Advanced Electro catalysts through Crystallographic Enhancement

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Fuel Cell Technologies Office Webinar

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This presentation is part of the monthly webinar series provided by the U.S. Department of Energy’s Fuel Cell Technologies Office (FCTO) within the Office of Energy Efficiency and Renewable Energy (EERE). Funding for research, development and innovation to accelerate progress in hydrogen and fuel cell technologies is provided by EERE FCTO.

During Q&A session:
Please type your questions to the chat box. **Send to: (HOST)**
Los Alamos National Laboratory

- Founded: 1943 for the Manhattan Project
- Location: Los Alamos, New Mexico
- $2.6B Budget, ~10,000 employees
Commercial Fuel Cell Vehicles

Toyota Mirai
MSRP: $58,365

Honda Clarity

Hyundai Nexo
Lease $399/mo.

Fuel cell car

Stack

Single cell

MEA
**Introduction - Polymer Electrolyte Fuel Cell**

- Anode: \( H_2 \rightarrow 2H^+ + 2 e^- \)
- Cathode: \( \frac{1}{2}O_2 + 2H^+ + 2 e^- \rightarrow H_2O \)
- Overall Reaction: \( H_2 + \frac{1}{2}O_2 \rightarrow H_2O + \text{Electricity} \)

- Slow \( O_2 \) reduction on the cathode is the main barrier to increased performance.
- Platinum alloy catalysts (PtCo, PtNi) are used to improve \( O_2 \) reduction kinetics, but cost and durability are issues.
Fuel cells need to be cheaper!

**Strategies:**

1. Better catalyst $\rightarrow$ less Pt $\rightarrow$ reduced cost
2. Higher performance $\rightarrow$ smaller fuel cell $\rightarrow$ reduced cost
Objectives

• Design active and durable nanoparticle ORR catalysts based on fully-ordered intermetallic alloys on highly graphitized nitrogen-doped carbon supports
  - Binary and ternary alloys of Pt with Co, Ni, other base metals
  - Project will avoid Fenton-active metals
  - Commercial supports used initially; N-doped C supports later
• Demonstrate catalysts in high-performance, durable MEAs and scale up to 50 cm²

Project Targets:

• Mass activity > 0.44 A/mg_{PGM} @ 0.9 V_{iR-free}
• <40% mass activity loss after catalyst AST
• <30 mV loss at 0.8 A/cm² after catalyst AST
• PGM total loading < 0.125 mg/cm²

• Power density > 1 W/cm²
• <40% mass activity loss after support AST
• <30 mV loss at 1.5 A/cm² after support AST
Ordered intermetallic catalysts

Primary material set:
1. $\text{L}_{10}-\text{M} \text{Pt}$ (also known as face-centered tetragonal) $\text{M} = \text{Co, Ni, other transition metals}$
2. $\text{L}_{10}-\text{M}_1\text{M}_2\text{Pt}$ (ternaries)

Alternative materials (risk mitigation):
1. $\text{L}_{12}$ structures ($\text{Pt}_3\text{M}$)
2. Doping with other elements
3. Other intermetallics

Adapted from Johnston-Peck et al., *Nanoscale*, 2011, 3, 4142
Approach: Synthesis

Use atomic-level ordering to increase performance and durability of Pt-based catalysts

- Synthesize intermetallic nanoparticles (CoPt, NiPt, ternaries)
  - Prepare fully-ordered cores to stabilize base metal
  - Further protect core with Pt skin
  - Use theory and computation (DFT, machine-learning techniques) to guide nanoparticle design
- Support nanoparticles on Fe-free, N-doped graphitic carbon
Approach: Characterization and Testing

Use atomic-level ordering to increase performance and durability of Pt-based catalysts

- Integrate supported nanoparticles into MEAs, test initial performance and durability
- Perform MEA diagnostics (impedance, limiting current methods) to characterize loss mechanisms and guide electrode design
- Perform initial and post-mortem characterization (XRD, XAS, XRF, SEM-EDS, TEM, STEM-HAADF, STEM-EDS) to guide synthetic work and determine effect of structure and composition on performance and durability
- Scale-up and validate MEA performance (5 cm$^2$ $\rightarrow$ 50 cm$^2$)
- Scale-up catalyst synthesis (gram-scale batches)
1. **Brown**: wet chemical synthesis of alloy nanoparticles in high-boiling solvents, followed by thermal annealing to create ordered structures (highest control, lowest scalability).

