Enhanced Crystal Quality of Al_xIn_{1-x}As_ySb_{1-y} for Terahertz Quantum Cascade Lasers

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Abstract: This work provides a detailed study on the growth of Al_xIn_{1-x}As_ySb_{1-y} lattice-matched to InAs by Molecular Beam Epitaxy. In order to find the conditions which lead to high crystal quality deep within the miscibility gap, Al_0.462In_0.538As_ySb_{1-y} with x = 0.462 was grown at different growth temperatures as well as As_2 and Sb_2 beam equivalent pressures. The crystal quality of the grown layers was examined by high-resolution X-ray diffraction and atomic force microscopy. It was found that the incorporation of Sb into Al_0.462In_0.538As_ySb_{1-y} is strongly temperature-dependent and reduced growth temperatures are necessary in order to achieve significant Sb mole fractions in the grown layers. At 480 °C lattice matching to InAs could not be achieved. At 410 °C lattice matching was possible and high quality films of Al_0.462In_0.538As_ySb_{1-y} were obtained.

Keywords: MBE; QCL; THz; Growth; AlInAsSb; Antimonides

1. Introduction

InAs is an interesting material for inter-subband devices such as quantum cascade lasers (QCLs) due to its low effective mass, since it is expected to enable higher performance by providing higher gain [1]. In order to exploit the beneficial properties of InAs as the well material, a suitable barrier material is necessary. The commonly used Al(As)Sb material, though enabling excellent results for mid-infrared (MIR) devices [2–5], requires the use of monolayer and sub-monolayer thin barriers for devices designed to emit in the terahertz (THz) regime, due to its very high conduction band offset. The growth of such thin layers is very difficult to control, and to date, no THz laser operation has been shown.

A viable option for the barrier material is the quarternary Al_xIn_{1-x}As_ySb_{1-y} material, which can be grown lattice-matched to InAs and should have properties tunable from InAs to AlAs_{0.16}Sb_{0.84}. By choosing an appropriate composition, it would be possible to grow InAs based THz QCLs with a low conduction band offset and therefore easy to control barrier thicknesses.

Reports about the growth of Al_xIn_{1-x}As_ySb_{1-y} at various compositions can be found in literature, however, most of these works do not specifically study the growth of Al_xIn_{1-x}As_ySb_{1-y} and hence little information exists on the effect of the various growth parameters on the crystal quality and layer composition. An overview of the compositions and the respective growth temperatures is given in Figure 1. Turner et al. [6] and Wilk et al. [7] report on the growth Al_xIn_{1-x}As_ySb_{1-y} with Al mole fractions below 0.20 at growth temperatures (T_g) of 430 °C and 420 °C, while Semenov et al. [8] show that compositions with Al mole fractions of up to 0.25 can be grown at a T_g of 450 °C to 500 °C. For
the greatest flexibility when it comes to designing devices employing $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$, composition with higher Al mole fractions have to be investigated and conditions have to be found which suit the widest range of compositions. The growth of $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ on GaAs with higher Al mole fractions is reported at a $T_g$ well below 400 °C. Kudo et al. [9] studied the incorporation of Sb into $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ with an Al mole fraction of 0.5 at a $T_g$ of 350 °C grown on GaAs. The authors report a high dislocation density, found in a transmission electron microscopy study of the grown material, resulting from the highly lattice-mismatched conditions. It is questionable whether this result is applicable to the growth of $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ on InAs. Waahington-Stokes et al. [10] report on the growth of $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ with an As mole fraction between 0.37 and 0.72 at a $T_g$ between 355 °C and 378 °C on GaSb substrates and report good crystal quality for the grown layers. The influence of the Sb and As beam equivalent pressures (BEP) or the $T_g$ on the quality and composition of the layers is not mentioned.

2. Experiment Section

2.1. Growth Optimization of $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$

The lack of information on the growth of $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ with Al mole fractions around 0.5 at a $T_g$ above 400 °C suggests that challenges, possible due to the presence of a miscibility gap [8], arise from such conditions. Since high crystal quality is expected to be achieved at high $T_g$, the upper limit for the growth of $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ and the dependence of the crystal quality on the $T_g$ have to be investigated.

