

Advanced Solder Materials for High-Temperature Application **HISOLD**
Expression of Interest for COST MP0602
WG2

**Factors affecting properties of liquid and semi-liquid
Pb-free solder alloys for high-temperature applications**

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- Foundry Research Institute, Cracow, POLAND



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TASKS

1) Alloy production (including IMC) by **liquid phase routes using different cooling rates, including analysis of chemical composition of the alloys**
(Dr. A. Karwinski, P. Darlak)

2) Production of solder/substrate couples under different processing conditions, i.e.:

- atmosphere, flux
- temperature
- time
- cooling rate
- type of contact

(Prof. N. Sobczak, R. Nowak, A. Kudyba)

4) Heat treatment of solder alloys and joints

(Prof. J. Sobczak, P. Darlak)

TASKS

4) Experimental investigation of high-temperature phenomena taking place in *liquid* and *semi-liquid* alloys as well as during their interaction with solid substrates, i.e. :

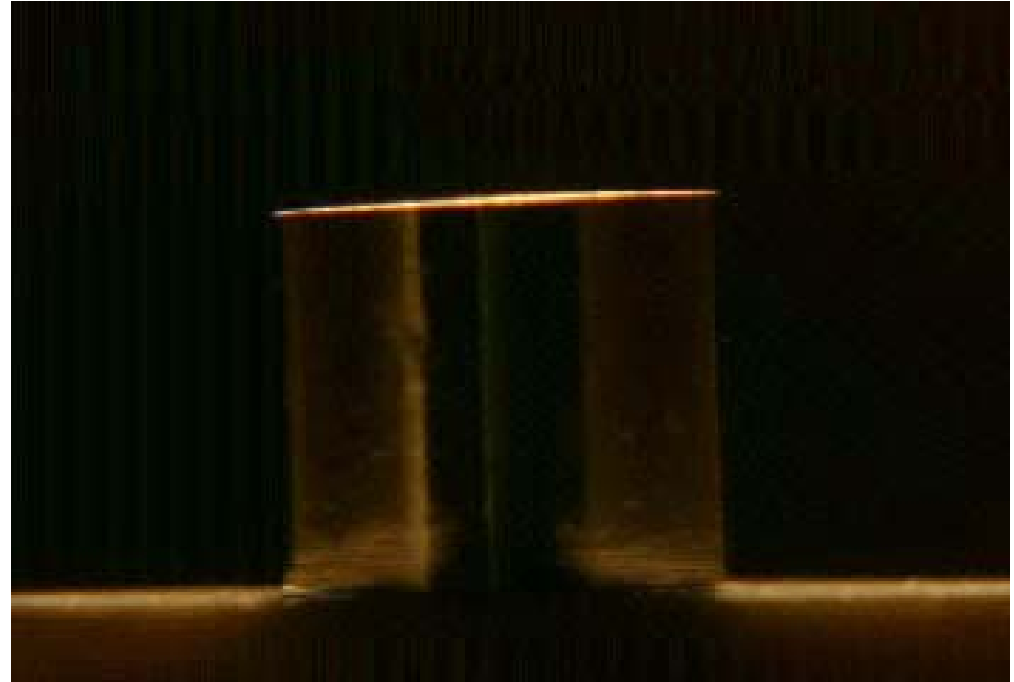
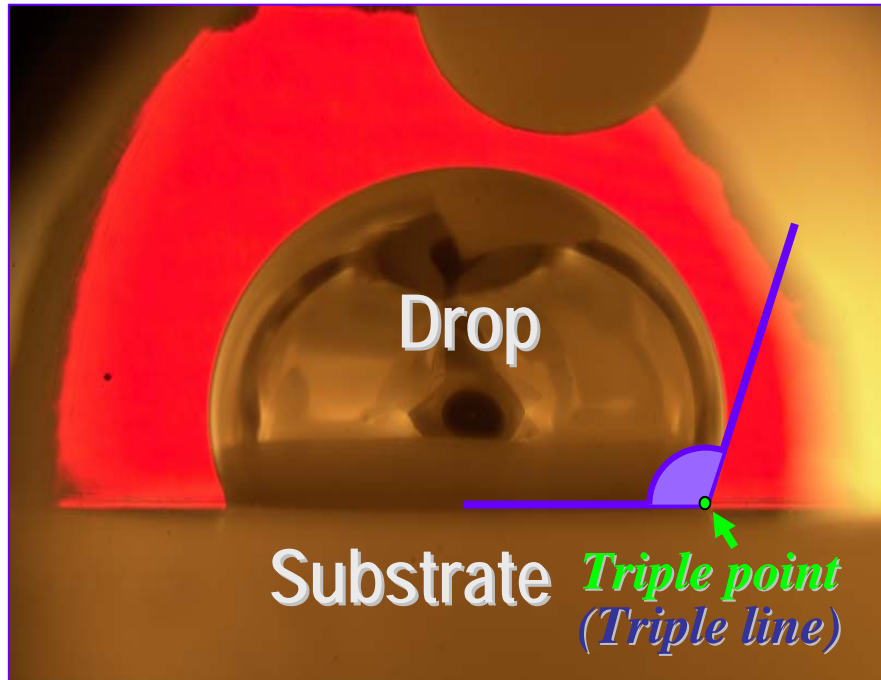
- **Solidification/melting**
- **Undercooling**
- **Phase transformations**

