Experimental characterization of model resist materials

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ABSTRACT

New resists are needed to advance EUV lithography. The tailored design of an efficient photoresist is impossible without a fundamental understanding of EUV-induced chemistry. The absorption of a EUV photon by a thin film resist leads to the emission of primary and secondary electrons. The electrons may travel up to tens of nanometers before losing their kinetic energy via collisions which initiate chemical reactions. The “blur” of an aerial image is directly related to the distance that electrons can travel and initiate chemistry in the resist. Thus, identifying how to measure and influence the absorption of EUV photons, emission of electrons, and distance traveled by the secondary electrons is extremely beneficial to the resist community.

In this work, we present several experimental techniques to probe model polymer materials to investigate the impact of specific chemical groups on three critical resist properties: EUV absorption, electron emission, and electron attenuation length (EAL). EUV absorption dictates the efficiency of the film to absorb photons. Total electron yield (TEY) provides information on the conversion of absorbed EUV photons to electrons, whereas photoelectron spectroscopy (PES) provides information on the energies and abundance of generated electrons. The EAL corresponds to the thickness of a material required to reduce the number of emitted electrons to 1/e of the initial value. The EAL reveals the distance the electrons can travel in a resist film, which is directly related to the electron blur. Correlations between the obtained experimental values are discussed.

Figure 1. The experimental spectrum of reflectivity vs. angle (black line) and fit of the experimental data (red line) for polystyrene.
EDITORIAL

High-NA EUV Lithography Challenges: Part I

Henry Kamberian, Photronics, Inc.

I attended this year’s SPIE Advanced Lithography conference in San Jose, California, and the latest ASML technology conference in Veldhoven, Netherlands. After more than two years of hiatus from in-person conferences due to the Covid 19 pandemic, it was refreshing to finally see old friends (no offense to us aging engineers!) and again meet with industry colleagues.

From these two conferences, one important topic for me was the status of High-NA EUV technology.

We all know ASML has been developing the High-NA EUV lithography platform for some time. Based on today’s EUV scanners with 0.33NA optics, the High-NA EUV platform is a completely new system, featuring 0.55NA optics, which enables scaling down to 8nm half-pitch resolution and below.

For all updates, installation of the first High-NA systems are expected in 2023 with volume manufacturing to start in 2025.

While some may consider the transition to High-NA EUV to be less drastic than the initial introduction of EUV lithography, most, as I do, believe this transition will bring significant changes in the EUV lithography ecosystem, especially to the EUV mask. Therefore, as mask makers, are we ready to meet the challenge of High-NA EUV at these aggressive timelines?

The 0.55NA is achieved through anamorphic projection optics. The anamorphic optics requires a change in mask-to-wafer reduction factor to 8x in scan direction and 4x in the other direction. Although the increase in image magnification 8x improves resolution with a reduction of shadowing effects, the drawback to the anamorphic projection optics is it cuts the exposure field size by half. This reduction in field size by half will force lithographers to pattern large chips using two masks through mask stitching methodology, which brings additional complexity to the patterning process. A key question: are there proven solutions for stitching device critical features at half-field boundaries?

The large NA in the 0.55NA platform brings another important change where the EUV illumination incidence angle is lower. This lower angle worsens the mask 3D effect further degrading exposure image contrast. The simplest approach to lessening mask 3D effect would be to reduce the thickness of existing absorbers. However, the lower thickness limit for the current Ta-based absorbers are around 55nm due to an increase in the absorber’s EUV reflectivity, which degrades the image contrast. As imaging features continue to shrink, this reinforces the need to change absorber materials. Novel absorber materials have been extensively studied and characterized over the past few years. Of the various novel absorbers, the low (n) absorber materials, classified as EUVPSM, have received the most attention and acceptance.

These two major changes to the current EUV mask lead to critical questioning and highlights the need for new solutions in key mask-making areas.