2. **Penn**: microwave synthesis and rapid thermal annealing (high risk, but may provide enhanced ordering, improved scalability).

3. **LANL**: seed-mediated synthesis by metal salt impregnation in Pt/C, followed by annealing to produce ordered structures (lowest control but highest scalability).

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**Brown synthesis:**
- Partially ordered fct-FePt
- Fully ordered fct-FePt

**Penn synthesis:**
- Localized superheating
- Convection currents
- Conventional heating
- Microwave heating

**LANL synthesis:**
- Impregnate with base metal precursors (e.g., Co salts)
- Protective layer coating
- Thermal processing
- Coating removal
- Supported fct-MPt nanoparticle
**Approach: N-doped Carbon Supports**

Key attributes:

- **N-doped** - improved dispersion and stabilization of nanoparticle catalysts
- **Highly graphitized** - improved durability
- **Fe-free** - avoids Fenton degradation
Accomplishments and Progress: DFT Computation

- Bulk diffusion barrier correlates strongly with potential energy difference between states A and D
- L1₀ intermetallics show much larger diffusion barriers than fcc Pt
- Results suggest that alternative mechanisms (e.g., oxygen place exchange) are more important in controlling base metal leaching – work is ongoing in this area
Mass Activity: 15 min hold at 0.9 V

Mass activity measured during last 1 min of 15 min hold

Catalyst AST: square wave between 0.6 and 0.95 V with 0.5 s rise time

Support AST: triangle wave between 1.0 and 1.5 V at 500 mV/s

H₂/O₂, 500/1000 sccm; 80°C; 100% RH; 150 kPa_\text{abs}; cathode: 0.1 mg_{Pt}/cm²; anode: 0.1mg_{Pt}/cm²

H₂/N₂, 200/200 sccm; 80°C; 100% RH; 150 kPa_\text{abs}; cathode: 0.1 mg_{Pt}/cm²; anode: 0.1mg_{Pt}/cm²
All MEA testing reported here uses MEAs made using standard techniques:

- Water/n-propanol inks, with catalyst and ionomer dispersed by sonication, and deposited by ultrasonic spray
- I/C = 0.9 for high surface area carbon or 0.5 for Vulcan carbon
- GDLs are 29BC (SGL), compressed by 20-25%
- Membranes are Nafion 211
- Testing used 5 cm² differential cells at 500/2000 sccm anode/cathode
- Target electrode loading 0.1 mg Pt/cm² (some sample-to-sample variation as reported in the test results)
- All testing was performed at 150 kPa_{abs} and 100% RH unless noted otherwise
Large Particle L1₀-PtCo
Large Particle L1₀-CoPt with Pt Shell

9 nm Co₄₉Pt₅₁

Loaded on carbon, annealed at 650°C for 6 h in 5% H₂/Ar

After acid leach:

- XRD, coercivity measurements, and TEM all demonstrate high degree of ordering
- Pt shell (~2 atoms thick) after acid leach
Large Particle L₁₀-CoPt with Pt Shell

AST at 60°C, ORR measured at 25 °C

-6 to 0 Current Density (mA cm⁻²)
-6 to 2.5 Mass Activity (A mg⁻¹)