(Prof. J. Sobczak, Dr. A. Gazda, P. Darlak)

- **Wetting**
- **Spreading**
- **Shrinkage/expansion during cooling/heating**
- **Reactivity and interfaces in solder/substrate couples**

(Prof. N. Sobczak, R. Nowak, A. Kudyba)

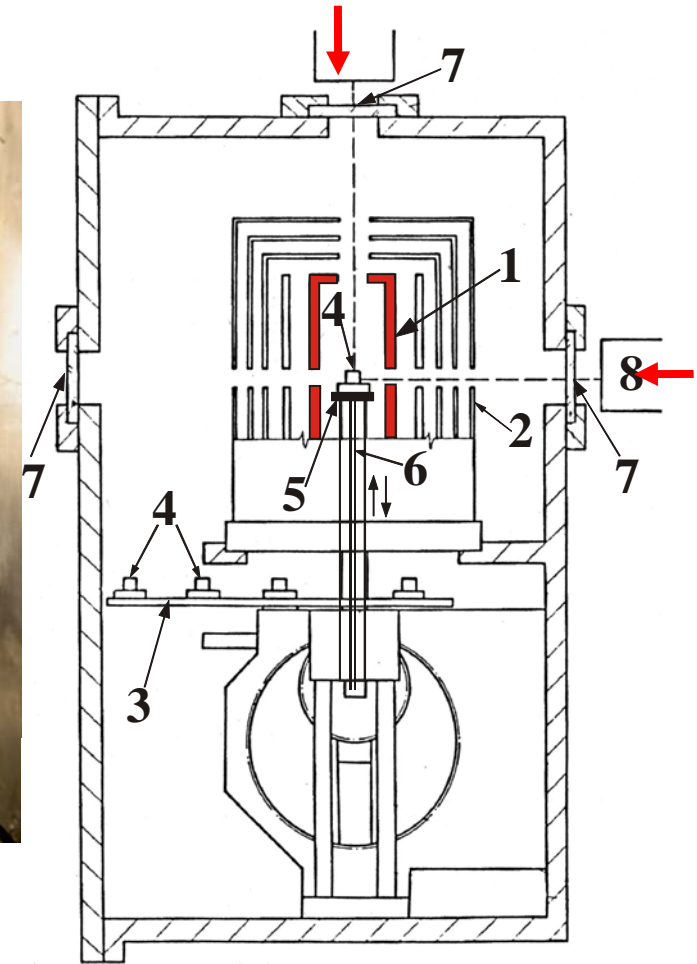
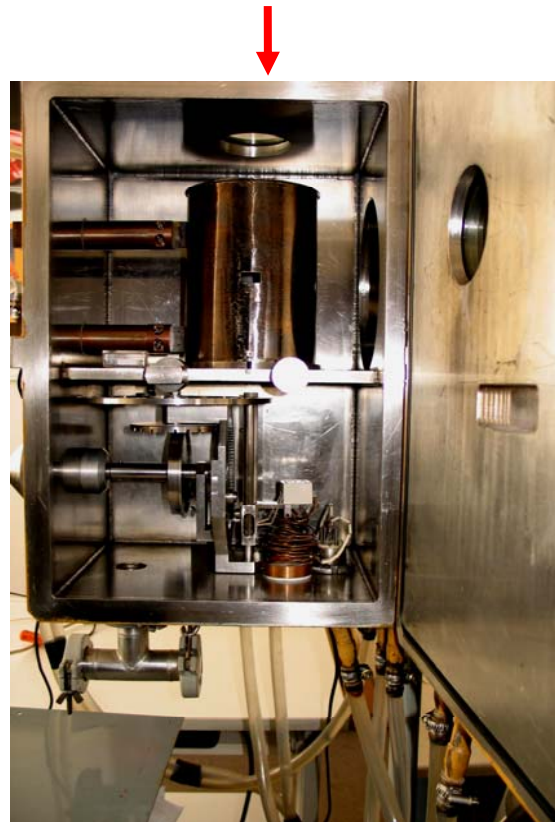