While these questions and possible solutions will be further discussed in Part II of this editorial, I would like to close, and keeping with Olympics closing ceremony tradition, call upon all youth (and aging) engineers of the world to assemble in Monterey, California on September 25 through 29 for the 2022 Photomask Technology and EUV Lithography conference. We should look forward to all High-NA EUV technology presentations and discussions at the conference, possibly gaining additional insight into new solutions or get our questions answered.
1. Introduction

In Extreme Ultraviolet (EUV) lithography, only a small fraction of EUV (92 eV) photons are absorbed by a resist film. About 10%–30% of EUV photons can be absorbed by a 30 nm thick film, depending on the film composition. The remaining EUV radiation will be absorbed by a substrate or an underlayer below the resist film. Absorption of a EUV photon by either of them will cause the emission of a photoelectron. The emitted electron can inelastically scatter and generate several slow secondary electrons. Considering the multiplicative nature of slow electron generation, EUV resists should be tuned to capitalize on the abundance of slow electrons to initiate chemical transformations that eventually lead to pattern formation. Therefore, it is very important to understand all processes initiated by electron-resist interactions as well as the length scale on which the electrons can propagate upon ionization. This length scale will ultimately define the resolution of EUV lithography.

In this work, we utilize several model polymer materials to investigate the impact of specific chemical groups on the properties of those materi-
als to absorb EUV photons, generate electrons after EUV absorption, and transport the generated electrons. For this, several types of measurement are performed on the model polymers, including EUV absorption, total electron yield (TEY), electron attenuation length (EAL), and photoelectron spectroscopy (PES). The EUV absorption measurement characterizes the efficiency of material to capture EUV photons, which are needed to start the lithography process. TEY determines the number of electrons escaping from the film after EUV photon absorption. That number is proportional to the number of electrons generated by EUV radiation inside the film. Together with EUV absorption measurement, it allows for the evaluation of the EUV photon to the electron conversion efficiency of a particular material. The EAL measurement characterizes distances which slow electrons can travel in a resist film, the value is directly related to the resolution of final photoresists. Photoelectron spectroscopy provides information not only on the abundance of emitted electrons but also on their energies, allowing for the characterization of the energy distribution of EUV-generated electrons.

### 2. Choice of Materials

We plan to systematically investigate the above-mentioned properties of EUV resist materials, starting from building blocks of the photoresists (polymers, photoacid generators (PAGs), etc.), and moving to composite materials. Such an approach will help with understanding the function of each resist component and its role in a complex resist concoction. A set of the model, polystyrene-based polymers, as well as poly(methyl methacrylate) (PMMA), were chosen to investigate how different functional groups affect their properties. Materials were obtained from commercial suppliers and used without further purification. The density of polymers was obtained from literature sources.

### 3. Reflectometry

To obtain EUV absorption properties of materials two approaches are used: experimental determination and calculations. The measurement of absorption was performed at the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory using the reflectivity method. In this approach, the reflectivity at 13.5 nm from the thin polymer films was measured as a function of the angle. The example of experimental data for polystyrene is presented in Figure 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density, g/cm³</th>
<th>β</th>
<th>Measured μ, µm⁻¹</th>
<th>Calculated μ, µm⁻¹</th>
<th>Measured μ/ρ, cm²/g</th>
<th>Calculated μ/ρ, cm²/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>poly(methyl methacrylate)</td>
<td>1.180</td>
<td>0.00587</td>
<td>5.46</td>
<td>5.18</td>
<td>46,310</td>
<td>43,892</td>
</tr>
<tr>
<td>polystyrene</td>
<td>1.000</td>
<td>0.00291</td>
<td>2.71</td>
<td>2.80</td>
<td>27,120</td>
<td>28,009</td>
</tr>
<tr>
<td>poly(4-hydroxystyrene)</td>
<td>1.160</td>
<td>0.00419</td>
<td>3.90</td>
<td>4.04</td>
<td>33,612</td>
<td>34,791</td>
</tr>
<tr>
<td>poly(4-methylstyrrene)</td>
<td>1.040</td>
<td>0.00286</td>
<td>2.66</td>
<td>2.90</td>
<td>25,608</td>
<td>27,897</td>
</tr>
<tr>
<td>poly(4-chlorostyrene)</td>
<td>1.220</td>
<td>0.00320</td>
<td>2.97</td>
<td>3.15</td>
<td>24,378</td>
<td>25,817</td>
</tr>
<tr>
<td>poly(4-cyanostyrene)</td>
<td>1.075</td>
<td>0.00358</td>
<td>3.34</td>
<td>3.31</td>
<td>31,030</td>
<td>30,811</td>
</tr>
<tr>
<td>poly(pentafluorostyrene)</td>
<td>1.406</td>
<td>0.01823</td>
<td>16.97</td>
<td>9.13</td>
<td>120,684</td>
<td>64,902</td>
</tr>
<tr>
<td>poly(4-methoxystyrene)</td>
<td>0.962</td>
<td>0.00390</td>
<td>3.63</td>
<td>3.27</td>
<td>37,709</td>
<td>33,983</td>
</tr>
<tr>
<td>poly(4-acetoxy styrene)</td>
<td>1.060</td>
<td>0.00464</td>
<td>4.32</td>
<td>4.03</td>
<td>40,784</td>
<td>38,058</td>
</tr>
</tbody>
</table>