BOL

10K Cycles
20K Cycles
30K Cycles

18.6 % loss in MA

Co₄₃Pt₅₇

Co₃₉Pt₆₁

BOL
EOL: After 30 K Cycles

AST at 60°C, ORR measured at 25 °C
**Large Particle L$_{10}$-CoPt@Pt: MEA Testing**

- **BOL mass activity:** 0.56 A/mgPGM (Target: >0.44 A/mgPGM)
- **Post-30K cycle mass activity (measured at 15 min):** 0.45 A/mgPGM
- **Loss after 30K cycles:** 20% (Target: <40%)
- **Loss at 0.8 A/cm$^2$ after 30K cycles:** 69 mV (Target: <30 mV), but mostly due to flooding
- **Power density:** 0.58 and 0.73 W/cm$^2$ at 150 and 250 kPa (Target: 1 W/cm$^2$)
Large Particle L₁₀-CoPt@Pt: XRD and XRF

- XRD superlattice peaks are slightly smaller after 30K cycle AST, suggesting surface leaching.
- Insignificant shift in peak position, indicating lattice established by ordered core remains unchanged.
- XRF indicates composition change \( \text{Pt}_{0.61}\text{Co}_{0.39} \rightarrow \text{Pt}_{0.71}\text{Co}_{0.29} \), indicating 36% of Co was lost.
STEM-EDS shows ~1 nm Pt shell surrounding Pt$_{50}$Co$_{50}$ core after AST (total particle composition Pt$_{70}$Co$_{30}$)

HAADF-STEM shows highly ordered core remains after AST, coated with a ~0.7-1.0 nm Pt shell (3-4 atoms thick)

Key conclusions:
- Ordered core remains intact even after AST
- Co leaching occurs only from surface, forming Pt shell that protects particle interior from further leaching
- Pt shell is too thick for significant ligand enhancement after AST, but kinetic enhancement due to strain remains even after 30K cycles

J Li et al., Joule 2018
<table>
<thead>
<tr>
<th>Units</th>
<th>Measured</th>
<th>Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass Activity</td>
<td>0.56 A/mgPGM</td>
<td>0.44 A/mgPGM</td>
</tr>
<tr>
<td>Mass Activity after Catalyst AST</td>
<td>0.45 A/mgPGM</td>
<td>0.264 A/mgPGM</td>
</tr>
<tr>
<td>Degradation at 0.8 A/cm² (Catalyst AST)</td>
<td>69 mV</td>
<td>30 mV</td>
</tr>
<tr>
<td>Current Density at 0.8 V</td>
<td>0.32 A/cm²</td>
<td>0.3 A/cm²</td>
</tr>
<tr>
<td>Power at 0.67 V, 150 kPa_{abs}</td>
<td>0.58 W/cm²</td>
<td>1 W/cm²</td>
</tr>
<tr>
<td>Power at 0.67 V, 250 kPa_{abs}</td>
<td>0.73 W/cm²</td>
<td>1 W/cm²</td>
</tr>
<tr>
<td>Cathode PGM Loading</td>
<td>0.105 mg/cm²</td>
<td>0.125 mg/cm²</td>
</tr>
<tr>
<td>Robustness, Cold</td>
<td>0.64 m²/gPt</td>
<td>0.7 m²/gPt</td>
</tr>
<tr>
<td>Robustness, Cold Transient</td>
<td>0.68 m²/gPt</td>
<td>0.7 m²/gPt</td>
</tr>
<tr>
<td>Robustness, Hot</td>
<td>0.19 m²/gPt</td>
<td>0.7 m²/gPt</td>
</tr>
<tr>
<td>ECSA</td>
<td>26 m²/gPt</td>
<td></td>
</tr>
<tr>
<td>ECSA after Catalyst AST</td>
<td>23 m²/gPt</td>
<td></td>
</tr>
<tr>
<td>Crystallite Size (XRD)</td>
<td>7.8 nm</td>
<td></td>
</tr>
<tr>
<td>Crystallite Size after Catalyst AST</td>
<td>9.6 nm</td>
<td></td>
</tr>
<tr>
<td>Particle Size (TEM)</td>
<td>8.9 nm</td>
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</tr>
<tr>
<td>Particle Size after Catalyst AST</td>
<td>8.7 nm</td>
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<tr>
<td>Composition</td>
<td>Pt61Co39</td>
<td></td>
</tr>
<tr>
<td>Composition after Catalyst AST</td>
<td>Pt71Co29</td>
<td></td>
</tr>
</tbody>
</table>

