



Experimental investigation of physical properties and melts states in Bi-, Ga- and Sn-based multicomponent alloys

Valeriy Sidorov

Ural State Pedagogical University, Ekatherinburg, Russian

Ekaterinburg, the main street



Ekaterinburg, the main square



Ekaterinburg, down town



Ural



Ekaterinburg, July, 2007



Properties under investigation:

- density (by gamma-penetration method);
- viscosity (by damping oscillating method);
- electrical resistivity (by contactless method in rotating magnetic field);
- ultra sound velocity (by pulse-phase method);
- magnetic susceptibility (by Faraday's method);
- optical characteristics (by elipsometric method);
- surface tension (by sessile drop method);
- laboratory for chemical and spectral analysis

Density studies by gamma-penetration method

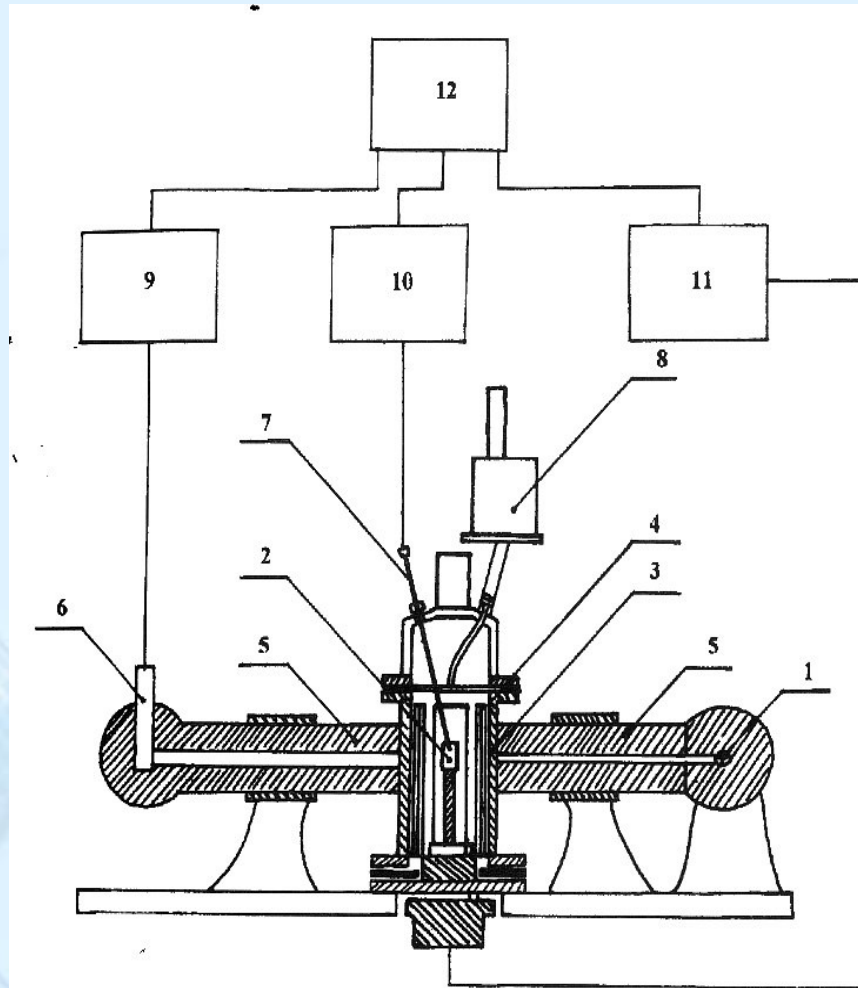
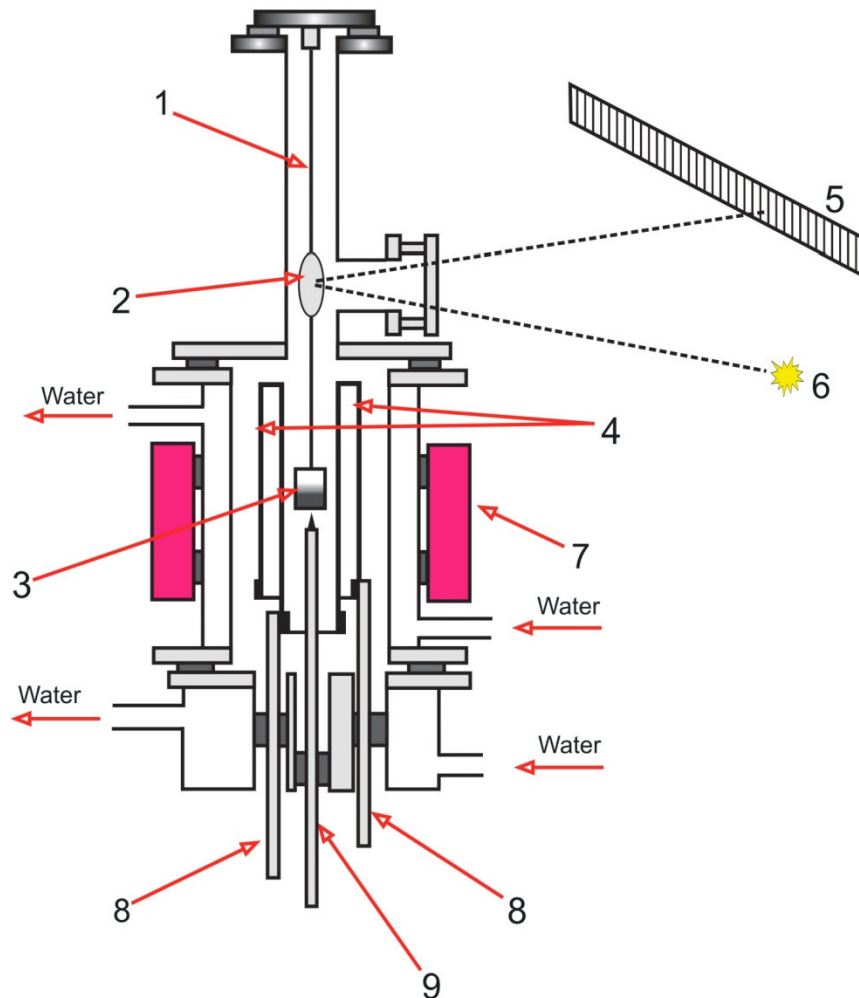


