New trends in the nucleation research

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Abstract. During the last half of century the most of efforts have been directed towards small molecule system modeling using intermolecular potentials. Summarizing the nucleation theory, it can be concluded that the nowadays theory is far from complete. The vapor-gas nucleation theory can produce values that deviate from the experimental results by several orders of magnitude currently. Experiments on the vapor-gas nucleation rate measurements using different devices show significant inconsistencies in the measured rates as well. Theoretical results generally are quite reasonable for sufficiently low vapor nucleation rates where the capillary approximation is applicable. In the present research the advantages and current problems of the vapor-gas nucleation experiments are discussed briefly and a view of the future studies is presented. Using the brake points of the first derivative for the nucleation rate surface as markers of the critical embryos phase change is fresh idea to show the gas-pressure effect for the nucleating vapor-gas systems. To test the accuracy of experimental techniques, it is important to have a standard system that can be measured over a range of nucleation conditions. Several results illustrate that high-pressure techniques are needed to study multi-channel nucleation. In practical applications, parametric theories can be used for the systems of interest. However, experimental measurements are still the best source of information on nucleation rates. Experiments are labor intensive and costly, and thus, it is useful to extend the value of limited experimental measurements to a broader range of nucleation conditions. Only limited experimental data one needs for use in normalizing the slopes of the linearized nucleation rate surfaces. The nucleation rate surface is described in terms of steady-state nucleation rates. It is supposed that several new measuring systems, such as High Pressure Flow Diffusion Chamber for pressure limit up to 150 bar will be created soon in the frame of the Russian Ministry of Science & Education project under Contract № 14.Z50.31.0041 issued by February 13th of 2017. The Project will provide the nucleation studies for basic problems of theoretical and practical applications. Published under licence in Journal Title by IOP Publishing Ltd.

1. Introduction

The nucleation rate measurements data have the key impotence for the nucleation theory development, experimental data on nucleation interpretation, a material production, etc. The first nucleation experiment can be considered to be associated with measurements of liquid and crystals supercooling done by Fahrenheit [1]. The nucleation of bubbles in gas saturated solutions was observed and the
concept of critical embryos of a new phase was introduced in nucleation science during the second half of 19th century [2]. The quality of vapor/liquid nucleation rate results has improved substantially beginning in 1980s because of the development of new measurement systems. For example, the first prototype of the Flow Diffusion Chamber for vapor nucleation rate measurements was developed by Anisimov et al. [3, 4]. As a rule, theoretical and experimental data on nucleation rate are not in a good agreement over a range of temperatures and/or pressures. It appears that there may be problems in both the experiments and theory and deficiencies can be identified in all versions of nucleation theories and practically all of the reported experimental results. Current theories correspond mostly to various modifications of Classical Nucleation Theory [5]. The theoretical results look quite reasonable for sufficiently low vapor nucleation rates where the droplet approximation is applicable [6]. At the present time, vapor-gas nucleation theory can produce values that deviate from the experimental results by up to several orders of magnitude [9, 10]. However, nucleation experiments using different devises also show significant inconsistencies in the measured rates, for example [10]. Both problems produce difficulties in establishing standard vapor/liquid nucleation systems that could be used to test accuracy of vapor-gas nucleation rate measurements. The advantages and current problems of the vapor-gas nucleation experiments are discussed below and a view of the future studies is presented based on the assessment of vapor-gas/liquid nucleation experimental results.

One can find in scientific literature a pretty fresh idea of the nucleation rate surfaces design over the diagrams of phase equilibria [6]. That idea looks like profitable for the nucleation theory development and for various practical applications where predictions of theory have no high enough accuracy for today. The usual way of any theory is to reduce the many-body problem to one body in some field. The features of one body and the field are adjusted usually to get the reasonable compliance to the empirical results. Relation of the theoretical and empirical results is not good enough in case of the Nucleation Theory.