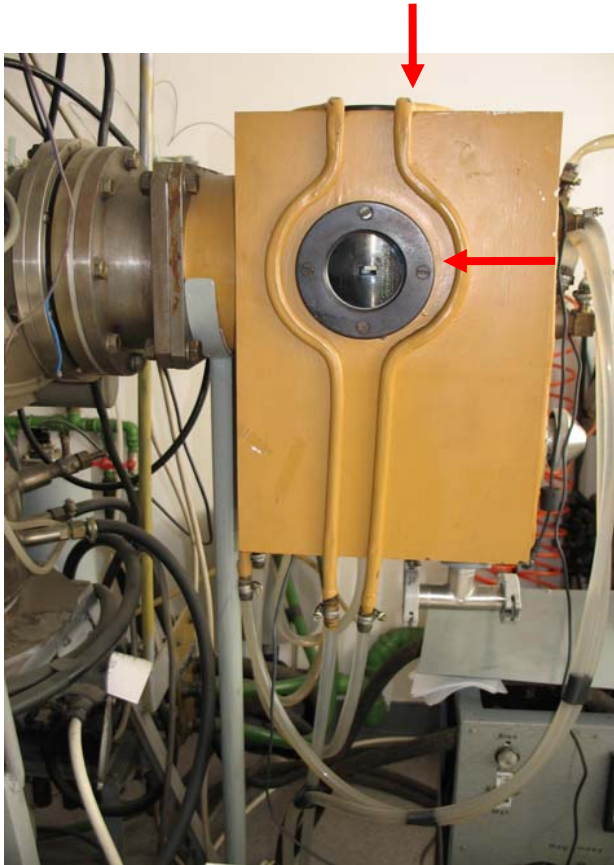
Sessile drop method



1. Properties of liquid phase:
 - surface tension
 - density
 - volume change during heating/cooling
 - shrinkage/expansion during solidification
2. Properties of a liquid/solid couples
 - Wettability kinetics
 - Spreading kinetics
 - Reactivity
 - Hysteresis of contact angle

