The Japanese Containerless Experiments
National Aerospace Laboratory
Hisao Azuma

1. Drop Dynamics Research in NAL
   a) Acoustic Levitation

   * FMPT related activity
      Liquid drop experiment by a tri-axis acoustic levitator in
      the Japanese First Material Processing Test (FMPT) is to be

   Objective of the experiment
   - Stable positioning of a liquid drop
   - Rotation of a drop
   - Deformation of a liquid drop
   - Stability of a liquid membrane

   Experiments on the Earth
   - Levitation and rotation of light weight samples (styrofoam spheres)
   - Deformation of a drop and formation of a liquid film

   Parabolic flight test
   - Separation of a drop from an injection needle in an acoustic chamber
   - Determination of experimental parameters to position a drop in low gravity

   Liquid drop experiment facility
   Levitation box dimension 100W*100H*110D
   Acoustic pressure 141-148 dB
   Speaker input power 10 Wmax
   Frequency 1400-1700 Hz
   Drop size 10, 19, 23 mm dia.
* High levitating force levitator
  - Levitation of large sized liquid drop and membrane on the earth
  - High ambient pressure

b) Large amplitude drop oscillation
Realization of three-dimensional spherical large amplitude oscillation, tetrahedron-tetrahedron, hexahedron-octahedron, dodecahedron-icosahedron by using drop tower. The oscillations were caused with surface tension variation by applying alternating current voltage.

2. Optical Materials Processing in an Acoustic Levitation Furnace in Industrial Research Institute, Osaka

"Preparation of Optical Materials used in Non-visible region" to be conducted in the FMPT

-65CaO-25Ga2O3-10GeO2(near infrared transmitted oxide glass) was chosen
-1400°C and platinum cage for preheating is needed

Parabolic flight test
- Levitation of heated sample was made sure
3. Electrostatic Levitator Development by Melco and IHI

a) Mitsubishi Electric Corporation

Development status
- Levitation and rotation of 0.1g platinum coated glass shell by a double ring type levitator
- Position data of 120Hz
- Levitation of 50g solid (metal and glass) will be tried soon by parabolic flight

Aimed performance goal
- Disturbance given to a sample should be less than 10^{-6}g
- Sample should be heated up to 2500°C (3kw AC power)
- Sample size should be larger than 20mm dia.

An important technology - Microwave Discharge Lamp
- High temperature in whole sphere of arbitrary size
- Choice of arbitrary gas inside the lamp to get desired wavelength light

b) Ishikawajima-Harima Heavy Industries Co., Ltd.

Development status
- Levitation of solid sample (4mm in dia. 1.5mg) by a levitator with quadrupole electrodes and a couple of spherical electrodes

Configuration of two dimensional alternating quadrupole levitation apparatus

![Diagram of two dimensional alternating quadrupole levitation apparatus]
THERMOPHYSICAL PROPERTIES OF SOLIDS AND LIQUIDS
(MAINLY METALS AND ALLOYS)

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WHY THERMOPHYSICAL PROPERTIES ARE NEEDED

(a) ENGINEERING DESIGN PARAMETERS

- Turbine blade alloys
- Ti forgings
- Al alloys
- Composites

(b) MATERIALS PROCESSING

- On Earth: Solid state combustion synthesis:
  \[ \text{Ti} + \text{C} = \text{TiC} + \text{Energy} \]

- In Microgravity environments:
  1. On Moon
     \[ \text{MO}_x + \text{F}_2 \rightarrow \text{MF}_y + \text{O}_2 \]
     \[ \text{MF}_y(I) \rightarrow \text{M} + \text{F}_2 \]
     Breathe \( \text{O}_2 \); Recycle \( \text{F}_2 \)
  2. Space Station
  3. Shuttle / Satellites
  4. Wake Shield experiments
TYPES OF MATERIALS

REFRACTORY METALS

ALLOYS-SUPERALLOYS

Inconel
TiAlx, NiAlx, etc...