Figure 1.2 Experimental setup for measuring density of high-temperature melts: (1) gamma-ray radiation source, (2) crucible filled with melt, (3) water-cooled housing of the vacuum resistance furnace, (4) molybdenum heater, (5) collimators, (6) gamma-ray scintillation detector, (7) tungsten-rhenium thermocouple, (8) metering tank for introducing alloying additives into melt, (9) gamma-ray beam intensity measuring unit, (10) melt temperature measuring unit, (11) heater controller, (12) computer.

Viscosity studies by torsion oscillations method

equipment for measuring viscosity by the damping of torsion oscillations



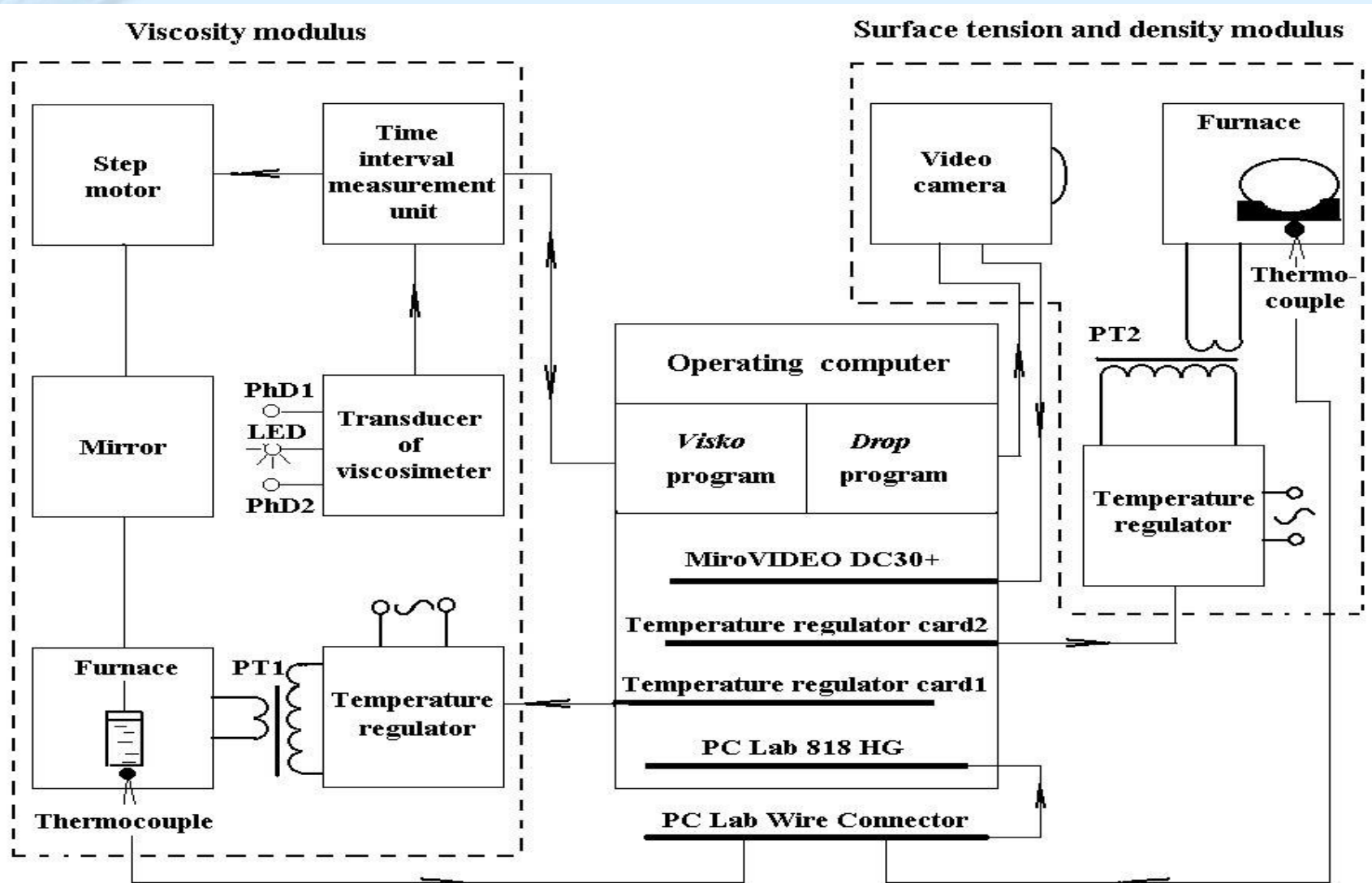
- (1) pendulum thread,
- (2) mirror for detecting oscillations, (3) crucible with specimen,
- (4) graphite heater,
- (5) oscillating amplitude scale,
- (6) source of narrow light beam,
- (7) winding for creating magnetic field,
- (8) current guides for the heater,
- (9) tungsten-rhenium thermocouple