2. The empirical methods
Initial measurements of vapor nucleation were made by Aitken [12] and Coulier [13, 14] using the rapid adiabatic expansion of air saturated with water vapor to provide the necessary supersaturation. Allen and Kassner [15] modified the experimental procedure using an expansion/compression cycle in a Wilson-type chamber where compression is introduced after the adiabatic expansion of a vapor-gas mixture. The resulting vapor supersaturation permits the growth of the nucleated clusters to optically detectable sizes. The pressure change, intensity of transmitted/scattered laser light, and other parameters are recorded. Temperatures are measured with uncertainties of the order of several tenths of a degree Kelvin. The shock tube is a version of the expansion technique for the nucleation research (for example, Peters & Paikert [18]). It has been thought that wave tube measurements are less accurate “because pressure pulses cannot always be accurately reproduced” in comparison with two-piston chamber (Holten et al. [19]). Supersonic jets have been used to produce the same adiabatic expansion scheme where cluster generation and growth are decoupled (Kim et al. [20]). The expansion techniques cover a total nucleation rate interval from 103 up to 1017 cm-3s-1. The available nucleation data involve the nucleation pressures from 100 kPa to 7 MPa.

3. Diffusion chambers
Langsdorf [21] created the first static diffusion chamber (SDC). The SDC is described in detail by Katz [22], Kacker and Heist [23], etc. The static diffusion chamber consists of two wet plates maintained at different temperatures. Vapor diffusion and temperature gradient produce vapor supersaturation and nucleation in the space between the plates such that clusters of the new phase are formed. Clusters then grow. Particles can move toward the cold plate direction by temperature and vapor concentration gradients. A particle drops when its mass gets sufficiently large. A special SDC design was used for vapor-gas nucleation measurements at elevated pressures up to 4.0 MPa (Heist et al.[24]). Idea of SDC is used for the Flow Diffusion Chamber (FDC) design. Initial versions of the FDC were described by Anisimov et al. [3, 4]. The FDC scheme uses a hot laminar vapor-gas flow
within cold boundary conditions. Hot vapor-gas flow enters the chilled tube (condenser). The vapor-gas velocity distribution at the condenser beginning is parabolic one. The distributions of temperature, T, vapor supersaturation ratio, S, and nucleation rate, J, along the tube axis can be revealed as the result of the Navier-Stokes equations solution. Embryos form in the nucleation volume and subsequently grow in the supersaturated vapor. A flow laminator is used to produce fully developed initial laminar flow. Parameters of that flow are used as boundary conditions for the stationary heat-mass transfer problem.

An algorithm for estimation of the average nucleation rate over FDC nucleation volume was suggested by Anisimov et al. [25]. The maximum experimental value of the nucleation rate, Jmax, in diffusion chambers can be measured using an obvious relation Jtheor/Ntheor=Jmax/Nexp, where Jtheor is maximum theoretical nucleation rate; Ntheor and Nexp are the theoretical and experimental particle concentrations respectively (Hyvarinen et al. [26]; Wagner and Anisimov [27]). That relationship can be used also for Jmax measurements using a supersonic nozzle (Streletzky et al. [28]).

The current FDC scheme has been used for vapor nucleation rate measurements at total pressures from 0.03 to 0.50 MPa. The FDC data span over six orders of magnitude in nucleation rate that can be measured in a single experimental system. Nucleation temperatures from 230 to 400 K can be obtained in these systems now. Versions of FDC have built in the USA (Warren et al. [31]; Brock et al. [32]; Brock et al. [33]) as well as in Finland (Anisimov et al. [29]; Lihavainen & Viisanen [30]), Czech Republic (Brus et al. [10]), Nguyen et al. [37] have studied homogeneous and heterogeneous nucleation of a single vapor, etc. Several groups have used rapid turbulent mixing to measure nucleation rates of the single and binary vapors (Kogan & Burnasheva [34]; Sutugin & Fuchs [35]; Okuyama et al. [36]; etc). Most studies have only measured the critical supersaturation for two-component vapor systems. The problem of a turbulent mixing scheme is associated with wide spectrum of nucleation conditions. Fluctuations of vapor supersaturation and nucleation temperature can be large enough in the pre-nucleation zone to generate some pre-particles before system achieves homogeneity. These pre-particles initiate a heterogeneous nucleation. Thus, turbulent flow systems are rarely used for heterogeneous (Lee et al. [43]; Mavliev et al. [44]) and ion-induced (Seto et al. [45]; Gamero-Castano, et al. [46]; etc) nucleation measurements.

