Progress in Ultrasonic Spray Pyrolysis for Condensed Matter Sciences Developed From Ultrasonic Nebulization Theories since Michael Faraday

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Abstract – *This review outlines briefly the history of the phenomenon of ultrasonic nebulization of liquids since the discovery of such an effect by Michael Faraday and the explanation of the phenomenon by capillary wave mechanism and "cavitation" hypothesis. Ultrasonic spray pyrolysis for materials processing and the theory that predicts the final particle are discussed. The popularity of the technique is shown by the rising number of research groups in the world processing various materials by this method due to its cost-effectiveness, purity of its products and controllability of particle size and final properties.*

Keywords: *Pyrolysis, Ultrasonic Spray, Surface Tension, Chemical Vapor Deposition, Viscosity*

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1. Introduction

Chemical vapour thermal deposition form one of the largest groups of techniques for realising a variety of materials in condensed matter science. The starting material is either a gas or liquid carefully chosen to end up into a stoichiometric material desired. The general process entails a source of chemical vapours/droplets which are carried into a heated zone for evaporation and decomposition and finally ending up either on a substrate (for thin films) or a filter (for powders) [see schematic in Fig. 1]. When dealing with vapours/gases as starting materials, the method is usually referred to as chemical vapour deposition (CVD); there are many forms of CVD. The term "spray pyrolysis" (SP) is used when dealing with liquid droplets or powders as precursor materials.

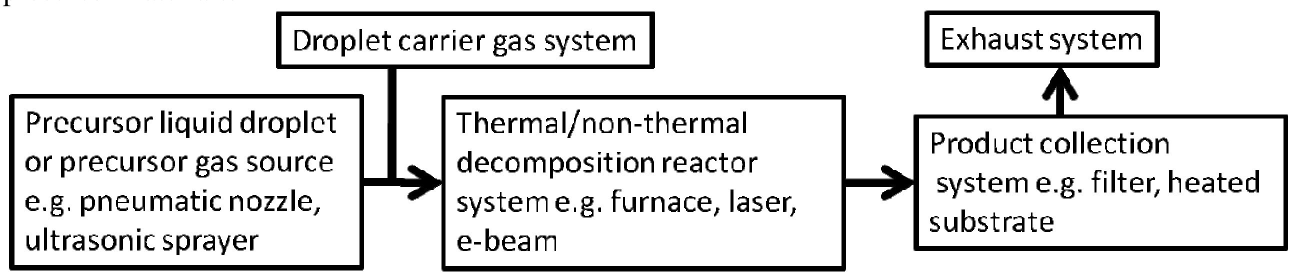

Fig. 1 Generalised schematic of chemical vapor deposition systems on which ultrasonic spray pyrolysis is based

The word pyrolysis is taken from a Greek word "pyre" which means "a pile of fuel or pile of wood" with specific reference to heating by flame [1]. Since such heating raises the precursor material to a plasma state where radicals, electrons and ions prevail, this process can be used in *in-situ* spectral analysis of elemental composition of the precursors in addition to the decomposition mechanisms, reaction kinetics and formation of new condensed matter. The source of heat can be a furnace (thermal CVD), a hot wire/filament (HWCVD, HFCVD), an intense light source such an I.R. CO_2 laser or a UV excimer laser (laser pyrolysis LP), plasma source (plasma enhanced PE-CVD), an I. R. lamp or, simply, a heated substrate.

A number of previous review articles have been presented on different forms of CVD: thermal CVD [2-11], plasma enhanced PE-CVD [12-19], hot-wire or hot filament (HWCVD or HFCVD) [20-28] and not many of them have been as exhaustive in their respective areas. Pyrolysis, although classified under CVD in some text, has become a wide area of research and technology covering synthesis of new products, qualitative and quantitative spectroscopic analysis of fluids and, lately, alternative route to production of debri-free x-ray sources; these aspects are elaborated further in the sections that follow. In spray pyrolysis the droplets or vapours can be generated either by pneumatic nozzles in whistle-type sprayers or ultrasonic nebuliser.

In the former the process is simply called spray pyrolysis (SP) and in the latter case, the process assumes the name "ultrasonic spray pyrolysis" (USP). An article on the versatility of spray pyrolysis by Pramod Patil [28] among other aspects tabulated publications up to early 1999 listing materials and spray pyrolysis parameters. Other reviews have been on specific materials employing spray pyrolysis as one of the wide range of methods used in producing such materials: superconductors [29], carbon nanostructres [30], ceramic nano-composites [31], diamond [32], semi-cokes [33], and semiconductors [34]. The present review chapter will restrict its discussion to ultrasonic spray (USP) technique on a wide range of materials especially from 1999 to the present and on laser spray (LP) pyrolysis. This is a period that has seen a lot of improvements to pyrolysis techniques to the extent that structures with new shapes and novel growth dimensionality have been produced in a controlled manner.

The scarcity of specific review papers in a period like this one where numerous publications pertaining to materials synthesis by various versions of pyrolysis was the main motivation of the present compilation. First, a historical outline of the droplet generation phenomenon by ultrasonic nebulisation is given. This has not been covered in most previous reviews except by Yule *et al.* [35], Barreras *et al.* [36] and Nevolin [37]. These reviews have not covered pyrolysis but restricted themselves to the nebulisation phenomenon. The triumphs and challenges in ultrasonic spray pyrolysis are also presented. A tabulated literature survey and data-base from 1988 to 2008 is given and some unsolved problems in pyrolysis for materials processing with regard to droplet and particle size under different pyrolysis parameters are discussed.

2. Ultrasonic nebulization phenomenon

Ultrasonic atomization is a very effective method for production of ultra-small droplets and, after the droplets are pyrolyzed, the realisation of nano-sized materials. Quantum dots have been produced by spray pyrolysis [38]. Three approaches are common in the droplet production: (1) passing the liquid across a standing ultrasonic wave, (2) depositing the liquid over an ultrasonic transducer and (3) immersing a focussing ultrasonic transducer in the liquid in such a way that the liquid depth is equal to the focal length of the ultrasound lenses in the transducer.

Generation of droplets by means of ultrasonic waves was first reported in 1927 by Wood and Lomis [39]. A number of mechanisms have been proposed to explain this phenomenon. At low excitation frequencies (20 – 100 kHz), we can imagine that only surface molecules respond to form droplets; such waves are called *capillary waves*. At higher excitation frequencies (0.1- 5 MHz) and intensities, bulk atoms of the liquids come into play and this effect is called *cavitation*.