GRAPHITE

BINARY CARBIDES

SiC, B4C, Al4C3
TiC, VCx, ...
ZrC, NbCx, MoCx
HfC, TaCx, WCx
ThCx, UCx, ...

BINARY SILICIDES

MoSi2, WSi2, etc...

BINARY BORIDES

TiB2, ZrB2, TaBx, ...

BINARY NITRIDES

Si3N4, BN, AlN
TiNx, VNx
ZrNx, etc...

TERNARY COMPOSITIONS:

METAL OXYNITRIDES
METAL OXYBORIDES
METAL OXYCARBIDES, etc...
CURRENT STATE OF KNOWLEDGE

<table>
<thead>
<tr>
<th>CLASS OF MATERIALS</th>
<th>SOLIDS</th>
<th>LIQUIDS</th>
<th>GASES</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metals</td>
<td>Good</td>
<td>Good</td>
<td>Excellent*</td>
</tr>
<tr>
<td>Alloys</td>
<td>Good</td>
<td>Fair</td>
<td>Good</td>
</tr>
<tr>
<td>Graphite</td>
<td>Good</td>
<td>Poor</td>
<td>Fair</td>
</tr>
<tr>
<td>Carbides</td>
<td>Good</td>
<td>Poor</td>
<td>Good</td>
</tr>
<tr>
<td>Silicides</td>
<td>Fair</td>
<td>Poor</td>
<td>Fair</td>
</tr>
<tr>
<td>Borides</td>
<td>Fair</td>
<td>Poor</td>
<td>Fair</td>
</tr>
<tr>
<td>Oxides</td>
<td>Good</td>
<td>Fair</td>
<td>Good</td>
</tr>
<tr>
<td>Nitrides</td>
<td>Fair</td>
<td>Poor</td>
<td>Fair</td>
</tr>
</tbody>
</table>

*EXCEPT FOR LASER ZAP OR EXPLODING WIRES

- TERNARY AND MORE COMPLICATED SYSTEMS ARE NOT WELL STUDIED.

- THEORIES ARE NOT EVEN ADEQUATE FOR METALS, VERY PRIMITIVE FOR LIQUID ALLOYS AND REFRACTORY COMPOUNDS.
PROPERTIES NEEDED

\[ \text{Cp}(T) \]
\[ (H_T - H_{298}) \]
\[ \Delta H_{\text{fusion}} \]
Phase Diagram
\[ \varepsilon(\lambda, T) \]
\[ \varepsilon_{\text{total}}(T) \]
(Calorimetry, Pyrometry)

\[ \rho(T) \]
CTE(T) for solids and liquids
\[ \Delta V_{\text{fusion}} \]
Supercooling, Nucleation, and Crystallization
(Fast Photography of weighed drops)

Surface Tension as \( f(T) \)

Viscosity as \( f(T) \)

Melting and Freezing
(Fast Photography of oscillating droplets)

Thermal Conductivity as \( f(T) \)

Thermal Diffusivity as \( f(T) \)

Resistivity as \( f(T) \)

Magnetic Properties as \( f(T) \)

* Laser flash heating
ELECTROMAGNETIC LEVITATION is versatile and provides rapid heating for good conductors in vacuum or in selected atmospheres

- Convenient for good conductors: metals, alloys, carbides, borides, etc.

- Heat C or SiC but not levitate

- ZrO₂, HfO₂, UO₂, etc. can be heated inductively after pre-heating

- Al₂O₃, SiO₂, NaCl, etc. neither heat nor levitate

ACOUSTIC LEVITATION

POOR ELEC. CONDUCTORS

LOW VP’S

GAS JET LEVITATION

POSSIBLE CONTAMINATION

MICROGRAVITY ENVIRONMENT

EVERYTHING LEVITATES

RADIATIVE, LASER OR INDUCTION HEATING
LIMITATIONS OF ELECTROMAGNETIC LEVITATION

1. Must be good conductor.

2. Must have adequate surface tension.

3. Must have low VP.
UNSOLVED PROBLEMS IN DETERMINING THERMOPHYSICAL PROPERTIES OF LIQUID METALS/ALLOYS AT HIGH TEMPERATURES

- Contamination - Apparatus
  - Atmospheres

- Calibration Standards - Precision (± 0.5%)
  - Accuracy

- Reliable T(t) and Standards for T > 2000 K

- Pre-Melting/Post-Melting Phenomena

- Clusters in Liquids?