Electrical resistivity studies in rotating magnetic field

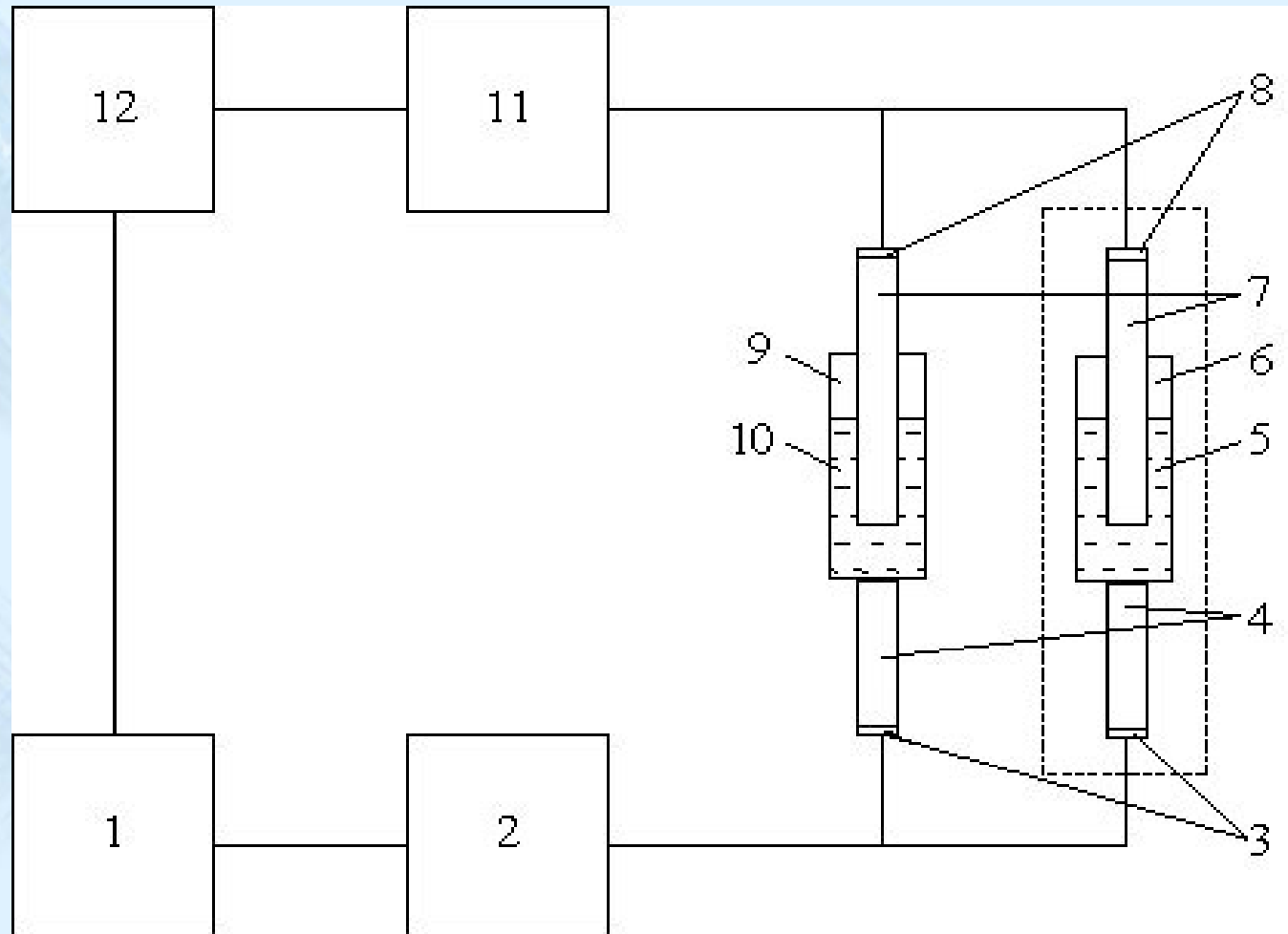
The resistance is determined from the angle of twist of the sample suspended on the elastic thread under the influence of the rotating magnetic field.