2.1. Capillary Wave Mechanism

The capillary wave proposal enjoyed intense research interest from the first known studies by Faraday [40] in 1831 to the present. It was Lord Kelvin, as elaborated in Rayleigh's book [41] in 1871, who derived the well-known equation for the wavelength of capillary waves as

$$\lambda = \left(\frac{2\pi\sigma}{\rho f^2} \right)^{1/3} \tag{1}$$

Here, λ is the wavelength, σ is the surface tension, ρ is the liquid density and f is the frequency of the surface waves. This equation was later modified by Rayleigh [41,42] to give

$$\lambda = \left(\frac{8\pi\sigma}{\rho F^2} \right)^{1/3} \tag{2}$$

Note that F which is equal to $2f$ is not the frequency of the surface waves but rather the frequency of the forcing sound. The fact that the frequency of the surface waves is *half* the exciting frequency was empirically obtained from experimental measurements. A number of experimental workers in the 1950's [43-48] pointed to unstable surface capillary waves as the origin of droplet formation relying on the simplified linear instability analysis. The 1962 experimental determination by Robert Lang [49] of the relationship between the wavelength of the capillary waves and the size of the droplets so formed spurred the capillary wave mechanism to greater heights. Lang showed that the droplet size, D_L, and the capillary wave length λ were related by the empirical equation

$$D_L = 0.34\lambda \tag{3}$$

The subscript L in Eq. 3 signifies the Lang's droplet diameter in distinction from other droplet diameter symbols to follow. Extra support from Sindayihebura & Bolle [50] in 1998 brought more assurance that capillary waves were probably the main mechanism. How drop formation may occur by unstable surface capillary waves was illustrated schematically as reproduced in Fig. 2 and this phenomenon is usually called the Taylor instability [52]. In the Taylor instability the liquid capillary waves are composed of crests (peaks) and troughs. Atomization takes place when unstable oscillations tear off the crests of the capillary waves away from the bulk of the liquid. Thus the droplets are produced at the crests whose size is proportional to the wavelength.

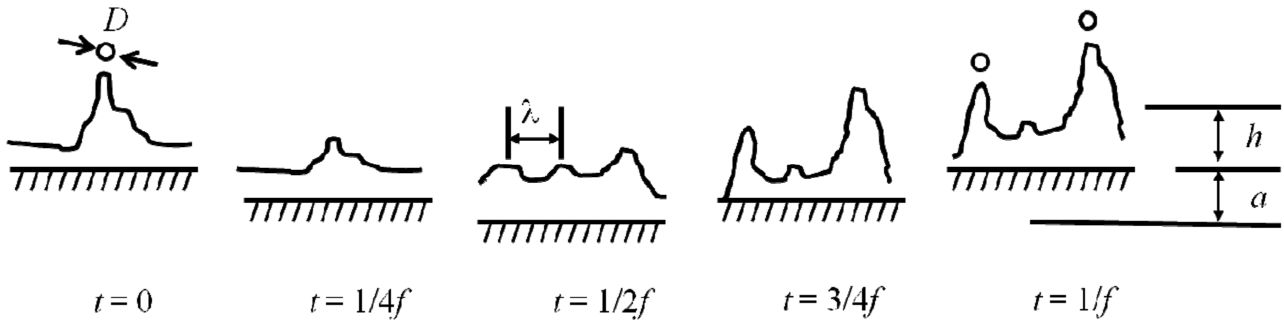

Fig. 2 A sketch showing idealized droplet formation from standing-wave crests showing one period of wall vibration.

A major revision to the Lang's equation was done by Peskin & Raco [52] in 1963 and later, 1996, by Jokanovic *et al.* [53] who, rather than adopting an existing empirical equation, chose to derive a general equation from first principles. The analysis especially by Jokanovic *et al.* started from applying the Bernoulli's equation to an incompressible fluid of density, ρ, surface tension, σ, under pressure, p, due to an ultrasonic excitation, f, from a depth, y, and thereby generating a disturbance of amplitude, ξ(x,t) given by

$$\rho g h + \rho \frac{\partial \varphi}{\partial t} + \sigma \frac{\partial^2 \varphi}{\partial t^2} = 0 \tag{4}$$

In this equation, φ is the rate potential. The boundary conditions employed were that when $y = -h$, $v = 0$ and $\partial^2\varphi/\partial x^2 = 0$ then

$$\varphi = \frac{1}{h}\frac{dy}{dt} c_J h\left[k_J\left(y + x\right)\right]e^{ikh} \tag{5}$$

Here, c_J is a constant, k_J was taken to be the wave-number ($2\pi/D_J$) where in turn D_J is the Jokanovic's aerosol droplet diameter (again to distinguish it from that of Lang above). The Mathieu's function was then adopted which was observed to explain the typical shape of the relationship between the amplitude of the oscillation of the meniscus surface and the wave-number. The Mathieu's function was given as

$$\frac{dy}{dt} + hk\left[\frac{\sigma k^3}{\rho}ht - kght\right]y = 0 \tag{6}$$

The solution of Eq. 2.6 for $h >> \xi(x,t)$, that is, for small disturbances, found by Jokanovic was seen to be similar to that previous found by Peskin & Raco using a different analysis route (not reproduced here)

$$D_J = \left(\frac{\pi\sigma}{\rho f^2}\right)^{1/3} = \frac{1}{0.68}D_L \tag{7}$$

Note that the relationship between droplet diameter and the Kelvin relation for capillary wave length can also be derived from dimensional analysis as shown in by Mwakikunga *et al.* [55] given as

$$D = k_M\left(\frac{\sigma}{\rho f^2}\right)^{1/3} \tag{8}$$

where k_M is a dimensionless constant which according to Lang is $0.68\pi^{1/3}$ while, according to theoretical derivation by Peskin & Raco and Jokanovic, the constant k_M is equal to $\pi^{1/3}$. This means the droplet diameter as calculated by Lang's equation is smaller by the factor of 0.68 in comparison with that determined by Jokanovic's equation. Jokanovic *et al.* were able to show experimentally that their freshly derived equation yielded better agreement between calculated and experimentally determined droplet sizes. It must also be noted that Jokanovic *et al.* have arrived at Eq. 7 using various forms of the equation of motion of the liquid at the surface including one given by [54]

$$\frac{\partial\varphi}{\partial t} + g\xi - \frac{\sigma}{\rho}\left[\frac{\partial^2\xi}{\partial x^2} + \frac{\partial^2\xi}{\partial z^2}\right] = 0 \tag{9}$$

However, a number of more recent studies employing ultrasonic spray pyrolysis (an application to be discussed in the next section) and using either the Lang's empirical formula (Eq. 3 and/or the Jokanovic's revision Eq. 7) have shown that both equations have limitations. A serious conflict between theory and experiment reported by Nedeljkovic *et al.* [56] states: "Comparison of the theoretically [d_J = 195 nm, d_L = 132 nm] obtained results with the experimentally determined [d_{exp} =286 nm] regardless of the equations being used for the determination of the aerosol droplets diameter undoubtedly shows that there is a substantial difference between the theory and the experiment, if the theoretical density of the particles packing was assumed…" And also according to Saponjic *et al.* [57] "Significant differences between the experimentally determined (285 nm) and the theoretically predicted values of the mean particle diameter (132 nm and 195 nm) were found indicating that the powder was highly porous…"

The reasons for this gross under–estimation by the theory of the experimentally determined particle size could be (1) the basic assumption in the Kelvin wavelength on which both the Lang's formula and that of Jokanovic *et al.* are based and (2) the absence of the dependence of droplet size on liquid viscosity and the volumetric flow rate which is contrary to experimental observations.