During the past five decades, several research groups have examined the effects of pressure and carrier gas composition on homogeneous nucleation. Classical nucleation theory assumes that the only role of the carrier gas is to maintain the temperature equilibrium of the clusters with the ambient media. Frank and Hertz [47] made the first observations of a gas-pressure effect. The result was reproduced in several other measurements (Katz et al. [48]; Brus and Zdimal [49], etc). Other experimental results have supported a dependence of the nucleation rate on the nature and total pressure of the carrier gas (Luijten and van Dongen [53]; Luijten et al. [54]; Anisimov et al. [42]; Lihavainen and Viisanen [55]. Gas-pressure effects were detected by van Remoortere et al. [56] although most of the measurements from this research group (i.e., Viisanen et al. [57]; Viisanen and Strey [58]) did not observe carrier gas influences on nucleation rate. Fladerer and Strey [9] did attempt to measure supersaturated argon nucleation using a cryogenic nucleation pulse chamber. Experimental results of different experimental schemes used for nucleation rate measurements at cryogenic temperatures are still inconsistent (Fladerer and Strey [9]) because of low accuracy of the experimental data. Several research groups have made comparative measurements of nucleation rates (Anisimov et al., [29, 59]; Wilck et al. [60]; Brus et al. [10], etc.) The nucleation rates of n-butanol in helium using both an FDC (Hyvarinen et al. [61]) and an expansion chamber (Strey et al. [16]) illustrate the data sets deviation on 4-5 orders of magnitude. The origin of these data discrepancies can be better understood through the consideration of nucleation rate surfaces (Anisimov et al. [62, 63]).

In the most cases, the gas is treated as an inert media to absorb the heat released from the phase transitions. Inconsistencies in the experimentally measured values from the different experimental schemes are a major problem for current vapor-gas nucleation experiments. Consideration of vapor-gas nucleation as a binary system is a reasonable way to resolve the data
inconsistencies. It is plausible to think that different experimental systems have the inconsistent trajectories along the nucleation rate surface when the vapor-gas system is treated as binary system. Several results, for example, Anisimov et al. [41, 63] illustrate that high pressure measuring techniques need to be designed to study multi-channel nucleation.

4. Reference system for nucleation rate measurements
A In order to test the accuracy of an experimental system, it is important to have a standard system that can be measured over a range of nucleation conditions. The n-pentanol–helium system was proposed in Prague, 1995 for such measurements. The available results from several research groups were collected and compared (Anisimov et al. [11]). They proposed a reference equation for nucleation rates of n-pentanol–helium as a practical test of any experimental measurement system for total pressures from 0.10 to 0.30 MPa. The problem of a nucleation standard can only be solved when consistent results have been obtained by independent groups that use the different experimental schemes.

5. Nucleation rate surfaces design
Nucleation experiment can be provided in very local nucleation conditions even the nucleation takes place from the critical conditions down to the absolute zero temperature limit and from zero nucleation rates at phase equilibria up to the spinodal conditions. Theory predictions have low reliability as a rule. It is well known that any phase diagram has several lines of phase equilibria. It is easy to show that each line of phase equilibria generates the nucleation rate surface in space of nucleation process parameters. It means that one has multi sheet nucleation rate surfaces in the common case. Each nucleation rate surface is related to single phase state generation, or it is related to a one channel of nucleation. Semiempirical design of the nucleation rate surfaces over diagrams of phase equilibria have a potential ability to provide a reasonable quality information on nucleation rate for each channel of nucleation. One reason for nucleation theory problem is the application of inconsistent assumptions that are used to interpret the experimental results. The most experimental data on vapor nucleation are interpreted as a one-surface (one nucleation channel) approximation of a nucleation theory. However, two or more nucleation channels are really present in most systems as it was reported by Anisimov et al [6]. To empirically detect a single nucleation channel one needs to have the appropriate measuring system, but these kinds of systems are not currently under active development. The semiempirical approach permits the prediction of a realistic topology for the nucleation rate surface. That topology can be designed over the phase equilibria diagrams [62, 66, etc]. One needs at least several experimental points on the nucleation rate surface and the phase diagram to effectively develop these surfaces [66].