Figure 2. Left panel: Experimental values of EUV linear attenuation coefficient (filled columns) compared with calculated values, shown as non-filled columns. Right panel: The same for mass attenuation coefficient.
where \( \delta \) is dispersive and \( \beta \) is absorptive components of the refractive index of the polymer film. The imaginary part \( \beta \) of the refractive index can be obtained by fitting the experimental data as shown in Fig. 1. From the obtained value of \( \beta \), the linear attenuation coefficient \( \mu \) of the polymer can be calculated by using Eq. (2):

\[
\mu = \frac{\rho N_A \sigma}{M} = \frac{4\pi}{\lambda} \beta,
\]

where \( \rho \) is the density, \( N_A \) is the Avogadro constant, \( M \) is the molar mass of a molecule, \( \sigma \) is the photoabsorption cross-section of a molecule, calculated using experimental atomic photoabsorption cross-sections from the CXRO X-ray database, and \( \lambda \) is the irradiation wavelength.

The mass attenuation coefficient \( \mu/\rho \) is thus

\[
\frac{\mu}{\rho} = \frac{N_A}{M} \sigma = \frac{4\pi}{\lambda \rho} \beta.
\]

Table 3. Experimental values of total electron yield, electron attenuation length, and Hammett para- substituent constants \( \sigma_p \).

<table>
<thead>
<tr>
<th>Material</th>
<th>TEY, (10^5) a.u.</th>
<th>EAL, nm</th>
<th>( \sigma_p )</th>
</tr>
</thead>
<tbody>
<tr>
<td>poly(methyl methacrylate)</td>
<td>11.49±0.57</td>
<td>2.15±0.10</td>
<td>—</td>
</tr>
<tr>
<td>polystyrene</td>
<td>6.15±0.31</td>
<td>—</td>
<td>0.00</td>
</tr>
<tr>
<td>poly(4-hydroxystyrene)</td>
<td>7.06±0.35</td>
<td>1.47±0.10</td>
<td>-0.37</td>
</tr>
<tr>
<td>poly(4-methylstylene)</td>
<td>6.32±0.32</td>
<td>—</td>
<td>-0.17</td>
</tr>
<tr>
<td>poly(4-chlorostyrene)</td>
<td>4.99±0.25</td>
<td>1.35±0.20</td>
<td>0.23</td>
</tr>
<tr>
<td>poly(4-cyanostyrene)</td>
<td>9.94±0.50</td>
<td>—</td>
<td>0.66</td>
</tr>
<tr>
<td>poly(pentafluorostyrene)</td>
<td>15.42±0.77</td>
<td>1.29±0.10</td>
<td>0.06 (0.34)*</td>
</tr>
<tr>
<td>poly(4-methoxystyrene)</td>
<td>7.95±0.40</td>
<td>2.00±0.15</td>
<td>-0.27</td>
</tr>
<tr>
<td>poly(4-acetoxy styrene)</td>
<td>8.30±0.42</td>
<td>1.78±0.10</td>
<td>0.315 or -0.026</td>
</tr>
</tbody>
</table>

*Constant for meta- substitution

Both calculated and experimental linear and mass attenuation coefficients are summarized in Table 2 and Fig. 2. A good correlation between the experimental and calculated values is observed. For poly(pentafluorostyrene) a big discrepancy between the measured and calculated values is noticed. During the measurement of that sample, it was observed that repeated measurements of the sample were not identical, suggesting sample degradation during the measurement.