- High mass activity at BOL; only 20% loss after AST
- Excellent ECSA retention (but low ECSA from the start)
- Degradation at 0.8 A/cm² due to increased flooding after AST
- High power performance is too low – probably due to thick electrode (~25 µm)
Small Particle L1₀-PtCo
Results for L10-CoPt@Pt/Vulcan under various conditions:

- **VIR_BOL_250 kPa**
  - Graph showing the relationship between voltage (E) and current density (j) for both BOL and EOL conditions.
  - Graph indicates a decrease in voltage with an increase in current density.

- **Advanced Electrocatalysts Webinar**
  - Image of a material sample with a scale bar of 20 nm.
$L_1^0$-CoPt@Pt/Vulcan
BOL Powder

After 30K cycles in MEA

Ordered particles remain after AST
A1-PtCo/Vulcan, BOL
A1-PtCo/Vulcan, EOL
L1₀-PtCo@Pt/Vulcan, BOL
$L_1^0$-PtCo@Pt/Vulcan, EOL
Accomplishment: Small L1₀-PtCo Particles

BOL Catalyst

After 30K cycles in MEA

Particle structure is similar before and after AST: Pt shell around L1₀-PtCo core
L1₀-PtCo/Vulcan: Fuel Cell Testing

<table>
<thead>
<tr>
<th>Units</th>
<th>Measured</th>
<th>Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass Activity</td>
<td>A/mgPGM</td>
<td>0.60</td>
</tr>
<tr>
<td>Mass Activity Loss</td>
<td>%</td>
<td>40</td>
</tr>
<tr>
<td>Degradation at 0.8 A/cm²</td>
<td>mV</td>
<td>26</td>
</tr>
<tr>
<td>Current Density at 0.8 V</td>
<td>A/cm²</td>
<td>0.41</td>
</tr>
<tr>
<td>Power at 0.67 V, 150 kPa abs</td>
<td>W/cm²</td>
<td>0.89</td>
</tr>
<tr>
<td>Power at 0.67 V, 250 kPa abs</td>
<td>W/cm²</td>
<td>1.10</td>
</tr>
<tr>
<td>PGM Loading</td>
<td>mg/cm²</td>
<td>0.106</td>
</tr>
<tr>
<td>Robustness (cold)</td>
<td></td>
<td>0.94</td>
</tr>
<tr>
<td>Robustness (cold transient)</td>
<td></td>
<td>0.91</td>
</tr>
<tr>
<td>Robustness (hot)</td>
<td></td>
<td>0.92</td>
</tr>
</tbody>
</table>

[1] 30K square wave cycles, 0.6-0.95 V
[2] Cathode

L1₀-PtCo®@Pt/XC-72 catalyst meets or approaches DOE catalyst and MEA targets

High durability of L1₀ ordered PtCo is due to decreased Co leaching – L1₀-PtCo has higher Co content than FCC-PtCo after 30K cycle AST

<table>
<thead>
<tr>
<th></th>
<th>LANL FCC-PtCo</th>
<th>Commercial FCC-PtCo</th>
<th>LANL L₁₀-PtCo</th>
</tr>
</thead>
<tbody>
<tr>
<td>BOL Co%</td>
<td>48%</td>
<td>22%</td>
<td>27%</td>
</tr>
<tr>
<td>EOL Co%</td>
<td>12%</td>
<td>7%</td>
<td>17%</td>
</tr>
</tbody>
</table>
Accomplishment: Ternary $L_{10}$ Development

