the $AsK\beta$ scan is less than that of the as-grown sample. For the annealed sample, $x=0.97$, slightly higher than that of bulk GaAs. The slight narrowing of the $AsK\beta$ scan of this sample may then be caused by the random scattering from these As precipitates. The slight increase in the $\chi_{min}(As)$ of this annealed sample as compared to the standard also indicates that $\sim 1-2\%$ of the As atoms in the sample are not in registry with the matrix as viewed along the $\langle 110 \rangle$ direction.

The PIXE/channeling results reveal that the excess As atoms in the as-grown LTMBE layer are not in exact interstitial positions but are sitting close to the normal As sites in the lattice. Therefore, the dechanneling factor σ_D [12] for these As atoms is expected to be smaller. With this adjustment in σ_D the calculated n_D from the RBS/channeling data is $\sim 2.5 \times 10^{20} \text{ cm}^{-3}$ and is still lower than $\Delta[As]$ in the layer. The only explanation is that a fraction of these excess As atoms are in exact substitutional position not contributing to the dechanneling. Recently, using

Table II. A summary of the critical half angles, $\psi_{1/2}$, and the x ratios for the $GaK\alpha$ and $AsK\beta$ scans from the bulk GaAs standard and the LTMBE GaAs layer obtained when the samples are tilted toward the As atom string along a (110) plane.

	$\psi_{1/2} \langle 110 \rangle$ toward the As row		x ratio
	$GaK\alpha$	$AsK\beta$	
Bulk GaAs	0.78	0.81	0.96
LTMBE GaAs	0.76	0.68	1.12

optical absorption and electron paramagnetic resonance techniques, Kaminska et al. [7] found $\sim 1.2-1.3 \times 10^{20} \text{ cm}^{-3}$ neutral As_{Ga} antisites in an as-grown LTMBE layer grown at $T_g = 200^\circ\text{C}$. This result is in good agreement with our observations on ion channeling.

CONCLUSIONS

We have found a high level of excess As, $\Delta[\text{As}] = 0.01$, in LTMBE GaAs layers grown at substrate temperature $\approx 200^\circ\text{C}$. The $\Delta[\text{As}]$ in the layer is believed to be responsible for the dilation of the lattice constant in the as-grown layers. Upon annealing in As atmosphere above 450°C these excess As coalesce forming As precipitates. A large fraction ($\sim 60\%$) of the excess As atoms in the as-grown layers are found to occupy a position very close to the normal As sites in the lattice. The rest of the excess As atoms are believed to be in As_{Ga} antisites.

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