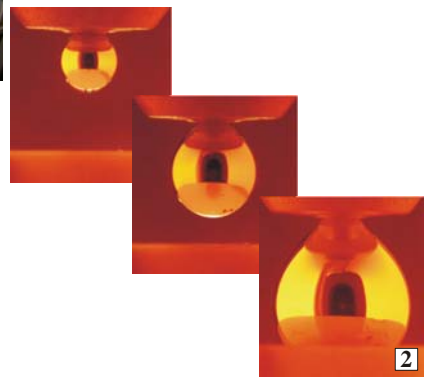
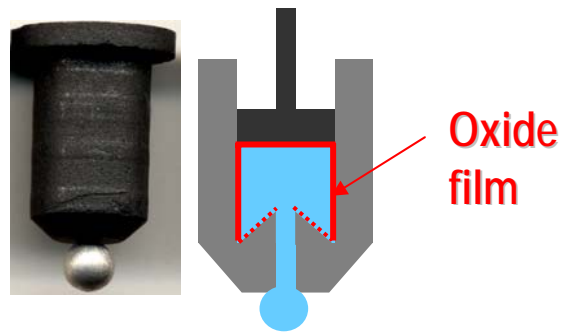
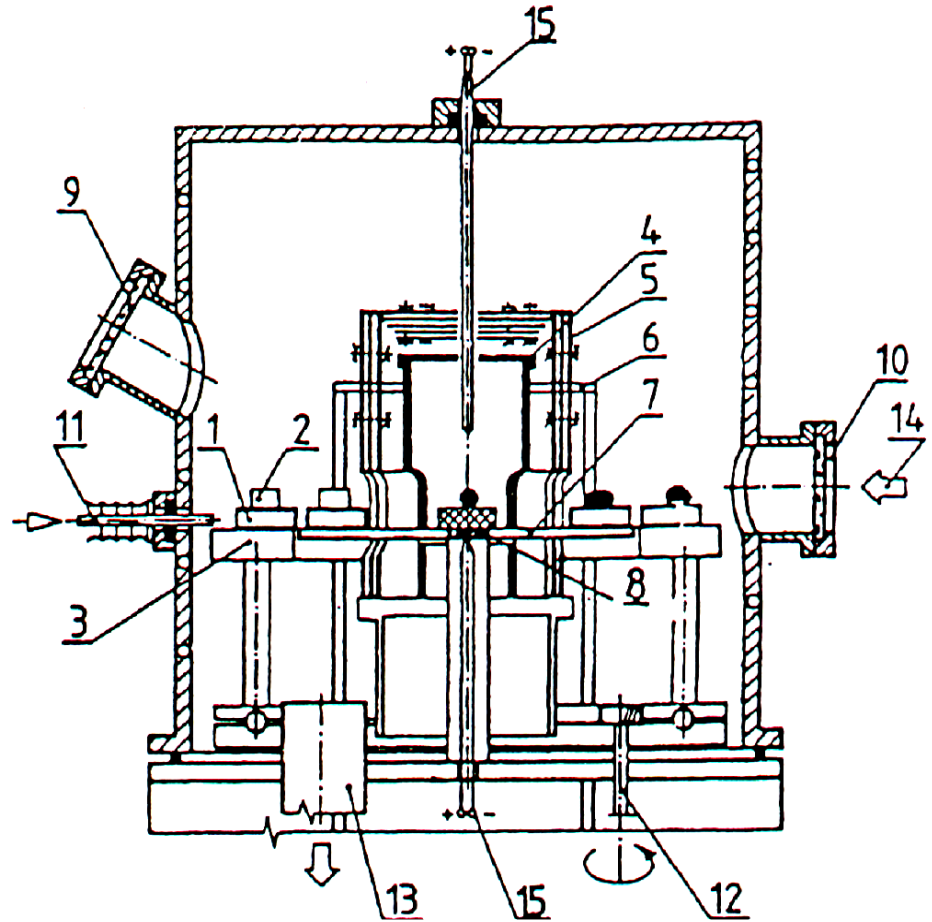
Classical sessile drop method
Contact Heating (CH) procedure

J. Galon, N. Sobczak, R. Ryglicki, Polish patent PL 50513; 28.10.1988



- **Temperatura up to 500°C**
- **Contact heating procedure**
- **Separated cold and hot parts**
- **Side-view and top-view**
- **Loading samples to and from experimental table**