This calls for the consideration of the liquid's bulk properties in the models. These shortfalls are discussed in the unsolved problems in USP in section.

2.2. Cavitation Mechanism

Cavitation hypothesis is generally applied to high frequency and high energy intensity systems. When a liquid is irradiated with an intense ultrasound field, cavitation bubbles are formed. During the implosive collapse of these bubbles near the surface of the liquid, high intensity hydraulic shocks are generated which in turn initiate disintegration into droplets. At such large intensities the excitation is beyond the liquid surface but extends into the liquid bulk contrary to the capillary hypotheses. Properties of the liquid bulk such as viscosity come into play as parameters affecting the nature of the final droplet. Sollner [58] was probably the first in 1936 to explain Wood & Loomis's ultrasonic atomization demonstration in terms of cavitation produced under the liquid film. While Lang and the other workers developed the capillary hypothesis, the cavitation hypothesis was almost abandoned thanks to Eknadiosynats and co-workers [59,60] who resumed this area in the mid-60's. Several studies after these tried to combine both hypotheses [61-64]. The effect of viscosity and surface tension on the Taylor instability has been studied [65], that the rate of growth of amplitude disturbance is affected by viscosity has been observed [66], the increasing importance of viscosity as surface tension decreases has been suggested [67] and the effects of density, viscosity, interfacial tension and relative fluid velocity on drop formation have been elaborated by Clark [68,69]. Another empirical equation for the prediction of the droplet size at high liquid flow rates was proposed in 1978 by Mochida [70] as

$$D = 31.7\left(\frac{\sigma}{\rho}\right)^{0.354} \mu^{0.303} Q^{0.139} \tag{10}$$

In this equation, σ and ρ have the usual meanings, μ is the viscosity and Q is the volumetric flow rate of the liquid. However, this equation does not account for the excitation frequency. Clark found that the dependence of droplet size on viscosity roughly followed the proportionality

$$D \approx \mu^{0.166-0.303} \tag{11}$$

Tsai *et al.* [71] found in 1996 that droplet size and volumetric flow rate were correlating approximately thus

$$D \approx Q^{0.25-0.30} \tag{12}$$

This was quite in conflict with Mochida with his exponent being outside the range set in the Tsai *et al.* improved measurement.

2.3. A Combination of Capillary and Cavitation Hypotheses

More careful observations have shown that apart from the traditional parameters of surface tension, viscosity, density, forcing frequency and volumetric flow rate additional parameters such as geometry of the vibrating surface, the amplitude of the oscillations, the intensity of the ultrasound power or the energy density have a lot to do with the size of the droplet so produced. To this end Rajan and Pandit [72] in 1996 developed a new correlation equation to take into account some of these extraneous parameters and was found to be

$$D = \left(\frac{\pi\sigma}{\rho . f^2}\right)^{1/3} \left[1 + A.(We)^{0.22} (Oh)^{0.166} (I_N)^{-0.0277}\right] \tag{13}$$

The symbols A, We, Oh, I_N are respectively the surface area of the droplet, the Weber's number (the number that describes atomization), the Ohnesorge's number (or the viscous number) and the intensity number (the number affected by the geometry of the vibrating surface) defined in the following expressions:

$$We = \frac{fQ\rho}{\sigma} \tag{14}$$

$$Oh = \frac{\mu}{f.A_m^2\rho} \quad (15)$$

$$I_N = \frac{f^2 A_m^4}{v_s Q} \quad (16)$$

Most symbols have usual meaning but *Am* is the amplitude, v_s is the speed of sound. Three alternate correlations to Eq. 13 were derived by Rajan and Pandit [72]: (1) using the Rayleigh instability criterion, (2) using the Walzel relation and (3) using Davies approach respectively

$$D = \frac{\mu Q k}{3.74\sigma A_m} \quad (17)$$

$$D = \frac{1.06kQ\dfrac{\mu}{\rho}\left(\dfrac{\rho}{\pi\sigma f}\right)^{1/3}\left(\dfrac{\rho f^2}{\pi\sigma}\right)^{2/3}}{fA_m\left[2+0.6\dfrac{\mu}{fA_m^2\rho}\right]^{1/3}} \quad (18)$$

$$D = k_1\left[\sigma + \tfrac{1}{4}\mu(A_m.f)\right]^{0.6}\rho^{-0.6}\left[\tfrac{1}{2}v_s A_m(2\pi.f)^2\right]^{-0.4} \quad (19)$$

A parity plot is one of the most convenient graphical techniques for evaluating the theoretically calculated quantity and the experimentally observed quantity. Rajan and Pandit [72] also presented parity plots for correlations in Eq. 13 and Eq. 19 and we reproduce them in Fig. 3

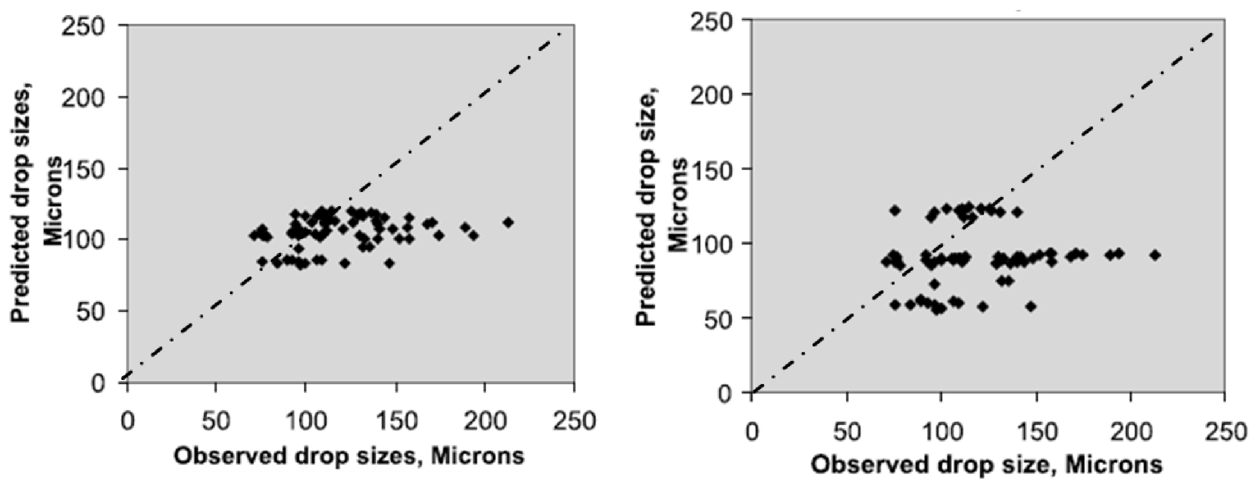

Fig. 3 Plots for correlations Eq. 13 and Eq. 19 for water droplets. The dotted line is a line that indicates the points where calculated quantity is exactly equal to the experimentally measured quantity.