It can be measured at the same equipment as viscosity

The blockscheem of the device for viscosity, surface tension and density measurements



Ultra-sound velocity studies by pulse-phase method



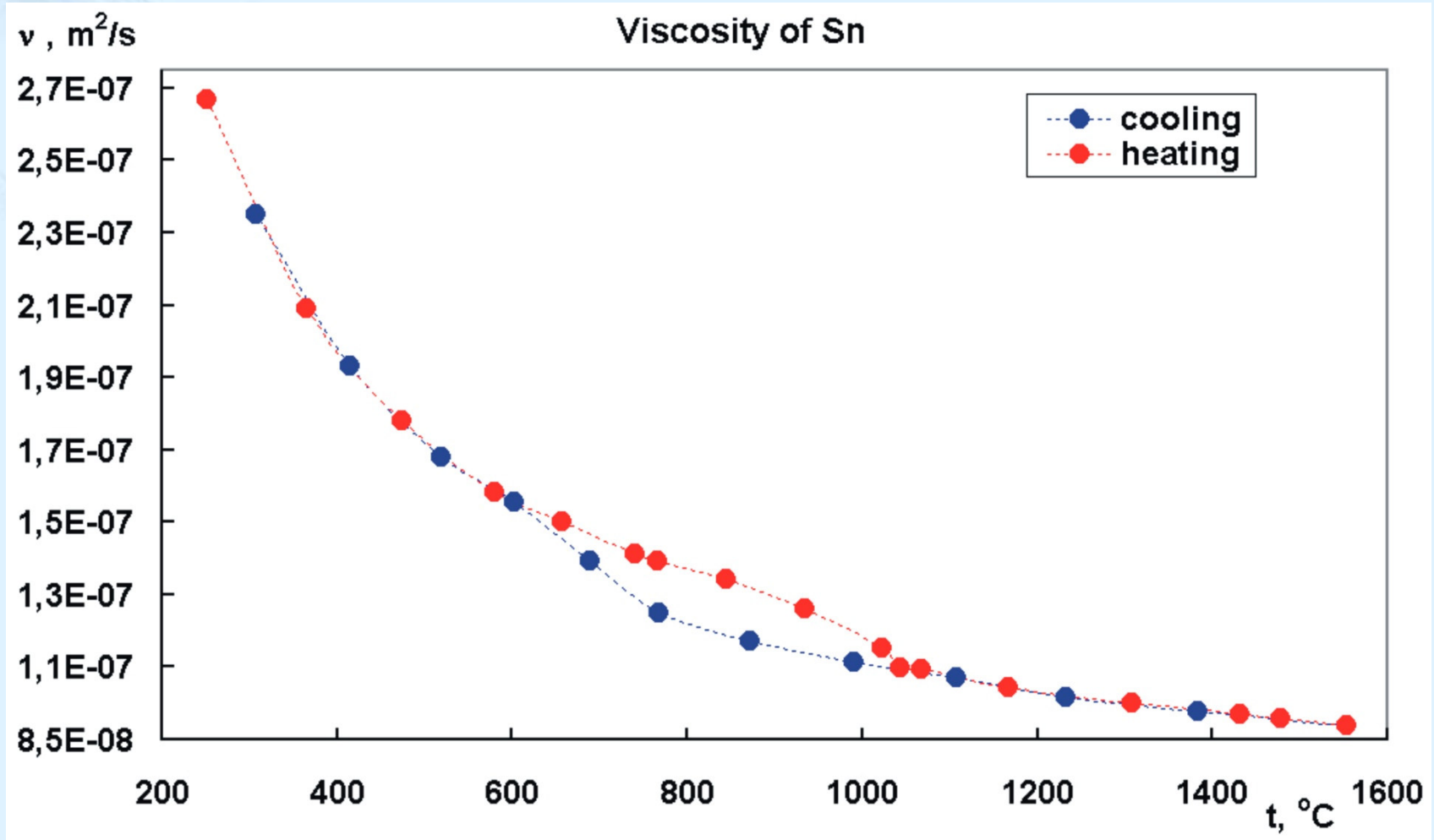
- Experimental methods mentioned above are the basic methods for detection and analysis of structural rearrangements in high-temperature metallic melts. They provide minimum amount of experimental data allowing one to determine the temperature or composition at which significant changes in melt structure occur.
- Experiments show that the short-range order rearrangements are most pronounced in temperature and concentration dependences of magnetic susceptibility and electrical resistivity. Variations in dispersity and in volume fraction of nano-meter sized colloidal particles are clearly seen in the corresponding viscosity and density curves