N. Sobczak, A. Kazakov, J. Schmidt,
 Polish patent. PL 166953; 26.07.1991

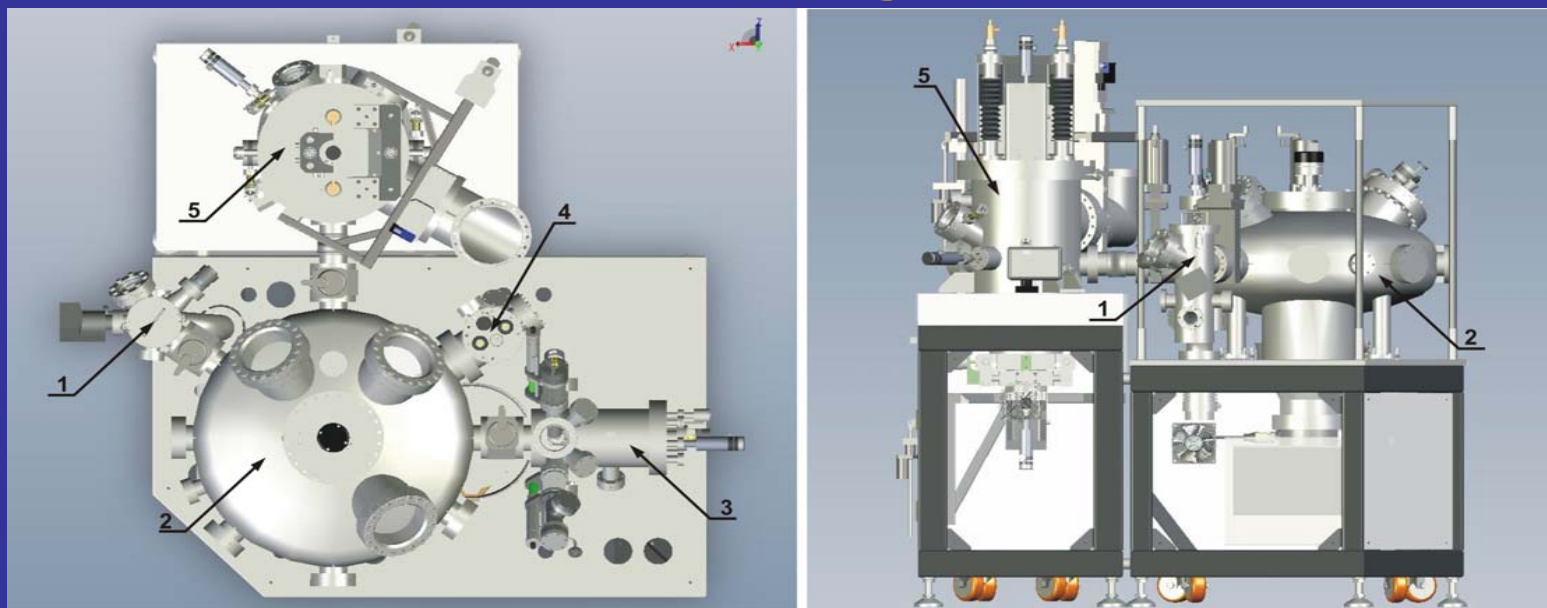


- ✓ Temperatura up to 1400°C
- ✓ Separated hot and cold parts
- ✓ Loading samples to and from experimental table
- ✓ Residual gas analyzer
- ✓ Capillary purification technique (but at constant height only!)

Experimental complex for high temperature studies



Experimental complex for high temperature studies



1- vacuum chamber for the first stage of sample preparation by preheating in vacuum up to 200°C in order to remove adsorbed gases;
2- chamber for transferring the samples between the chambers using a manipulator that allows to bring the samples of different sizes and various shapes;
3- analytical chamber containing a) Auger spectroscope for surface characterization of examined materials before and after high temperature treatment, b) ion beam for etching/cleaning samples and removal of surface films from examined samples;
4- portable chamber (vacuum "traveling-bag") for storage and collection of specimens after testing;
5- experimental chamber for high-temperature studies of materials in solid, semi-solid or molten states, containing a) experimental table with rotation and up-and-down movement, the heater and screens with up-and-down movement, additional windows for observation and recording, b) Quadrupole residual gas analyzer for real time recording of chemical composition of vacuum, c) capillary with up-and-down movement (for capillary purification procedure or for removal of a drop after testing, for example in order to "open" the interface/reaction products at the interface), d) manipulator, located under a drop/substrate couple, which allows to delivery another substrate (sandwiched drop procedure) or alloying additions (*in situ* alloying in vacuum chamber), e) automatic real-time temperature control by 4 thermocouples located in selected positions.