From the parity plots one sees that most points are below the equality line signifying that the measured drop size is mostly less than the calculated drop size. This means that the Rajan-Pandit correlations are over-estimating the observed droplet size.

Avvaru *et al.* [73] have recently, in 2006, modified Eq. 13 to suit the so-called "Newtonian viscous liquids" given as

$$D = \left(\frac{\pi\sigma}{\rho f^2}\right)^{1/3} + 0.0013(We)^{0.008}(Oh)^{-0.14/n}(I_N)^{-0.28} \quad (20)$$

In order to validate their theory, one of the Newtonian liquids — glycerine — was used in their study and the parity plots done using correlations in Eq. 13 and 19 were presented and are herein reproduced in Fig. 4.

In comparison with the Rajan-Pandit parity plots, one can clearly see a remarkable improvement in the alignment of the calculated –experimental points to the equality line.

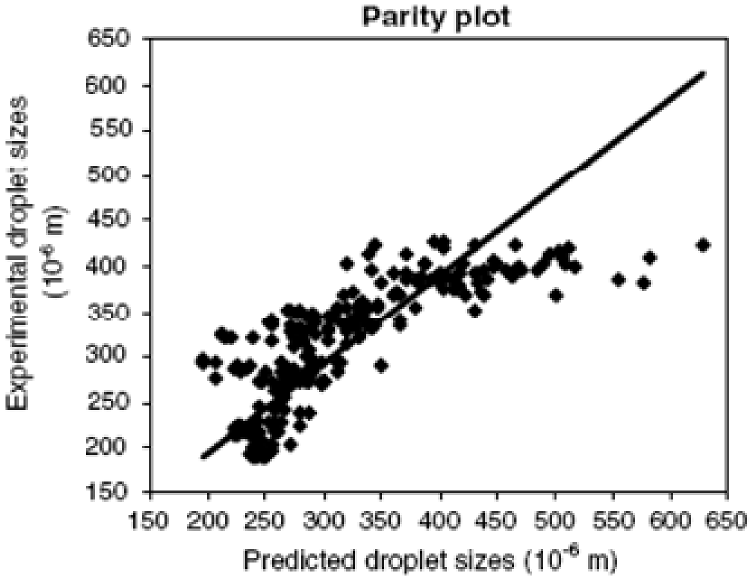

Fig. 4 A typical parity plot for a Newtonian liquid, glycerine (From Avvaru *et al* Ref. 117).

However, one can also see departure from the ideal equality line when droplet diameters exceed 400 μm. Therefore beyond this point the Avvaru correlation over-estimated the droplet sizes. Also even within the region where there is apparently good agreement, more points are above the equality line indicating that, in this region (200 – 350 μm), the Avvaru correlation under–estimated the droplet size. Overall, however, the Avvaru correlation is a big improvement over that of Rajan-Pandit. Avvaru *et al.* are also able to confirm and demonstrate the presence of cavitation in the droplet ejection by arranging an ingenious experiment. In this experiment, the ultrasonic generator is tilted horizontally and the force of droplet ejection is balanced with the environmental drag force from which the ejection velocity is determined. Using their so-derived differential equation, they are able to show that the Newtonian liquids such as glycerine yield an ejection velocity of 12.6 ms^{-1} whereas the non-Newtonian liquids yield an ejection velocity of 3.5 ms^{-1}. In both cases the ejection velocity is higher than the cavitation-less ejection velocity of 0.144 ms^{-1} which is attributed to capillary theory.

3. Effects of Pressure and Temperature on Surface Tension, Density and Viscosity of Fluids

With improvements of the theory, it is hoped that the future is bright with regard to understanding the phenomenon of ultrasonic generation of droplet from liquids. One of the many unsolved problems concerning the droplet size as a function of the liquid properties of surface tension, σ, viscosity, γ, density, ρ and so forth involves finding from thermodynamics how these properties vary when the liquid temperature and pressure change. In Mwakikunga *et al.* [Ref. 55], such a temperature–and–pressure dependent droplet size was dealt with by considering that droplet size took the expression in Eq. 8. Eq. 20 could also be re-written in the like manner as

$$D = \left(\frac{\pi \sigma(p,T)}{\rho(p,T) f^2} \right)^{1/3} + 0.0013 \left(We(p,T) \right)^{0.008} \left(Oh(p,T) \right)^{-0.14/n} \left(I_N(p,T) \right)^{-0.28} \quad (21)$$

3.1. Surface Tension as a Function of Temperature and Pressure

One of the earliest experimental studies on surface tension determination at varying pressure was carried out by Lynde [74] in 1906. In this study the surface tension at the interface between two liquids was determined via the derived equation

$$\sigma \cos\theta = \tfrac{1}{2} H_D r_{tube}(\rho_2 - \rho_1) \tag{22}$$

θ was the angle of contact, H_D was the difference in height between the two liquids in the manometer, r_{tube} was the radius of the capillary tube and ρ_2, ρ_1 were densities of the two respective liquids. Taking a differential of Eq. 22 with respect to pressure p, Lynde got

$$\frac{\delta\sigma}{\delta p} = \tfrac{1}{2} r_{tube}(\rho_2 - \rho_1)\frac{\delta H_D}{\delta p} + \tfrac{1}{2} H_D r_{tube}\left(\frac{\delta\rho_2}{\delta p} - \frac{\delta\rho_1}{\delta p}\right) \tag{23}$$

By dividing Eq. 23 by Eq. 22, Lynde arrived at the following expression

$$\frac{1}{\sigma}\frac{\delta\sigma}{\delta p} = \frac{1}{H_D}\frac{\delta H_D}{\delta p} + \frac{1}{\rho_2 - \rho_1}\left(\frac{\delta\rho_2}{\delta p} - \frac{\delta\rho_1}{\delta p}\right) \tag{24}$$

The first term on the right hand side of Eq. 24 was measured experimentally by observing the change in height at varying pressure. The second term was determined from compressibility factors of the two liquids at varying pressure since $d\rho/dp$ is compressibility factor in the first place. With these measurements, Lynde was able to establish that a plot $(1/\sigma)(\delta\sigma/\delta p)$ versus p was a positive linear graph for mercury–water system and for mercury–ether system. The same was a negative linear plot for water – ether system and for chloroform–water system. However, for the carbon bi-sulphide – water system a parabolic line-shape was obtained. These results showed that the surface tension–pressure relation depends on not only on the liquid types but also on how the liquid densities vary with pressure which is discussed in the next few pages. For the case where the $(1/\sigma)(\delta\sigma/\delta p)$ versus p graphs are linear,

$$\frac{1}{\sigma}\frac{\delta\sigma}{\delta p} = \pm k_\sigma p \tag{25}$$

k_σ is a proportionality constant in Pa^{-2}. Surface tension can then be written in terms of pressure as follows:

$$\sigma(p) = \sigma_0 \exp\left(\pm \tfrac{1}{2} k_\sigma \left(p^2 - p_0^2\right)\right) \tag{26}$$

σ_0 is the surface tension at atmospheric pressure p_0. Sachs *et al.* [75] in 1995 summarized all σ-p data from methane-water system up to that time [75-79] and their charts are reproduced in Fig. 5

For one to see the effect of temperature on surface temperature, one can turn to the important work of S. J. Palmer [80] in 1976. Palmer's theory based on (1) the calculation of the difference in energies of interaction between molecules in bulk and those on the surface or 'excess energy' (2) the minimum potential energy of these molecules due to a balance between attractive and repulsive forces at a critical temperature T_c.