From the analysis of tables 1 and 2 and figure 2, it is apparent that the addition of oxygen or fluorine leads to higher EUV absorption. Also, more dense materials have better absorption than the less dense in the case that their monomers have the same photoabsorption cross-section. Two notable cases are poly (methyl methacrylate) and poly(4-methoxystyrene). Both have comparable cross-sections of monomers (7.30 Mb and 7.57 Mb, correspondingly), but PMMA has better linear and mass attenuation coefficients due to its 23% higher density.
4. Total Electron Yield

To measure the total electron yield, the drain current was measured as a function of time. Apart from the properties of the material, the incident photon flux would also affect the drain current. Beamline conditions vary from one experiment to another, resulting in changes in photon flux. To meaningfully compare multiple samples, one measures the electron yield per unit of photon flux. Therefore, prior to each experiment, the photon flux is measured with a silicon diode and the drain current data is normalized with the diode current. ALS storage ring current fluctuations would manifest as fluctuations in flux. The storage ring current is therefore measured concurrently with both diode and drain current measurements to mitigate this problem.

As most samples are to a certain extent sensitive to EUV radiation, only the measurements when EUV exposure of samples just started (t = 0s) are representative of a sample in its original state. However, measuring only one data point would make the experiment very sensitive to noise. Moreover, as we would explain later, the time dependence could provide useful information about the materials.

To reduce the uncertainty in the TEY intensity at t = 0s ($I_0$), we fit the TEY time evolution with a simple model assuming there are three components. The model value of $I_0$ essentially uses information from neighboring data points to mitigate the uncertainty of a single data point measurement.

$$I(t) = C_c + C_r \left[1 - \exp \left(-\frac{t}{\tau_r}\right)\right] + C_d \exp \left(-\frac{t}{\tau_d}\right)$$
In equation (4), $C_c$ is the constant component, $C_r$ is the rising component, and $C_d$ is the decaying component. To make error propagation more transparent, new variables are introduced to make $I_0$ itself a model parameter:

$$I_0 = C_c + C_d$$

$$r_r = \frac{C_r}{I_0}$$

$$r_d = \frac{C_d}{I_0}$$

As a result, equation (4) is rewritten:

$$I(t) = I_0 - I_0r_d + I_0r_r \left[ 1 - \exp \left( \frac{-t}{t_r} \right) \right] + I_0r_d \exp \left( \frac{-t}{t_d} \right)$$

$$= I_0 \left[ 1 - r_d + r_r \left[ 1 - \exp \left( -\frac{t}{t_r} \right) \right] + r_d \exp \left( -\frac{t}{t_d} \right) \right]$$

$$= I_0 \left[ 1 + r_r \left[ 1 - \exp \left( -\frac{t}{t_r} \right) \right] + r_d \exp \left( -\frac{t}{t_d} \right) - 1 \right]$$

$$= I_0 \left[ 1 + r_r \left[ 1 - \exp \left( -\frac{t}{t_r} \right) \right] + r_d \exp \left( -\frac{t}{t_d} \right) - 1 \right]$$

All data traces are therefore fitted against equation (6) to extract $I_0$ and its uncertainty. We note that the current uncertainty ceiling inferred from reproducibility is around 5%, which is way above the usual fitting uncertainty. The extracted $I_0$ values are summarized in Table 3 and shown...
described previously.
The experimental approach to determine electron attenuation length is as observed in Fig. 4.

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**5. Electron Attenuation Length**

The experimental approach to determine electron attenuation length is described previously. Briefly, polymer films of different thicknesses (1-10 nm) were prepared by spin-coating on silicon wafers. The coupons, cut from the wafers, were illuminated to 200 eV X-rays, which mostly penetrate through the polymer film, but are absorbed by the silicon wafer. Part of the electrons, generated by X-rays in the wafer, penetrate through the polymer film. Measurement of the current, generated by electrons transmitted through the films of different thicknesses, allows for the determination of electron attenuation length — the thickness of resist material required to reduce the number of emitted secondary electrons to 1/e of the initial. The method assumes that most electrons leaving the Si wafer will be low kinetic energy secondary electrons, with energies below 20 eV. For the EAL measurement, it is also extremely critical to know the thickness of the films.