$\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ with an aluminum mole fraction fixed to 0.462, was grown on InAs. The growth rates of AlAs (on InAs) ($R_{\text{AlAs}}$) and InAs ($R_{\text{InAs}}$) were constant for all samples. $R_{\text{AlAs}}$ was set to 0.25 monolayers per second (ML s$^{-1}$) which corresponds to an aluminum BEP 1.3 × 10$^{-7}$ torr. $R_{\text{InAs}}$ was set to 0.29 ML s$^{-1}$ equaling an indium BEP of 4.5 × 10$^{-7}$ torr. For the comparison of the group V BEPs to other MBE systems, the As$_2$ flux was calibrated by the (2 × 4) to (4 × 2) transition during growth at different temperatures and normalized to 1 ML s$^{-1}$. A fit to the exponential function $P_{\text{As}}(T) = a + b \exp(cT)$ resulted in the parameters $a = 0.531$ torr, $b = 2.14 \times 10^{-14}$ torr, $c = 23.24$ K$^{-1}$. A plot of this experiment can be found in the supplementary material section.

To find the optimum growth conditions, $T_g$, As$_2$ BEP ($P_{\text{As}_2}$) and Sb$_2$ BEP ($P_{\text{Sb}_2}$) were varied. The range of $T_g$ used in this study was 410 °C to 480 °C. Since $\text{Al}_x\text{In}_{1-x}\text{As},\text{Sb}_{1-y}$ should be used in heterostructures together with InAs, $P_{\text{As}_2}$ is, in principle, dictated by the optimum BEP to grow high quality InAs [11]. In this study, $P_{\text{As}_2}$ was varied in order to investigate its influence on the composition of $\text{Al}_{0.462}\text{In}_{0.538}\text{As},\text{Sb}_{1-y}$. The growth parameter regions investigated in this study are indicated by the gray shaded areas in Figure 1.

To investigate the influence of growth parameters, a set of samples was grown in a Riber 32 MBE system on indium-bonded n+ InAs (001) wafers. For the group V materials, valved cracking cells were used with the cracking zone temperatures set to 850 °C and 1000 °C to produce As$_2$ and Sb$_2$ respectively. For all samples, first the oxide was thermally desorbed under $P_{\text{As}_2}$ of 1.5 × 10$^{-5}$ torr at 510 °C for 20 min. The temperature was then reduced to 480 °C to grow a 150 nm InAs buffer layer to enhance the surface quality after thermal desorption of the oxide. Next, the $T_g$ was adjusted to the desired growth temperature, at which a 150 nm thick $\text{Al}_{0.462}\text{In}_{0.538}\text{As},\text{Sb}_{1-y}$ layer and a 5 nm InAs cap, were grown.

All fabricated heterostructures were measured with high resolution x-ray diffraction (HRXRD) to investigate the crystal quality and composition. The As mole fraction (y) in the grown $\text{Al}_{0.462}\text{In}_{0.538}\text{As},\text{Sb}_{1-y}$ layers was corroborated by measuring their in-plane and out-of-plane lattice constant in reciprocal space maps around the InAs (224) diffraction peak and applying Vegard’s law. Atomic force microscopy (AFM) was used to determine the surface roughness of all samples.
To investigate the incorporation of Sb into Al$_{1-x}$In$_x$As$_y$Sb$_{1-y}$, three series of samples were grown. For each series P$_{As2}$ and the T$_g$ were kept constant while P$_{Sb2}$ was varied. P$_{As2}$ was chosen such that high crystal quality InAs can be grown under the same conditions [11]. The results are summarized in Figure 2a. It shows that with increasing P$_{Sb2}$, the rate of Sb incorporation is lowered and a maximum achievable Sb mole fraction is approached asymptotically. Only at a T$_g$ of 410°C can lattice matching with InAs achieved. An explanation for this behavior may be found in the As-for-Sb exchange reaction found for other mixed group V materials like GaAsSb [14]. Due to its negative enthalpy, this reaction is favored with respect to the reverse reaction and its rate rises with T$_g$.