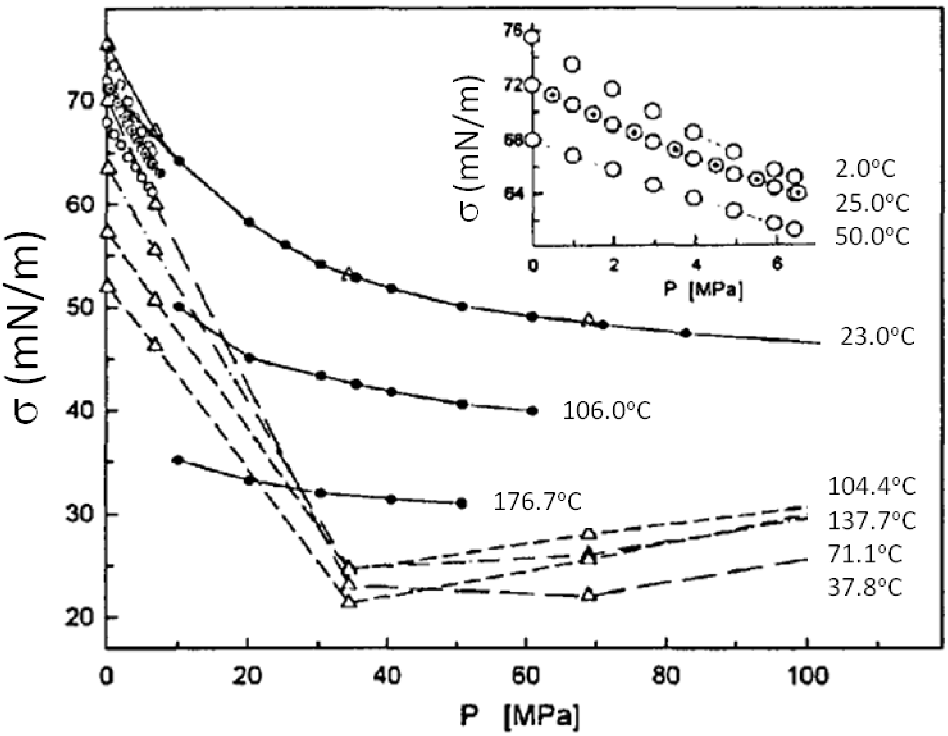

Fig. 5 Pressure and temperature dependence of the surface tension σ in the system methane-water; all data published in Ref. 119 until 1995. Δ, Ref. 120; •, Ref. 121; ⊕, Ref. 122 and o, Ref. 123.

The derivation led to the following expression:

$$\sigma(T) \approx \tfrac{1}{4} n \left(\frac{N_0 \rho}{M}\right)^{2/3} k_B (T_c - T) \tag{27}$$

Where n and N_0 are the co-ordination numbers or the number of nearest neighboring molecules around one molecule in bulk and on the surface respectively, ρ is the density of the liquid, M is the molar mass of the liquid and k_B is the Boltzmann's constant.

It was shown in Mwakikunga *et al.* [Ref. 55] that based on fundamental thermodynamics, the general relationship between surface tension and temperature is given as [81-83]

$$\sigma(T) = H + T\frac{d\sigma}{dT} \tag{28}$$

where H is the energy required to increase the area of the liquid in contact with air by a unit area. It should be noted that H is always positive. Since σ always decreases as T increase, in accordance also with the Palmer equation in Eq. 27, then the derivative $d\sigma/dT$ is always negative. It can be shown that Eq. 28 carries the same meaning as Eq. 27 with $H = (n/4)(N_0\rho/M)^{2/3}k_BT_c$ and $d\sigma/dT = -(n/4)(N_0\rho/M)^{2/3}k_B$. Based on the two separate relationships of surface tension as a function of pressure according to the current generalization of Lynde's empirical study and temperature from Palmer's theory, one can write a combined relationship as follows

$$\sigma(p,T) \approx \tfrac{1}{4} n \left(\frac{N_0 \rho}{M}\right)^{2/3} k_B (T_c - T)\sigma_0 \exp\left(\pm \tfrac{1}{2} k_\sigma \left(p^2 - p_0^2\right)\right) \tag{29}$$

However, from Lynde's experiments, it is difficult to ascertain the σ-p relationship since the nature of dependence is also dependent on the ρ-p dependence which was not yet known but which will be shown in the sections that follow. Also in Palmer's theory, density of the liquid is assumed constant with temperature. However, so far this could be the only equation that combines the effect of pressure and temperature on surface tension.

There have been other recent $\sigma(p,T)$ equations specific to some materials such as the one by Park and co-workers [83] who showed empirically the effect of surface tension of polystyrene droplets in supercritical carbon dioxide which was found to be

$$\sigma(p,T) = 38.7032 - 0.0559T - 0.01p + 2.596\times10^{-5}\, pT \tag{30}$$

And which was true only in the temperature range from 170°C to 210°C and from pressure of 500psi to 2500 psi. Their experimental results on $\sigma(p,T)$ were plotted on a chart which is reproduced in Fig. 6

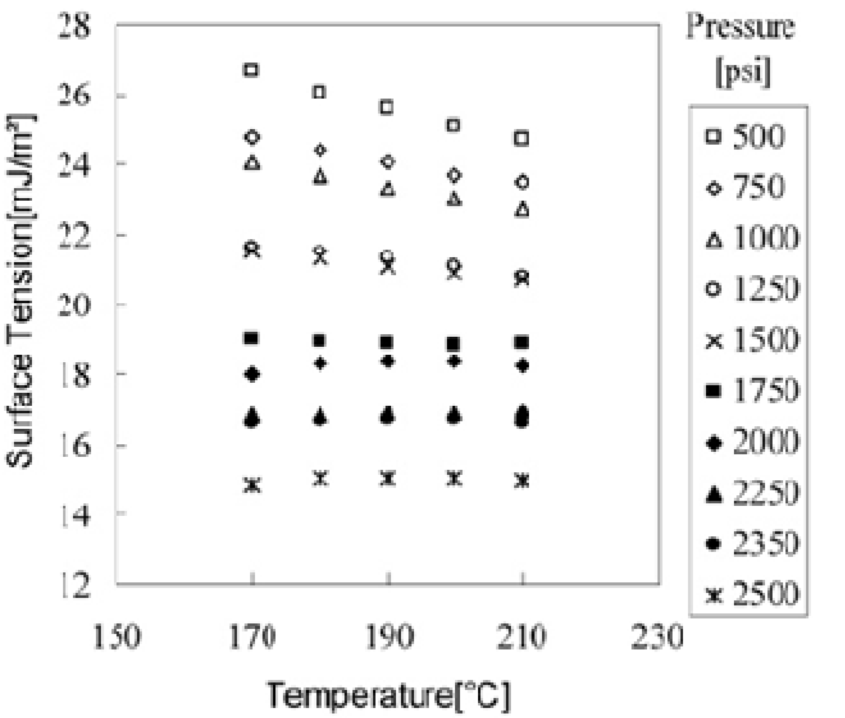

Fig. 6 Surface tension as a function of temperature and pressure for glycerine [From Park *et al.*, J. Phys. Chem. (2007)]