- When measuring temperature or concentration dependences of any property, false anomalies may appear due to imperfection of the method or instruments, interaction between the melt and the crucible or atmosphere etc. Only the combined study of properties in independent techniques may insure the researcher against the errors.

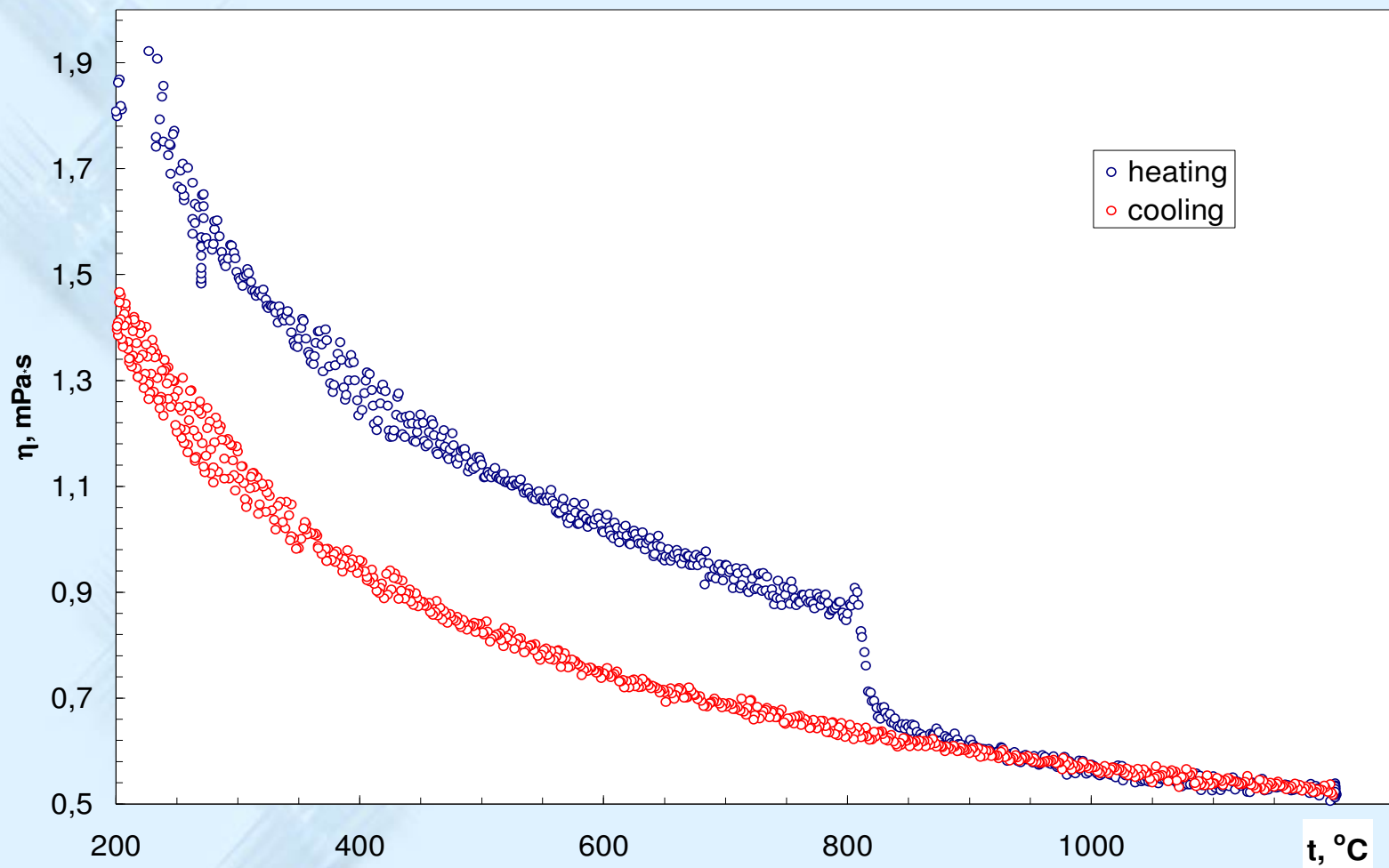
Sn-Cu-Ag

- The temperature curves are found to be smooth in cases of copper and silver. It means that no sharp structural changes take place in these melts during heating and the following cooling.
- As for liquid tin, three temperature ranges with abnormal behavior of properties were discovered: t_1 near 400° C (anomalies in density and electroresistivity); $t_2 = 1000-1050^{\circ}$ C (anomalies in viscosity and electroresistivity); t_3 approx. 1400° C (anomalies in density, electroresistivity and magnetic susceptibility).