**Figure 1.** Reports on the growth of Al$_{1-x}$In$_x$As$_y$Sb$_{1-y}$ found in literature. (a) Compositions and (b) their respective T$_g$. The dashed lines in (a) indicate the compositions of Al$_{1-x}$In$_x$As$_y$Sb$_{1-y}$ which are lattice matched to InAs or GaSb according to Vegard’s Law. (●) condition explicitly mentioned; the range studied is indicated by the outlined areas) Semenov et al. [8] grown on InAs; similar results were obtained by Rojas-Ramirez et al. [12]; (●) Washington-Stokes et al. [10] grown on GaSb; (●) Turner et al. [6] grown on GaAs; (●) Kudo et al. [9] grown on GaAs; (●) Wilk et al. [7] grown on InAs; (●) Sarney et al. [13] grown on a lattice-constant shifting AlGaInSb buffer on GaSb; The regions of temperature and composition studied within this work are indicated by the gray shaded areas.

![Figure 1](image1.png)

**Figure 2.** (a) As mole fraction (y) in Al$_{0.462}$In$_{0.538}$As$_y$Sb$_{1-y}$ layers with respect to P$_{Sb2}$ at the values for T$_g$ and P$_{As2}$ shown in (b–d). Lattice-matching (indicated by the dashed line at y = 0.588) is only achieved at 410°C. High resolution x-ray diffraction (HRXRD) measurements around the InAs (004) diffraction of samples grown at (b) 480°C, P$_{As2}$=1.5 x 10$^{-5}$ torr (c) 445°C, P$_{As2}$=4.7 x 10$^{-6}$ torr (d) 410°C, P$_{As2}$=3.0 x 10$^{-6}$ torr. Fully strained films are found for samples grown at intermediate P$_{Sb2}$ values at 410°C and 445°C. P$_{Sb2}$ in the subfigures (b–d) given in 1 x 10$^{-6}$ torr starting from the left most peak: (b): 2.13 0.99 (c): 2.57 1.19 1.00 0.54 0.00 (d): 2.55 1.19 0.58 0.39 0.24 0.00.
HRXRD measurements, shown in Figure 2b–d, show high crystal quality for $P_{\text{Sb}_2}$ between $0.5 \times 10^{-6}$ torr and $1 \times 10^{-6}$ torr at 410°C as well as $1 \times 10^{-6}$ torr and $2.5 \times 10^{-6}$ torr at 445°C. This is indicated by sharp layer peaks and Pendellösung fringes. At lower $P_{\text{Sb}_2}$, the layer peaks are broadened due to partial relaxation resulting from the high lattice mismatch. At 410°C, even higher $P_{\text{Sb}_2}$ also leads to peak broadening. The respective (224) reciprocal space map shows partial relaxation. Strong peak broadening due to partial relaxation was also found for all samples grown at 480°C.

AFM measurements performed on the same heterostructures, shown in Figure 3, confirm these observations. Layers containing very little Sb show crosshatch patterns, a sign of partial relaxation. At intermediate $P_{\text{Sb}_2}$, smooth films are obtained. At high $P_{\text{Sb}_2}$, high surface roughness is observed, scale bar = 2 μm. Peak-to-valley values are: (a) 15.9 nm; (b) 3.2 nm; (c) 3.9 nm; (d) 6.4 nm; (e) 23.7 nm; (f) 1.8 nm; (g) 1.5 nm; (h) 44.2 nm.