Achieving film uniformity is a challenge for films less than ~10 nm, yet film uniformity is critical to obtaining accurate EAL values for a material. The scanning electron microscopy (SEM) images shown in figure 5 provide two examples of poor-quality films alongside a good-quality film of the same material. The recipes used for all four films in Fig. 5 are remarkably similar, with just slight differences in solvent ratios and spin-coating duration. We found it to be critical to verify film quality with either SEM or atomic force microscope (AFM). Measuring the thickness of these films with ellipsometry is also more difficult than normal due to the degeneracy of the film thickness, the index of refraction of the film, and the thickness of the native oxide on the wafer. The index of refraction of the material and the thickness of the oxide layer need to be measured first from separate samples so that those parameters can be held constant when fitting for the film thickness.

If the thin films are not inspected using AFM or SEM, there could be errors in the determination of EAL. For example, the same film thickness could be measured by ellipsometer for both good and poor-quality films, such as shown in Fig. 5. While the good quality films will generate useful data for EAL determination electron signal, in the case of poor-quality films, most of the electrons will be released directly by regions of the film uniformity is critical to obtaining accurate EAL values for a material.

The experimental values of EAL in the polymer films are presented in Table 3 as well as in Figure 6. Most of the EAL values fall in a range between 1 and 2 nm with PMMA having the largest EAL value of 2.15 nm and poly (pentafluorostyrene) — having the smallest EAL of 1.29 nm.
As was discussed above for the case of the TEY time dependence, the electron-withdrawing groups in the case of poly (pentafluorostyrene) or poly (4-chlorostyrene) may lead to efficient electron capture and therefore lead to smaller values of EAL. A Hammett substituent constant is used to characterize electron donating or withdrawing properties of substituents. Thus, it will be instructive to compare the trend observed for EAL with the Hammett parameter, which is summarized in table 3. While there is a correlation between the Hammett parameter and the EAL for chloro-, hydroxy-, and methoxy- substituted polystyrenes, the results are less clear for poly (pentafluorostyrene) and poly (4-acetoxy styrene). For the first one, the direct comparison is not viable because the fluorine group occupies not only para- but also meta- and ortho- positions. For the acetoxy- group two very different Hammett constants could be found: from very positive, electron-withdrawing,\(^1\) to slightly negative, electron-donating.\(^6\) We are planning to expand the EAL determination to the missing samples, which may clarify if there is a correlation between electron donating or withdrawing properties and the value of the EAL.

6. Correlation Between Measurements

Similarly, to the case of near edge X-ray absorption fine structure (NEXAFS) spectroscopy, the intensity of collected TEY signal is a function of the density \((\rho_e)\) of electrons emitted after absorption of EUV photons and EAL, which defines the probability of electrons to escape from the sample and be detected in measurement.\(^7\) In the general case where the photoelectron spectra, the high kinetic energy signal originates due to the emission of primary, valence electrons after absorption of EUV photons. The fluorine atoms, included in PAG, may also emit electrons from the 2s shell at the lower kinetic energy of about 56 eV. Most of the electron signal below 60 eV is due to the emission of secondary electrons. Those electrons appear because of the inelastic scattering of primary electrons. There could be other, minor channels to generate secondary electrons, such as Auger electron emission.

One may see a very small difference between all three photoelectron spectra shown in Fig. 8a, with the signal between 20-80 eV slightly increasing with an increase of PAG loading. The total number of emitted electrons is increased by 10% for 20wt% PAG loading and by 9% for 40wt% PAG loading. To test if the increased electron emission is due to the enhanced EUV absorption, the mass attenuation coefficients were calculated for all three samples. The increased loading of PAG leads to the increase of the EUV absorption mostly due to the fluorine atoms in PAG. The calculated increase of EUV mass attenuation coefficient is 8% for 20wt% PAG and 13% for 40wt% PAG samples, which is close to what was observed in the experiment.

An interesting difference is observed for the high (40 wt.%) PAG loading sample. Firstly, in the experiment, we observe a smaller total number of emitted electrons than in the case of 20 wt.% PAG, which is contradictory to the increased EUV absorption. Secondly, we observe a change in the distribution of slow vs. fast electrons. While both pure polymeric matrix and 20 wt.% PAG sample has 81.9% and 81.4% of slow electrons (electrons with energy below 20 eV), correspondingly. Whereas the 40wt% PAG sample has 79.0% of slow electrons. This small decrease of the number of slow electrons, coupled with the smaller total number of electrons emitted from the sample could indicate that some electrons, generated by EUV exposure in the sample, can be trapped by the PAG.