Kinematic viscosity of liquid Sn (99.9%)



Dynamic viscosity of liquid Sn (99.999%)

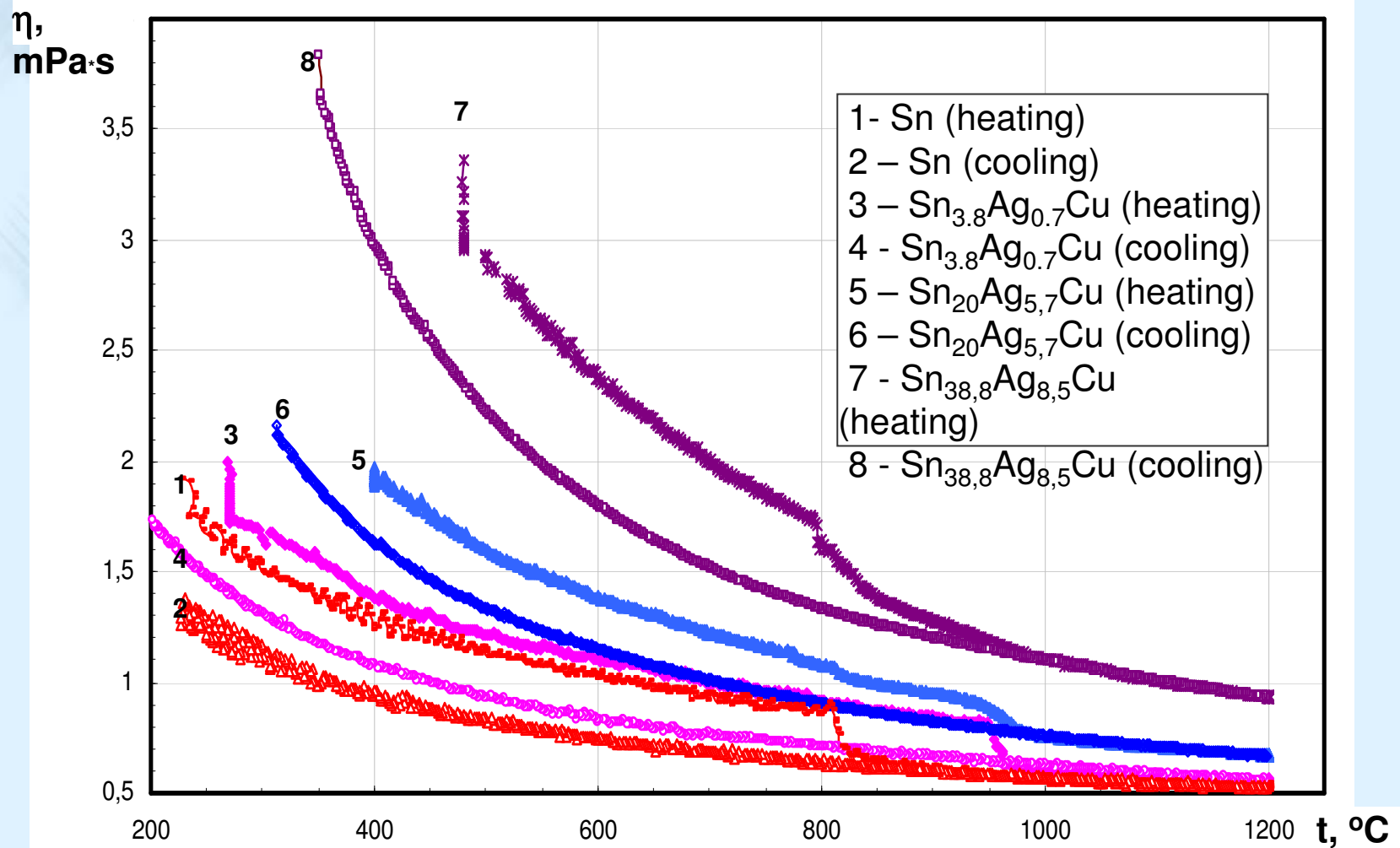


- The decrease in heating rate from 4 to 1^o C/min shifts t_{an} down to 800^o C, and the purification of samples from 99.9 to 99.999% provides the incoincidence of heating and cooling curves for all the temperatures below 800^o C.