An article on surface tension given by Escobedo & Mansoori (1996) [84] based on the 1923 proposal by Macleod that surface tension of a liquid could be expressed in terms of its vapour ρ and liquid ρ_v densities thus:

$$\sigma = \Pi(\rho - \rho_v)^4 \tag{31}$$

where Π is called the parachor. Although it was thought to be a constant but, lately, it has been realized that parachor is in turn temperature dependent since both surface tension and density are temperature dependent. From statistical calculations, Boudh-Hir & Mansoori (1990) [85] derived an expression for Π of the following nature:

$$\begin{aligned} \Pi &= \tfrac{1}{4} k_B T\left(1-\frac{T}{T_c}\right)^{4-2B} \frac{z}{z_c}\varsigma(\tau,\rho_l,\rho_v); \\ z &= \left(\frac{2\pi mk_B T}{h^2}\right)^{1/2} \exp\left(\frac{\mu_c}{k_B T}\right) \end{aligned} \tag{32}$$

where B is an exponent, the subscript c denotes the critical temperature values, z is the activity, μ_c is the chemical potential, h is the Planck's constant and $\zeta(\tau, \rho, \rho_v)$ is a statistical-mechanical function that shows liquid surface tension dependency on its liquid-state and vapour-state densities and temperature.
Another theoretical and empirical study of the surface tension data by Pandey [86] of ternary liquid system comprising liquid nitrogen, liquid oxygen and liquid argon revealed the relation to take the form a relation developed by Brock & Bird in 1955. This expression for non-polar liquids was derived by utilizing the power law concept applicable to temperature away from the critical point and is here given by

$$\sigma(T) = \left(P_c^2 T_c^2\right)^{1/3}\left(\frac{0.432}{Z_c} - 0.951\right)\left(1 - \frac{T}{T_c}\right)^{11/9} \tag{33}$$

P_c, T_c and Z_c are respectively critical temperature, pressure and compressibility factor.

3.2. Liquid Density as a Function of Temperature and Pressure

When one needs to consider the effects of pressure on density, thermodynamical equations of state (EOS) are used. Wong *et al.* (1996) [87] used the van der Waal's EOS to study the pressure and temperature effects on density of liquid lubricants. They found that density increases with increasing pressure but decrease upon a raise in temperature as confirmed by their experiments. The van der Waal's equation of state for a real gases was used to find the $\rho(p,T)$ expression which was used to modify the droplet equation in Eq. 8 [55]. An improved and more appropriate EOS for liquids was proposed by Redlich & Kwong in 1949 [88] that accurately predicts densities of fluids thus

$$\begin{aligned} p &= \frac{\rho RT}{1 - b\rho} - \frac{a\rho^2}{T^{1/2}(1+\rho b)} \\ b &= \frac{0.08664 RT_c}{P_c}\delta \end{aligned} \tag{34}$$

δ is a parameter which further depends on temperature. By finding ρ as the subject of this equation,

$$\begin{aligned} &\alpha_1\rho^3 + \alpha_2\rho^2 + \alpha_3\rho + \alpha_4 = 0 \\ &\alpha_1 = ab \\ &\alpha_2 = RT^{3/2}b + pT^{1/2}b^2 - a \\ &\alpha_3 = RT^{3/2} \\ &\alpha_4 = pT^{1/2} \end{aligned} \tag{35}$$

one can find the expression of density as a function of temperature and pressure by solving the Eq. 35 and this variation of density is sketched in Fig. 7 (a) and (b)

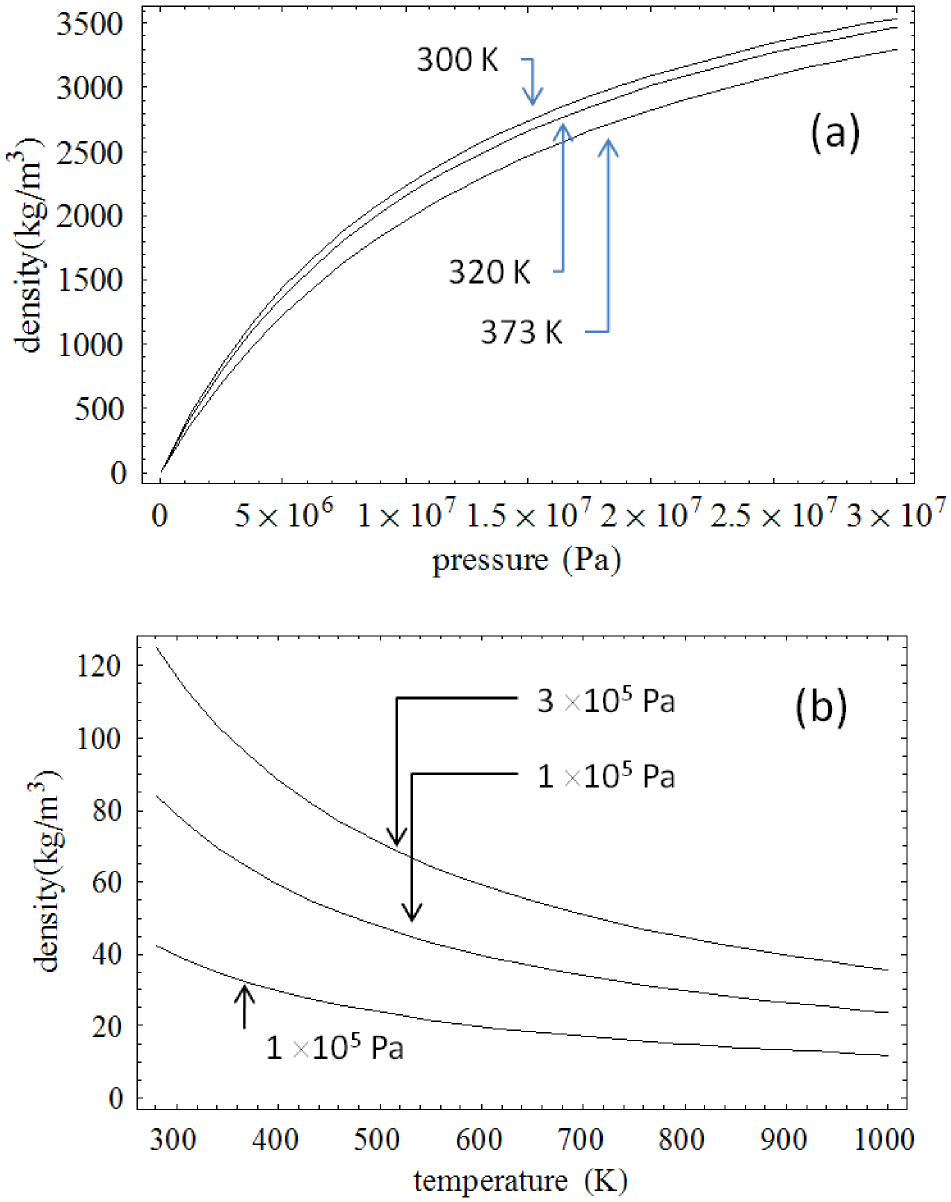

Fig. 7 Plots showing how density varies with (a) pressure and (b) temperature from Eq. 34 (Density values are not realistic and are not specific to any materials)