8. Conclusions

We present the results of fundamental research of model polymers using several experimental techniques. We determined the absorption properties of these materials using reflectometry. The total electron yield measurement characterizes the property of the material to generate electrons after EUV absorption and the temporal evolution of the TEY signal provides information on electron capture by the material. For the same range of materials, we determined the average distance the emitted electrons can travel in the film, characterized by the EAL value. We also discuss the correlation between these three experimental values. The photoelectron spectra of the resist materials can provide information on the abundance and kinetic energies of electrons generated by EUV photons, as well as the ratio of slow vs. fast electrons. The demonstrated techniques will be applied to study more complex resist materials, such as complete resist systems and inorganic resist materials. The acquired data provides a new fundamental understanding of EUV-generated electrons which presumably can be leveraged for the development of better EUV resist materials.

9. Acknowledgements

We appreciate the assistance of Eric Gullikson during measurements and absorption data analysis and Luke Long for PES sample preparation. This work was supported by Intel and Samsung through the U.S. Department of Energy under Contract No. DE-AC02-05CH11231. This research used resources of the Advanced Light Source, a U.S. DOE Office of Science User Facility under contract no. DE-AC02-05CH11231.

10. References


Industry Briefs

■ Is There A Limit To The Number Of Layers In 3D-NAND?

Karen Heyman, Semiconductor Engineering

Memory vendors are racing to add more layers to 3D NAND, a competitive market driven by the explosion in data and the need for higher-capacity solid state drives and faster access time. Micron already is filling orders for 232-layer NAND, and not to be outdone, SK Hynix announced that it will begin volume manufacturing 238-layer 512Gb triple-level cell (TLC) 4D NAND in the first half of next year. Perhaps even more significant, chipmakers say privately they will leverage industry learning about stacking NAND for 3D-ICs, which currently are in development.

“Moore’s Law for processors has arguably been lagging the last few years, but is alive and well for NAND flash,” said Ben Whitehead, technical product manager at Siemens EDA. “It’s a good thing, because modern compute and networking have an insatiable appetite for fast storage.”


■ The Truth About SMIC’s 7-nm Chip Fabrication Ordeal

Majeed Ahmad, Electronic Design News

Semiconductor Manufacturing International Corp. (SMIC) reaching the 7-nm chip fabrication process has been a jaw dropper. Still, while it’s making headlines in the technology and trade media, it’s critical to examine the true value of SMIC’s so-called great leap forward. Can SMIC mass produce chips at its newly developed 7-nm node?

According to some industry observers, SMIC’s 7-nm yields per wafer are in the range of 15%. That makes the chips manufactured at this process node very costly, around 10 times the market price of a chip manufactured at TSMC’s 7-nm node. It’s also worth noting that the crypto-miner chip known to have been manufactured at SMIC’s 7-nm node features a highly parallel design, which implies lower complexity.

In the final analysis, SMIC’s 7-nm story relates more to China’s political cause of semiconductor self-sufficiency than market economics. At the same time, however, it’s a quasi-7-nm chip manufacturing process that could become a stepping stone for a true 7-nm process node. Here, the missing link is ASML’s EUV technology, currently banned for semiconductor fabs in China.

https://www.edn.com/the-truth-about-smics-7-nm-chip-fabrication-ordeal/

■ Mexico Joins the List of Nations Wanting to Lure Chipmakers

Dan Robinson, The Register

Mexico is the next country in line to offer incentives to tempt semiconductor manufacturers to set up shop and may be aiming to take advantage of the US desire to bring manufacturing closer to home. Both the US and the EU have this year unveiled strategies aimed at encouraging chip companies to on-shore semiconductor production and reduce the reliance of their economies on silicon imported from Asia, especially Taiwan.

Mexico is now hoping to take advantage of the US desire to bring chip manufacturing closer to home. As a neighbor of the American economy, the country is well-placed to take advantage of US investment, and has already seen many companies locate factories there, so adding semiconductor manufacturing to the mix might seem a logical step.

https://www.theregister.com/2022/08/22/mexico_joins_the_list_nations/
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