Although it is convenient to keep $P_{\text{As}_2}$ at a value that is optimal for the growth of the InAs throughout the whole growth, it is possible to change the conditions for each layer, since modern As cracking cells are equipped with valves that allow for the quick change of the flux. It could be beneficial to change $P_{\text{As}_2}$ to a lower value for the growth of $\text{Al}_{0.462}\text{In}_{0.538}\text{As}_{1-y}\text{Sb}_{1-y}$ in order to achieve a lower As mole fraction in the layer and hereby circumvent the restrictions imposed by the growth of InAs. To investigate the influence of $P_{\text{As}_2}$ on the composition of $\text{Al}_{0.462}\text{In}_{0.538}\text{As}_{1-y}\text{Sb}_{1-y}$, a series of samples was grown in which $P_{\text{Sb}_2}$ (1 × 10^{-6} torr and 2.5 × 10^{-6} torr) and the growth temperature ($T_g$ = 445°C) were kept constant while $P_{\text{As}_2}$ was varied. AFM measurements of these samples are shown in Figure 4. From Figure 5a it can be seen that, as expected, decreasing $P_{\text{As}_2}$ leads to a lower As concentration ($y$) in the layer. Figure 5b, shows that the $P_{\text{As}_2}$ can only be adjusted within a very small range since too little As$_2$ leads to a dramatic increase in surface roughness, due a 3-D growth mode (Figure 4c). This behavior is even more pronounced for samples grown at $P_{\text{Sb}_2}$ of 2.5 × 10^{-6} torr (Figure 4a). When $P_{\text{As}_2}$ is decreased below $3 \times 10^{-6}$ torr no epitaxial growth is possible, illustrating that there is a lower limit to the $P_{\text{As}_2}$. This was determined by both a vanishing RHEED pattern during growth and the absence of a layer peak in the HRXRD measurement.
Figure 4. AFM of samples grown at 445 °C at $P_{Sb_2} = 1.0 \times 10^{-6}$ torr and $2.5 \times 10^{-6}$ torr. $P_{As_2}$ is increased left-to-right. At both values of $P_{Sb_2}$ smooth films were obtained at a $P_{As_2}$ of $4.8 \times 10^{-6}$ torr. When $P_{As_2}$ is reduced below this value, the roughness is increased dramatically. This is even more pronounced at the higher value of $P_{Sb_2}$, indicating that this behavior is not caused by a too low total group V flux. It can be concluded that in absence of As, high values of $P_{Sb_2}$ are causing the roughening of the surface. Growing Al$_{0.462}$In$_{0.538}$As$_y$Sb$_{1-y}$ at $P_{Sb_2}$ of $1 \times 10^{-6}$ torr and $P_{As_2}$ of $6.4 \times 10^{-6}$ torr leads to a high lattice mismatch, which is causes crosshatch and an increased surface roughness. Scale bar = 2 µm. Peak-to-valley values are: (a) 236.2 nm; (b) 6.4 nm; (c) 103.6 nm; (d) 6.9 nm; (e) 2.9 nm; (f) 13.2 nm.

Figure 5. (a) Arsenic mole fraction and (b) root mean square surface roughness ($R_q$) of Al$_{0.462}$In$_{0.538}$As$_y$Sb$_{1-y}$ with respect to $P_{As_2}$, at two different $P_{Sb_2}$ values and $T_g=445$ °C. Lattice matching with InAs is indicated by the dashed line in (a). By decreasing $P_{As_2}$, the As fraction in the grown layer is decreased. The minimum of the surface roughness was found around $P_{As_2} = 4 \times 10^{-6}$ torr. Below $P_{As_2} = 3 \times 10^{-6}$ torr no epitaxial grown is possible under these conditions.

To understand its origin, samples showing high roughness but no cross hatch in the AFM analysis have been inspected by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). The micrographs of these samples are shown in Figure 6. Figure 6a shows the effect of low $P_{As_2}$ on the surface morphology. The too low As flux on the surface leads to a lower surface mobility of the group III adatoms, breaking the layer-by-layer growth mode and resulting in a rough surface. EDX measurements at different spots of the sample show that the four atom species (Al,In,As,Sb) are equally distributed ($\sigma < 0.5$ at.%). Figure 6b shows the effect of high $P_{Sb_2}$ at low $T_g$. Similar to Figure 6a 3-D
growth was found. Since this sample was grown with excess $P_{As_2}$, group V deficiency can be ruled out as the source. As for the previous sample, the EDX analysis showed that all four atom species are homogeneously distributed ($\sigma < 0.5$ at.%). Increasing $P_{Sb_2}$ well beyond lattice-matching seems to limit the diffusion length of the group III elements and promote a 3-D growth mode. For both sample (a) and (b) no signs of phase separation due to immiscibility were found and the 3-D morphology seems to be caused by the III/V BEP ratios. High surface roughness was also found for the sample shown in Figure 6c. Unlike for samples (a) and (b) it seems to be due to crystallites with an octagonal base that are well ordered to the substrate. The EDX measurement on this sample shows that the crystallites are rich in antimony while in the surrounding area the composition is close to that found in the HRXRD study. Al and In are homogeneously distributed across the whole sample. This result suggests that $Al_{0.462}In_{0.538}As_ySb_{1-y}$ decomposes into AlInAs and AlInSb under these conditions.