- Are There Defects in Liquids?

- Super-Cooling; Amorphous Phases; Crystallization

- Electronic Effects: Is \( \partial \) a f(T)?

- Limits on \( T_{\text{max}} \) by VP

- Vaporization Losses as f(T, t, Metal)

- Lack of a Comprehensive Theory for Liquid Metals/Alloys: \( R(T); C_p(T); \varepsilon(T); \)
  - Hall Effect
SPECIAL NEW TECHNIQUES

- Pulsed Laser Heating & EM Levitation
- Polarized Laser Pyrometry Yields \( \varepsilon(\lambda, T, t) \) and True \( T \) as \( f(t) \)
- High-Speed Photography of Levitated and Falling Drops
- Hybrid Levitators (EM, Acoustic, Gas Jet)
GOALS OF LEVITATION STUDIES

THERMODYNAMIC PROPERTIES

\( C_p(T), \Delta H_{\text{fusion}}, C_p(T) \)

PHYSICAL PROPERTIES

Density as \( f(T) \)
Thermal expansivity
Emissivities as \( f(T) \)
Surface tension as \( f(T) \)
Viscosity as \( f(T) \)

QUESTIONS

Is \( C_p \) for liquid metals:

a. Greater than, equal to, or less than \( C_p \) for the solid?

b. Increasing, constant, or decreasing with increasing \( T \)?

c. Appreciably higher at 5000 K than at 3000 K for liquid Mo?

d. Approximately 3\( R \), 5\( R \), 6\( R \),... for liquids at high \( T \)?
RECENT LEVITATION STUDIES AT RICE UNIVERSITY

LIQUID SILVER \((1281 \, K < T < 1549 \, K)\)

\[
(H_T - H_{298}) = 32.644 \, T - 2944.9 \, \text{J/gram.atom} \\
C_p^l = 32.64 \pm 2.06 \, \text{J/Gram.atom K} \\
\Delta H_{\text{fusion}} = 10916 \pm 435 \, \text{J/Gram.atom} \\
\epsilon_{650 \, \text{nm}} = 0.11 \pm 0.10
\]

LIQUID GALLIUM \((587 < T < 1630 \, K)\)

\[
(H_T - H_{298}) = 26.460 \, T - 7677.0 \, \text{J/gram.atom} \\
C_p^l = 26.46 \pm 0.71 \, \text{J/Gram.atom K} \\
\epsilon_{645 \, \text{nm}} = 0.14 \pm 0.10
\]

Also Studies of:

TUNGSTEN, BRASS ALLOYS, SUPERALLOYS
SPECTRAL EMISSIVITIES OF LIQUID METALS AS A FUNCTION OF WAVELENGTH
SPECTRAL EMISSIVITIES OF LIQUID METALS AS A FUNCTION OF WAVELENGTH
### Spectral Emissivities of Pt (liquid) as a Function of Temperature for Various Wavelengths

<table>
<thead>
<tr>
<th>Wavelength (nm)</th>
<th>$a$</th>
<th>$b \times 10^2$</th>
<th>Temp. Range (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>486</td>
<td>0.646</td>
<td>-7.41</td>
<td>2266-2646</td>
</tr>
<tr>
<td>514.5</td>
<td>0.831</td>
<td>-17.12</td>
<td>2307-2449</td>
</tr>
<tr>
<td>632.8</td>
<td>0.270</td>
<td>-4.800</td>
<td>2006-2475</td>
</tr>
<tr>
<td>1064</td>
<td>0.356</td>
<td>-2.998</td>
<td>2109-2386</td>
</tr>
</tbody>
</table>