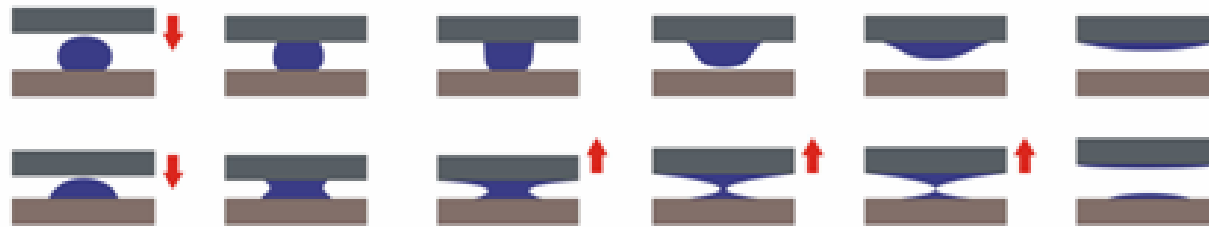
Advantages

Possibility for testing by different methods and procedures

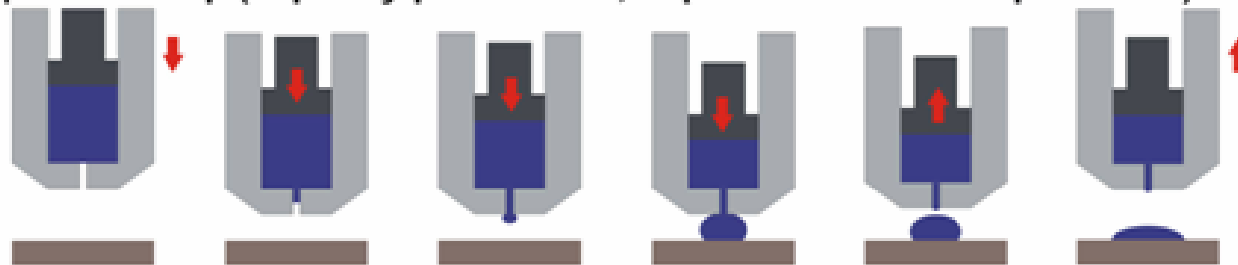
1. Classical sessile drop method



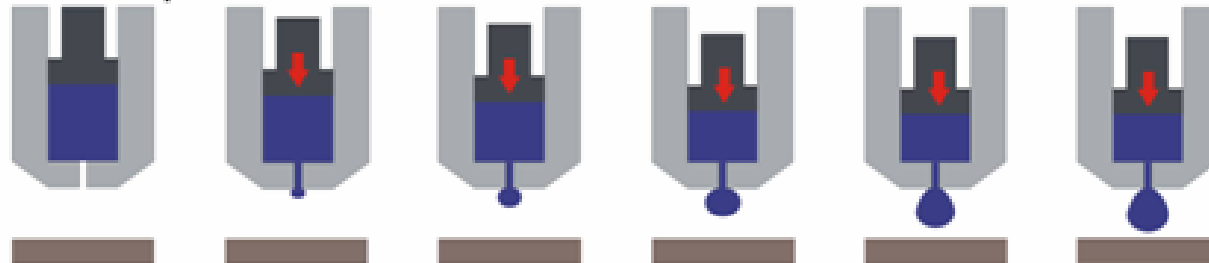
2. Transferred drop (sandwiched method, double plates method)