Using the information gathered in this study, two samples were grown on a Riber C21 MBE system on freestanding n+ InAs (001) wafers at a $T_g$ of 410°C. Al mole fractions of 0.25 and 0.53 were chosen and the $As_2$ BEP was adjusted for high quality InAs. $P_{Sb_2}$ was then adjusted in order to achieve lattice matching with InAs. For both compositions high-quality material was obtained, again confirmed by HRXRD and AFM measurements.

2.2. Low Temperature Growth of InAs Based Inter-Subband Devices

The experimental data presented in this work show that low $T_g$ is necessary in order to achieve high crystal quality for both, InAs and $Al_{0.462}In_{0.538}As_ySb_{1-y}$. Growth performed at very low $T_g$, however, can lead to low device performance due to a deterioration of the optical properties and interface quality of the grown materials. To ensure that the requirement to grow at low $T_g$ does not prevent the realization of inter-subband devices, InAs-based devices using AlAsSb barriers were designed and grown at a $T_g$ of 400°C. A quantum cascade detector (QCD) structure with a vertical transition was designed using a semi-classical Monte-Carlo approach. The device was processed into a mesa structure and illuminated through a 45° wedge face. It showed a peak specific detectivity $2.7 \times 10^7 \text{ cm}^2 \sqrt{\text{Hz W}^{-1}}$ at 300 K and a center detection wavelength of 4.84 $\mu$m [15]. Moreover, a THz QCL structure based on a 3-well resonant phonon depletion design was grown. Lasing emission at
3.8 THz was observed by applying a magnetic field at liquid helium temperature. This is the first report of laser performance in the THz regime for InAs-based structures [16].

3. Discussions

A summary of all samples used in this study that have been grown at \( T_g = 445 \, ^\circ\text{C} \) (a) and \( 410 \, ^\circ\text{C} \) (b) is given in Figure 7. In both cases, a large \( P_{As_2}/P_{Sb_2} \) ratio leads to cross hatch due to a large lattice mismatch. For a \( T_g \) of 445 \( ^\circ\text{C} \), a \( P_{As_2} \) below \( 3.3 \times 10^{-6} \) torr leads to a rough surface. Roughening was also found for samples grown at a \( T_g \) of 410 \( ^\circ\text{C} \) for the highest \( P_{Sb_2} \) of \( 2.5 \times 10^{-6} \) torr. While partial relaxation was found in the reciprocal space map around the InAs (224), the roughening seems to be mainly caused by the high \( P_{Sb_2} \) since no sign of cross hatch was found in either EDX or AFM measurements. Growing at higher \( P_{Sb_2} \) at a \( T_g \) of 445 \( ^\circ\text{C} \) results in lower film quality, due to the formation of ordered crystallites with an octagonal base. Since these crystallites were found to be rich in Sb in the EDX analysis, it appears that decomposition, probably due to the miscibility gap of the material occurs under these conditions. A first order approximation, shown as the gray shaded area in Figure 7, was used to locate the region of pressures for which lattice matching can be expected. At a \( T_g \) of 445 \( ^\circ\text{C} \) this region is close, if not within, the region which leads to decomposition and the formation of Sb rich crystallites. At a \( T_g \) of 410 \( ^\circ\text{C} \) the region at which lattice matching is expected is far below the region for which the roughening due to high \( P_{Sb_2} \) is observed and the growth of lattice-matched high quality material is easily achieved.