- DFT results suggest adding a 3rd component (e.g. Ni) to $L_{10}$-PtCo could provide near-optimal O/OH binding energy
- XRD shows Co:Ni:Pt = 1:1:2 gives good ordering
- Based on DFT and XRD, ternary $L_{10}$-CoNiPt looks promising
L1₀-CoNiPt: MEA Testing

- RDE performance promising, but initial MEA results lower than expected
- Ternary L1₀ development still a work in progress

<table>
<thead>
<tr>
<th>Units</th>
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<th>Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass Activity</td>
<td>A/mgPGM</td>
<td>0.33</td>
</tr>
<tr>
<td>Mass Activity Loss [1]</td>
<td>%</td>
<td></td>
</tr>
<tr>
<td>Degradation at 0.8 A/cm² [1]</td>
<td>mV</td>
<td></td>
</tr>
<tr>
<td>Current Density at 0.8 V</td>
<td>A/cm²</td>
<td>0.23</td>
</tr>
<tr>
<td>Power at 0.67 V, 150 kPaabs</td>
<td>W/cm²</td>
<td>0.64</td>
</tr>
<tr>
<td>Power at 0.67 V, 250 kPaabs</td>
<td>W/cm²</td>
<td></td>
</tr>
<tr>
<td>PGM Loading [2]</td>
<td>mg/cm²</td>
<td>0.091</td>
</tr>
</tbody>
</table>

[1] 30K square wave cycles, 0.6-0.95 V
[2] Cathode
Buffalo L$_{12}$-CoPt

Heat treatment of Co ZIF, followed by Pt impregnation and additional heat treatment, produces ordered PtCo on N-doped carbon.

- High fuel cell performance in unoptimized MEA

XX Wang et al., Nano Letters 2018
Buffalo Mn-Hydrogel Supports

- Polymer hydrogels carbonized in presence of Mn have highly graphitic structure
- Graphitic structure prevents C corrosion, enhancing fuel cell stability
Accomplishment: Mn-Hydrogel Supports

- Graphitic structure reduces corrosion
- Hydrogel supports meet or approach durability targets with pure Pt
- Need more active catalyst for performance targets

<table>
<thead>
<tr>
<th>Units</th>
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<th>Target</th>
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</thead>
<tbody>
<tr>
<td>Mass Activity A/mgPGM</td>
<td>0.18</td>
<td>0.44</td>
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<tr>
<td>Mass Activity Loss [%]</td>
<td>39</td>
<td>40</td>
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<tr>
<td>Degradation at 1.5 A/cm² [1]</td>
<td>31</td>
<td>30</td>
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<tr>
<td>Current Density at 0.8 V</td>
<td>0.14</td>
<td>0.3</td>
</tr>
<tr>
<td>Power at 0.67 V, 150 kPa&lt;sub&gt;abs&lt;/sub&gt;</td>
<td>0.49</td>
<td>1</td>
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<tr>
<td>Power at 0.67 V, 250 kPa&lt;sub&gt;abs&lt;/sub&gt;</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>PGM Loading [2]</td>
<td>0.13</td>
<td>0.125</td>
</tr>
</tbody>
</table>

[1] 5K triangle wave cycles, 1.0-1.5 V
[2] Cathode

Qiao et al., Energy Environ. Sci., 2019, Accepted
**L1₀-PtCo/Hydrogel**

Hydrogel-based support enables improved dispersion of 2-4 nm L1₀-PtCo

Small L1₀ ordered PtCo

PtCo particles on folded graphene sheets

July 31, 2019 Advanced Electrocatalysts Webinar
L1₀-PtCo/Hydrogel

- Combination of LANL PtCo technology and Buffalo support technology produces extremely high mass activity and good durability
- L₁₀-PtCo on hydrogel support: small, monodisperse, ordered
- MEA optimization needed to improve power density

Primary goal of support work is to improve performance and durability through better dispersion of intermetallic nanoparticles. Meeting support durability targets is secondary goal.