3.3. Effect of Temperature and Pressure on Liquid Viscocity

The principal observed qualitative facts are that (1) all gases at ordinary pressures become more viscous as the temperature is raised, (2) most liquids become less viscous as the temperature is raised, (3) highly compressed gases resemble liquids, they become less viscous and (4) for a few liquids (such as liquid helium and liquid sulphur).

There is a range of temperatures over which the viscosity increases as the temperature is raised. As was the case with surface tension, the variation of viscosity with pressure is expected to be one of the inverse nature.

It is known from Wright [89] that, as early as 1886, Reynolds proposed an expression for the change of viscosity with temperature for liquids and compressed gases given as $\mu \sim \exp(\text{const}/T)$. This was based on the observation of the similarity of viscous flow to diffusion (diffusion coefficient is given by D being $\propto \exp(const/T)$ and also by regarding the flow of molecules past each other as analogous to a chemical reaction (the effect of temperature on the rate of chemical reaction R being also $\propto \exp(E/RT)$ where E is the activation energy). The general form of the pressure [90] and temperature [91] dependence of viscosity has been known for at least 50 years. Viscosity is now known to vary with temperature in a greater than exponential manner and temperature –viscosity equations generally allow for an unbounded viscosity at some characteristic temperature. At high pressure, the pressure-viscosity response is likewise greater than exponential, often following a less exponential response at low pressures [91].

Fein [92] considered that the low shear viscosity, μ, was an exponential of fluid density. Later, the so called “free volume model was developed [93]. A viscosity model that can describe the temperature and pressure response is the pressure modified equation introduced by Yasutomi *et al.* using the free volume model [94] given here as

$$\mu(p,T) = \mu_g e^{\frac{-2.3\left\langle T-\left(T_{g0}+A_1 \ln 1+A_2 p\right)\right\rangle\left(1-B_1 \ln\left(B_2 p\right)\right)}{C_2+\left\langle T-\left(T_{g0}+A_1 \ln 1+A_2 p\right)\right\rangle\left(1-B_1 \ln\left(B_2 p\right)\right)}} \quad (36)$$

μ_g is the viscosity at a glass transition temperature T_g given by the expression in the triangular brackets. The expression in curly brackets is the relative free volume expansivity and A_1, A_2, B_1, B_2, C_1, C_2 and T_{g0} are parameters that are determined by fitting Eq. 36 to experimental data for a specific fluid. Eq. 36 suggests that increase in pressure raises viscosity whereas the raise in temperature drops viscosity as shown in Fig. 8

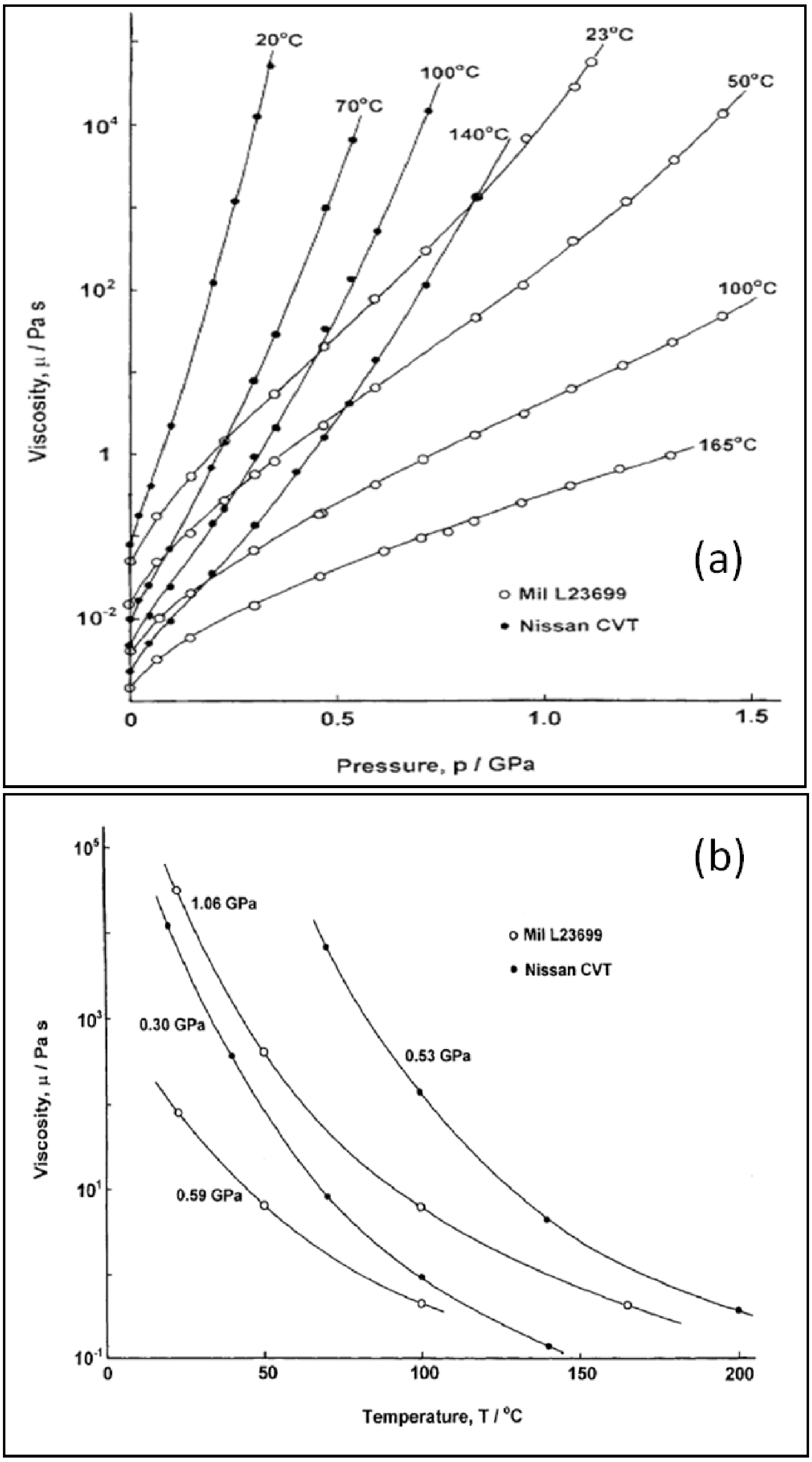

Fig. 8 Variation of viscosity with pressure and temperature (a) and temperature (b) for jet lube Mil L23699 (open circles) and a traction liquid (closed circles) [S. Bair *et al.* (2001)]

3.4. Final Droplet and Particle Size Formula

Every ultrasonic transducer/nebulizer generates heat into the liquid which it is intended to produce droplets from. Since ultrasonic spray pyrolysis set-ups are closed systems, an increase in the temperature accompanies an increase in pressure. The subsequent increases in temperature and pressure affect the density, surface tension and viscosity. As such the droplet size, which is heavily dependent on these parameters, is also affected. In this section, the study on how these changes in temperature and pressure in the precursor liquid would affect the droplet size and hence the final particle sizes after pyrolysis are consolidated.