![Figure 7. Summary of all samples used in this study that have been grown at \( T_g = 445 \, ^\circ\text{C} \) (a) and \( 410 \, ^\circ\text{C} \) (b). At both \( T_g \), high \( P_{As_2}/P_{Sb_2} \) ratios lead to cross hatch due to a large lattice mismatch. For a \( T_g \) of 445 \( ^\circ\text{C} \), \( P_{As_2} \) below \( 3.3 \times 10^{-6} \) torr leads to a rough surface. Growth performed at high \( P_{Sb_2} \) leads to the formation of crystallites which suggest that the material is decomposing due to the miscibility gap. At higher values of \( P_{As_2} \) high quality material is grown. At this \( T_g \) the region in which lattice matching can be achieved is close to the region for which the formation of crystallites is observed. Samples grown at a \( T_g \) of 410 \( ^\circ\text{C} \) show roughening of the surface at high \( P_{Sb_2} \). At this \( T_g \) the region in which lattice matching is possible is far away from the region in which surface roughening was found.](image)

The data presented in this study suggests that a \( T_g \) of 445 \( ^\circ\text{C} \) is the upper limit for lattice matched growth of \( Al_{0.462}In_{0.538}As_ySb_{1-y} \). For a stable growth and high quality material, however, it is advised to use even lower \( T_g \). This is even more critical if the conditions for high-quality InAs and \( Al_{0.462}In_{0.538}As_ySb_{1-y} \) have to be met simultaneously.

4. Conclusions

We investigated the growth of \( Al_{0.462}In_{0.538}As_ySb_{1-y} \). It was shown that the incorporation of Sb into \( Al_{0.462}In_{0.538}As_ySb_{1-y} \) is strongly temperature-dependent and that this is determining the upper
limit of the $T_g$ for which lattice matching can be achieved. Moreover, it was found that the quality of the grown material is very sensitive to the used $P_{As}$, and that low pressures, which are necessary to achieve lattice matching, dramatically increase the roughness of the grown layer. Moreover, it was found that the growth of compositions close to lattice matching at a $T_g$ of 445 °C leads to decomposition of $\text{Al}_x\text{In}_{1-x}\text{As}_y\text{Sb}_{1-y}$ into $\text{AlInAs}$ and $\text{AlInSb}$ and the formation of crystallites which significantly decrease in crystal quality. Below this limit, the requirements for the growth of high quality $\text{Al}_{0.462}\text{In}_{0.538}\text{As}_y\text{Sb}_{1-y}$ and lattice matched growth can be met simultaneously. At a growth temperature of 410 °C, high quality $\text{Al}_{0.462}\text{In}_{0.538}\text{As}_y\text{Sb}_{1-y}$ layers, confirmed by both HRXRD and AFM measurements, were grown. To ensure that growth at low $T_g$ does not prevent the use of this material for inter-subband devices, two InAs based inter-subband structures, a QCD and a THz-QCL, were grown. The QCD showed a peak specific detectivity of $2.7 \times 10^7 \text{ cm}\sqrt{\text{Hz W}^{-1}}$ at 300 K at a center detection wavelength of 4.83 μm. For the THz-QCL, we were able to show the first lasing emission in the THz regime, at an emission frequency of 3.8 THz, for InAs-based structures.

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Author Contributions: T.Z. conceived and designed the experiments, performed HRXRD, SEM, EDX and AFM measurements and analyzed the data; T.Z. and A.M.A. grew samples and wrote the paper; D.M., H.D. and W.S. configured the MBE for the growth of AlAs, and lattice matched growth can be met simultaneously. At a growth temperature of 0.462 $\text{In}_x\text{As}_{1-x}$ layers, confirmed by both HRXRD and AFM measurements, were grown. To ensure that growth at low $T_g$ does not prevent the use of this material for inter-subband devices, two InAs based inter-subband structures, a QCD and a THz-QCL, were grown. The QCD showed a peak specific detectivity of $2.7 \times 10^7 \text{ cm}\sqrt{\text{Hz W}^{-1}}$ at 300 K at a center detection wavelength of 4.83 μm. For the THz-QCL, we were able to show the first lasing emission in the THz regime, at an emission frequency of 3.8 THz, for InAs-based structures.

Conflicts of Interest: The authors declare no conflict of interest.

References


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