- For Sn (99.9% purity)-0.5 wt.% Cu melt the abnormal behavior of viscosity (jump down) was found at $t_{an} = 790^{\circ} \text{C}$. At subsequent cooling the hysteresis of heating and cooling curves appears above 700°C . This phenomenon takes place at each overheating the melt above 800°C . When the highest temperature in the scan does not exceed 700°C , the heating and cooling curves are smooth and no hysteresis was fixed.

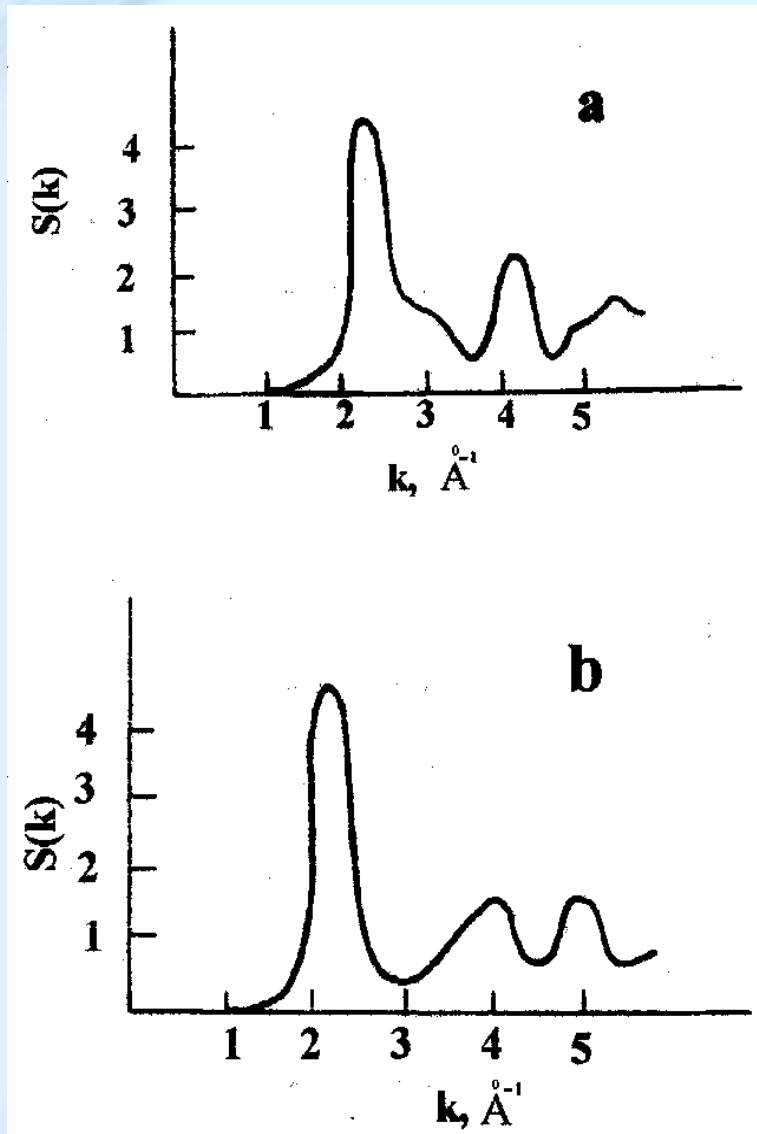
- The increase of copper content give the following: for Sn-0.7 wt.% Cu melt the temperature of anomaly shifts down for 20⁰; for Sn-1.0 wt. % Cu melt it increases for 20⁰ and becomes equal $t_{an} = 810^0$ C. From practical point of view, Sn-0.7 wt.% Cu alloy, corresponding to eutectic composition, seems to be the main attractive one for heat treatment.

Dynamic viscosity of Sn (99.999%)-Cu-Ag melts



- The anomalies in physical properties found for binary melts near 790° C and for ternary near 960° C are the evidence of sharp transformations in the structure of the melts. Hence, by varying regimes of heat treatment of the melts it becomes possible to obtain samples with different properties in solid state.