3. Dispensed drop (capillary purification, improved sessile drop method)



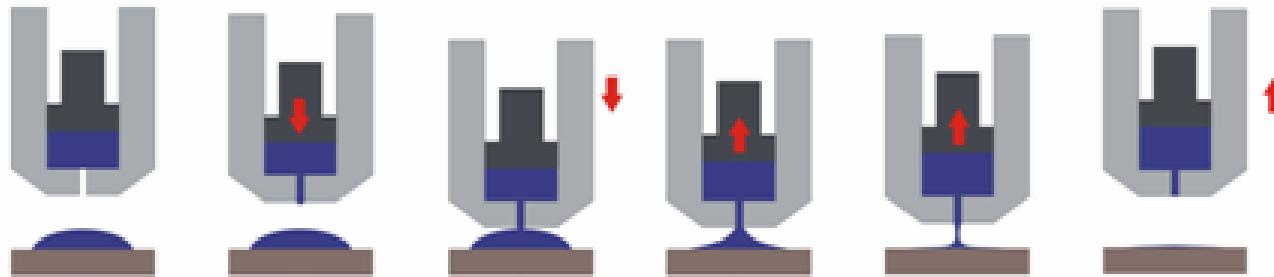
4. Pendant drop



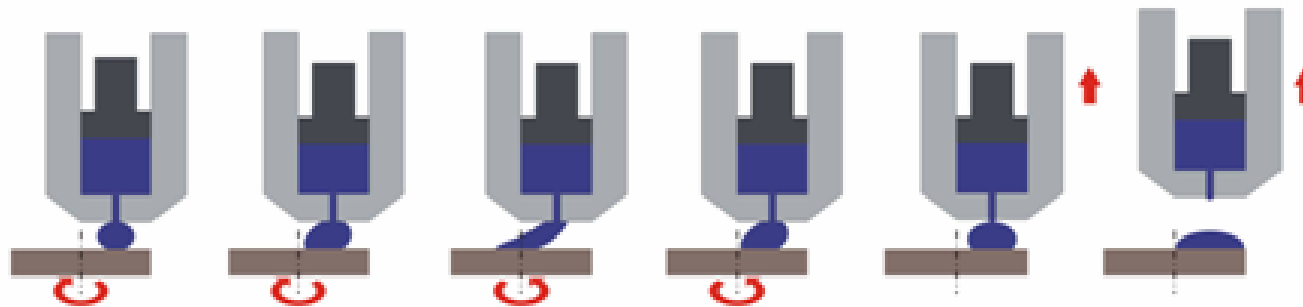
Advantages

Possibility for testing by different methods and procedures

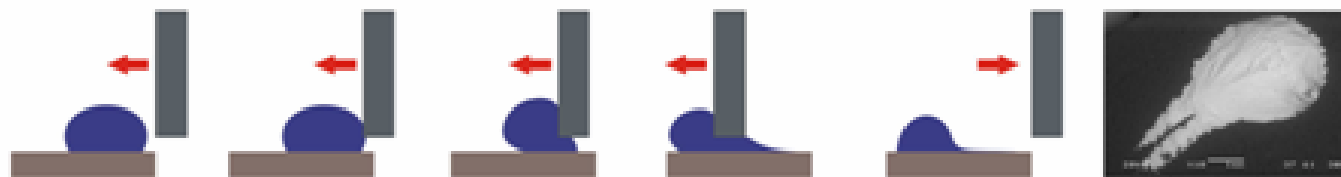
5. Metal suction



6. Substrate rotation



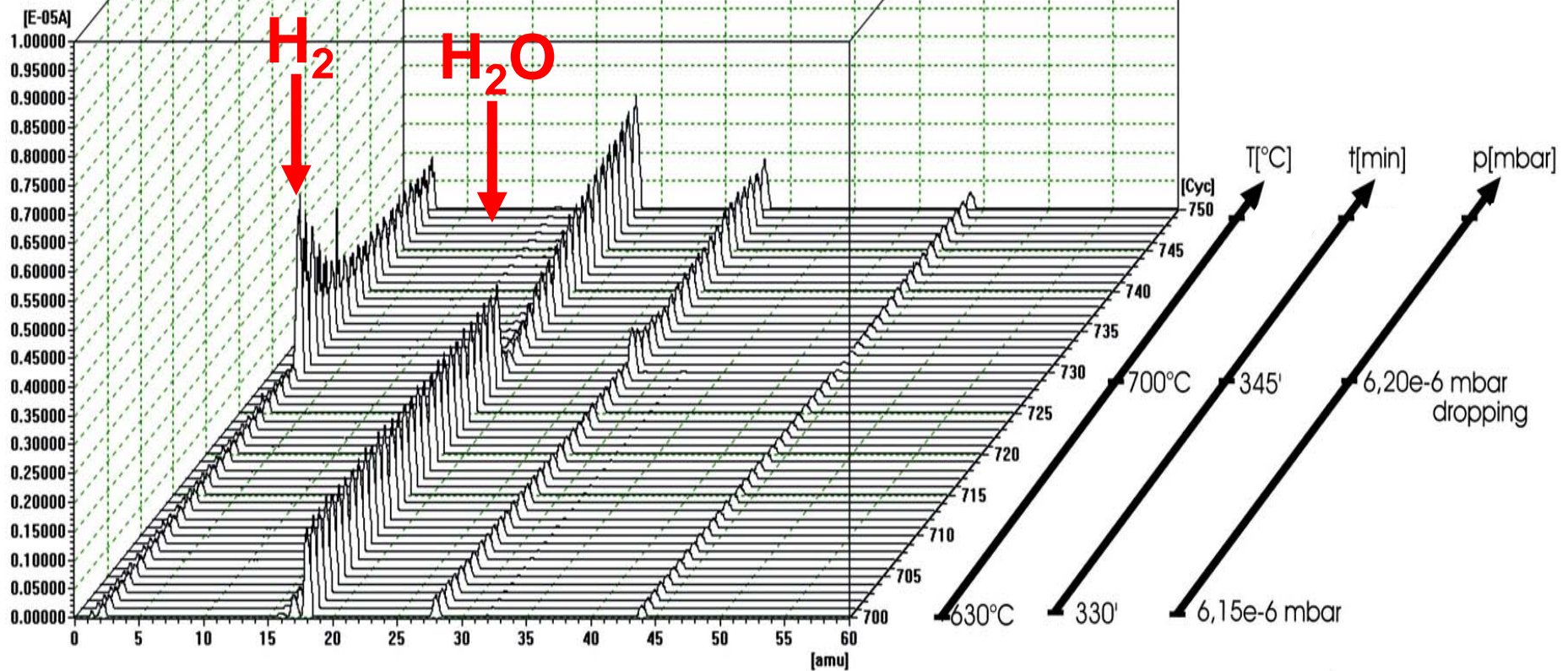
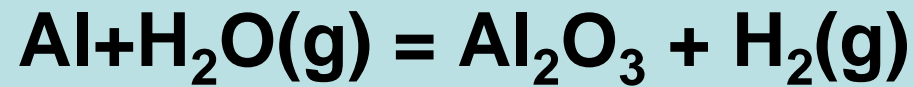
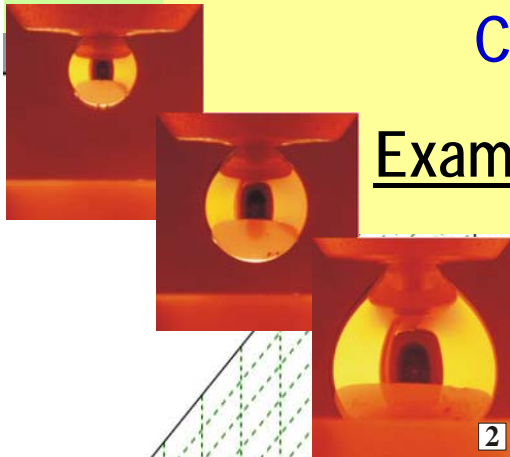
7. Drop pushing



Advantages

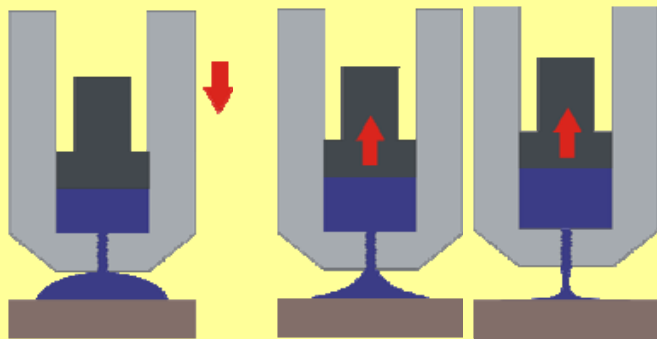
Control of residual gases in vacuum chamber

Example 1: Interaction of Al drop with residual water vapour



Example 2: Application of drop sucking procedure for *in situ* „opening” interfaces

Effect of **carbon coating** on Al_2O_3 on its interaction with AlSi22 alloy (1000°C)



Carbon coating improves wetting due to the formation of wettable reaction product