<table>
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<tbody>
<tr>
<td>Mass Activity</td>
<td>A/mgPGM</td>
<td>0.79</td>
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<tr>
<td>Mass Activity Loss [1]</td>
<td>%</td>
<td>37</td>
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<tr>
<td>Degradation at 0.8 A/cm² [1]</td>
<td>mV</td>
<td>31</td>
</tr>
<tr>
<td>Current Density at 0.8 V</td>
<td>A/cm²</td>
<td>0.34</td>
</tr>
<tr>
<td>Power at 0.67 V, 150 kPa abs</td>
<td>W/cm²</td>
<td>0.77</td>
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<td>Power at 0.67 V, 250 kPa abs</td>
<td>W/cm²</td>
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</tr>
<tr>
<td>PGM Loading [2]</td>
<td>mg/cm²</td>
<td>0.108</td>
</tr>
<tr>
<td>ECSA</td>
<td>m²/gPt</td>
<td>72</td>
</tr>
<tr>
<td>ECSA after Catalyst AST</td>
<td>m²/gPt</td>
<td>37</td>
</tr>
</tbody>
</table>

[1] 30K square wave cycles, 0.6-0.95 V  
[2] Cathode

0.108 mg_{PGM}/cm², N211, 150 kPa_{abs}, 80°C, 100%RH

30K square wave cycles, 0.6-0.95 V
Intermetallic L1₀-CoPt developed in this project is compatible with multiple carbon supports (XC-72, Ketjen, and hydrogel-based carbons) and has high performance in large and small batches.
### Project Status

<table>
<thead>
<tr>
<th>Units</th>
<th>L1₀-PtCo/XC-72</th>
<th>L1₀-PtCo/Hydrogel</th>
<th>Target</th>
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<tbody>
<tr>
<td>Mass Activity</td>
<td>0.60</td>
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<td>1.10</td>
<td>1</td>
<td>1</td>
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<tr>
<td>PGM Loading [2]</td>
<td>0.106</td>
<td>0.108</td>
<td>0.125</td>
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<tr>
<td>Robustness (cold)</td>
<td>0.94</td>
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<td>0.7</td>
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</table>

[1] 30K square wave cycles, 0.6-0.95 V  
[2] Cathode

- L1₀-PtCo/XC-72 meets most DOE catalyst and durability targets; further work on power density underway
- L1₀-PtCo/Hydrogel provides path to higher mass activity
- L1₀ ordering improves durability by decreasing Co leaching; ordering is retained even after 30K cycle AST in MEA
Future Work

- Increase high-current performance and durability through improved L1₀-PtCo dispersion – to be achieved via improved control of synthesis, improved N doping in supports
- Extend gram-scale synthesis to multi-gram batches that match performance of small batches
- Perform MEA optimization on the two most promising catalysts (small particle L1₀-CoPt/XC-72 and L1₀-CoPt/Hydrogel Carbon)
- Scale up MEA testing from 5 cm² to 50 cm²
Summary

• Intermetallic PtCo catalysts provide high activity and durability in MEAs
• Ordered catalysts with Pt skins can keep high Co content even after durability testing
• L1₀ ordering still apparent even after 30,000 voltage cycles
• Best catalysts can meet DOE performance and durability targets

• MEA testing is critical to evaluate ORR catalysts!
• We collaborate to test promising catalysts – contact me at spendelow@lanl.gov
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Prof. Shouheng Sun
Shubham Sharma
Prof. Andrew Peterson

Oak Ridge National Lab
Dr. David Cullen
Dr. Karren More

University at Buffalo
Zhi Qiao
Prof. Gang Wu
Question and Answer

• Please type your questions to the chat box. Send to: (HOST)
Thank you

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