Electron diffraction in Sn-26.1 at.% Pb melt

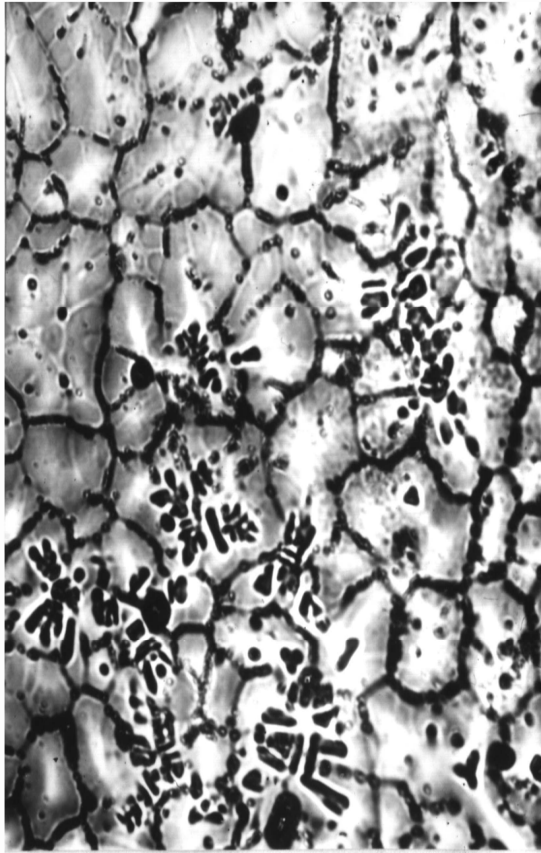


- After melting the sample, the maxima of the structure factor can be considered as a superposition of structure factors of liquid tin and lead
- At $t=480^\circ\text{C}$, corresponding to the anomaly in the temperature dependence of density, the maxima of the structure factor are strongly distorted and change their position. This situation remains stable during cooling up to crystallization

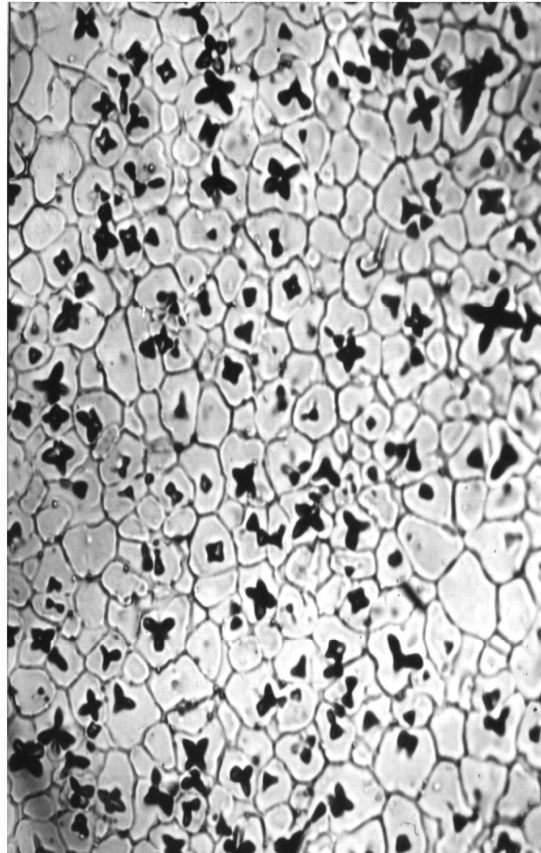
Microstructure of Al-17 wt.% Si alloys after different heat treatments of the melt



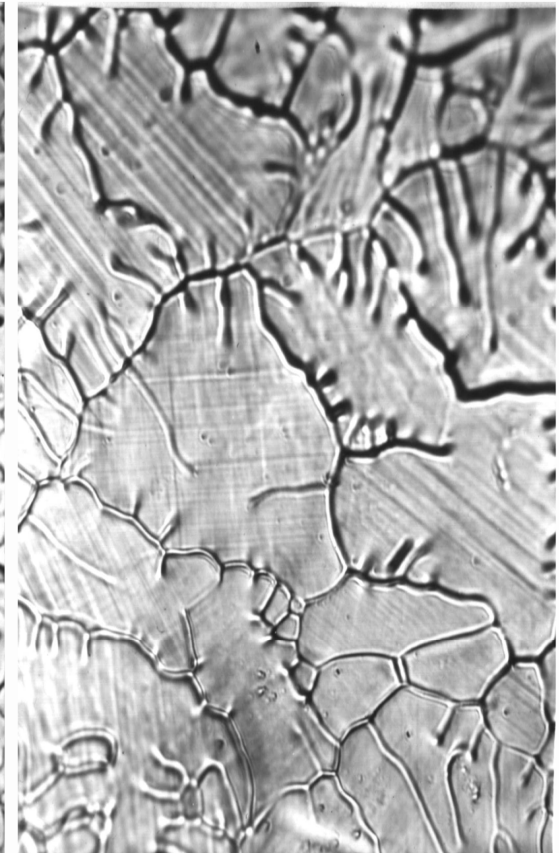
Microstructure of Al-0.6 at.% Zr alloys after different heat treatments of the melt



a



b



c

Industrial applications

- *Method* : Heat treatment of the melt.
- *Organisations where it was applied* : Aircraft industry, Aerospace industry, Metallurgy, Automotive industry.
- *Common features for all the objects after application of the method* :
 - - increase in mechanical properties and their stability for all the alloys in as-cast state;
 - - increase of melts supercooling before crystallization ;
 - - decrease of gas concentrations in ingots.

What do we suggest?

- experimental data on physical properties of metallic melts;
- regimes of melts heat treatment before solidification.

We are open for all your suggestions and proposals!!!