A short history of semiempirical design of the nucleation rate surfaces over phase equilibrium diagrams can be discussed briefly. This approach involves a simple idea developed by Anisimov et al. [62, 65, etc], that nucleation rate surfaces arise up from the phase equilibria lines. It means that each line of the real and metastable phase equilibria produces two nucleation rate surfaces both surfaces reflect the nucleation kinetics for each of two metastable phases that can be joined by an equilibrium line. Experimental detection of nucleation rate for each of two individual vapor nucleation rate channels, which are generating two single nucleation rate surfaces for vicinity of the triple point, one can see in article [67]. That result is unique to the present time. Theory involves a number of assumptions to describe small clusters [68]. Further, when the size dependencies of the surface tension and density of nuclei were taken into account [6] and the inherent degrees of freedom were used to calculate the statistical sum for a nascent cluster [69], agreement between theoretical and experimental results get worse. Semiempirical design over diagram of phase equilibria is applied for metastable volume construction [65-68]. That way has a considerable potential in the nucleation rate surface description. A major problem in detection of the nucleation rate surface singularities is the random error inherent in any experimental data. This problem exists even one has experimental results of relatively high accuracy as it is shown by Anisimov [6]. A conceptual problem
in vapor-gas to liquid (or to solid phase) nucleation is its treatment of the nucleation as a single component problem instead as a two component system. Results of the present research illustrate clearly that gases can generate a phase transitions in a condensed state of any vapor-gas system. A direct mass spectrometric measurements show the presence of the carrier gas in the critical cluster under some nucleation conditions, for example, that result was shown in case the monosilane-argon system [71].

6. Conclusions
In the present review, advantages and problems related to aerosol generation experiments are discussed. It can be concluded that the development of accurate experimental techniques for vapor-gas nucleation research are still in progress. The current recommendations for vapor-gas nucleation rate measurements can be summarized such as: vapor nucleation and cluster growth events should be decoupled to allow the embryos to grow before light scattering detection. Theory independent algorithms are needed to estimate experimental nucleation rates based on FDC and others flow systems experimental data. Homogeneous nucleation rates should be measured at the sufficiently high vapor supersaturation [4] to reach conditions of the vapor homogeneous nucleation. Impurities in the vapor substance must not exceed 0.2 % for the present time. This level of impurities shifts the nucleation rate values within one order of magnitude as shown by Anisimov et al. [39], Strey et al. [17], etc.

Vapor-gas systems are strongly recommended to be considered as binary nucleation systems. The problem of experimental data inconsistencies should be resolved to create a nucleation rate standard. Aerosol size distribution measurements are strongly recommended to identify the possibility of two and more channels for homogeneous and heterogeneous nucleation. It can be hoped that the uncontrolled parameter(s) will be identified in the near future and permit consistent nucleation rate data to be derived from different research methods. Introduction of the nucleation standard(s) is a major current problem. Success in the nucleation standard development and its introduction in nucleation research practice is a key issue for current nucleation experiments.

Empirical results illustrate clearly that gases can generate a phase transitions in a condensed state of matter in the critical line vicinity. A direct mass spectrometric measurements [71] show the presence of the carrier gas in the critical cluster under some nucleation conditions. Several contemporary achievements are collected in the present review. It is clear that idea of a semiempirical design of the nucleation rate surfaces is promising tool for the nucleation theory development. The data base file and the digital information on nucleation rates, equations of states, phase equilibria diagrams, binodal and spinodal conditions, etc will be intensively collected without doubt. Time when soft to the nucleation rate surface design will be available for each scientist and engineer is approaching and the knowledge on nucleation will get position of powerful tool which is effective in scientific research and industrial applications.

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