From sections 2.1 to 2.3 is seen that all the three parameters decrease as temperature is increased. However, as pressure is increased, only surface tension decreases; the other two parameter- density and viscosity- increase.

$$D=\left(\frac{\pi\sigma(p,T)}{\rho(p,T).f^{2}}\right)^{1/3}+$$

$$\left[1+0.0013.(We(p,T))^{0.008}(Oh(p,T))^{-0.14}(I_N(p,T))^{-0.28}\right] \quad (37)$$

$$We=\frac{fQ\rho(p,T)}{\sigma(p,T)} \quad (38)$$

$$Oh=\frac{\mu}{f.Am^{2}\rho(p,T)} \quad (39)$$

$$I_N=\frac{f^{2}Am^{4}}{v_s.Q} \quad (40)$$

After substituting the pertinent parameters, the droplet size can be written in terms of the temperature and pressure dependent liquid density, viscosity and surface tension from Eqs. 29, 35 and 36 as

$$D=1.14\times10^{-4}\left(\frac{\sigma}{\rho}\right)^{1/3}+0.021\frac{\left(\frac{\rho}{\sigma}\right)^{0.008}}{\left(\frac{\mu}{\rho}\right)^{0.14}} \quad (41)$$

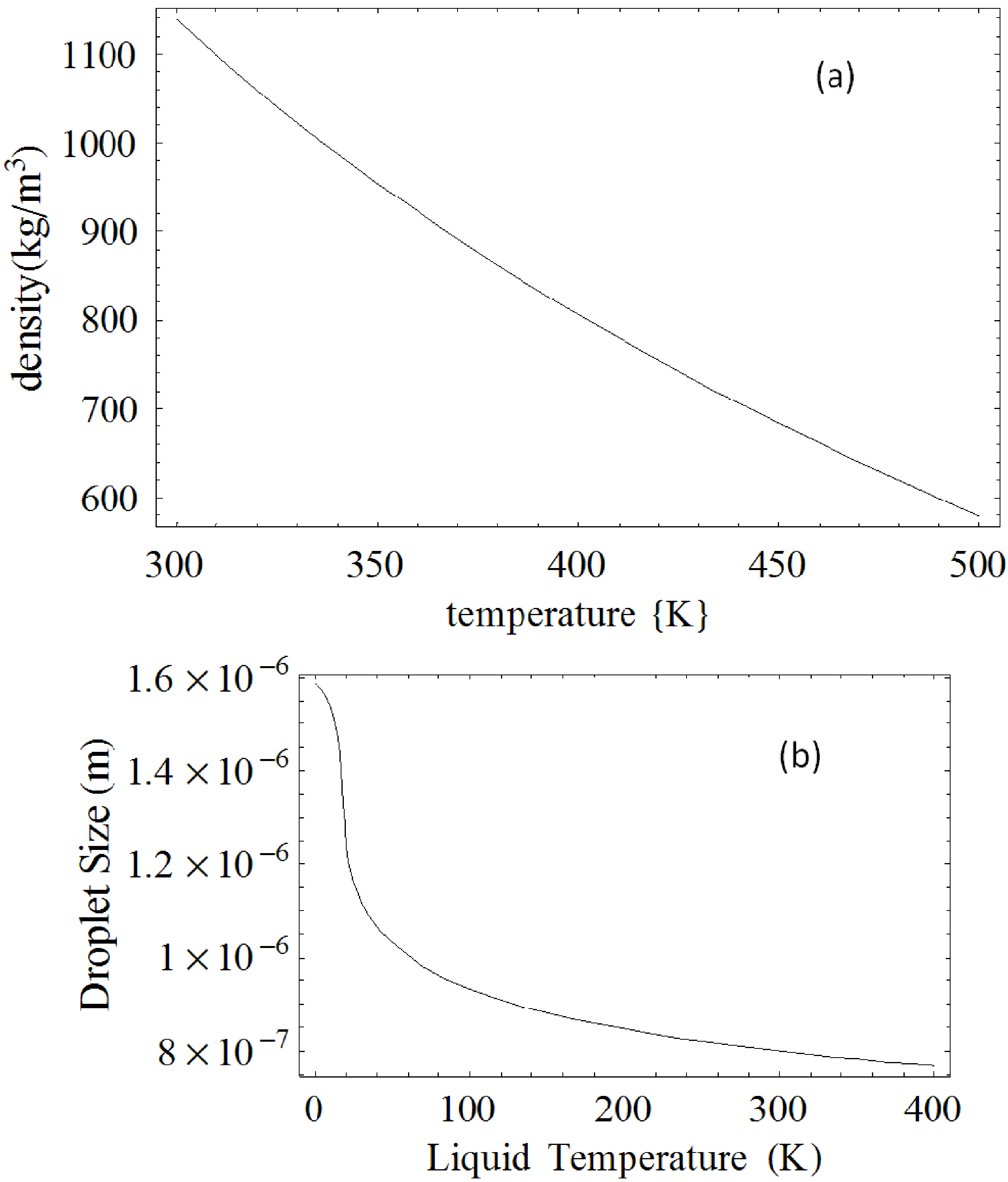

Fig. 9 A plot showing the variation of density (Eq. 35) and droplet size (Eq. 41) with liquid temperature

The density-temperature function was adopted from the Redlich-Kwong equation in Eq. 34, surface tension was taken from the presently derived expression from Lynde and Palmer theories given in Eq. 29 and viscosity-temperature profile was determined from Eq. 36, plotted in Fig. 9, shows the variation of droplet size as a function of liquid temperature. The droplet size decreases as temperature is increased.

The small changes in liquid pressure in a typical pyrolysis session lead to very small changes in surface tension, density and viscosity and hence on the droplet size. The droplet-size versus liquid pressure is therefore not shown.

4. Theory of Pyrolysis for Predicting Final Particle Size

Pyrolysis is an application of the phenomenon of droplet generation from liquids by ultrasound waves. It involves materials deposition by carrying the so-produced liquid droplets into a heated zone where the droplets undergo (1) evaporation, (2) decomposition (3) reaction into new products and (4) condensation of the new product onto a filter or a substrate [Fig. 10]

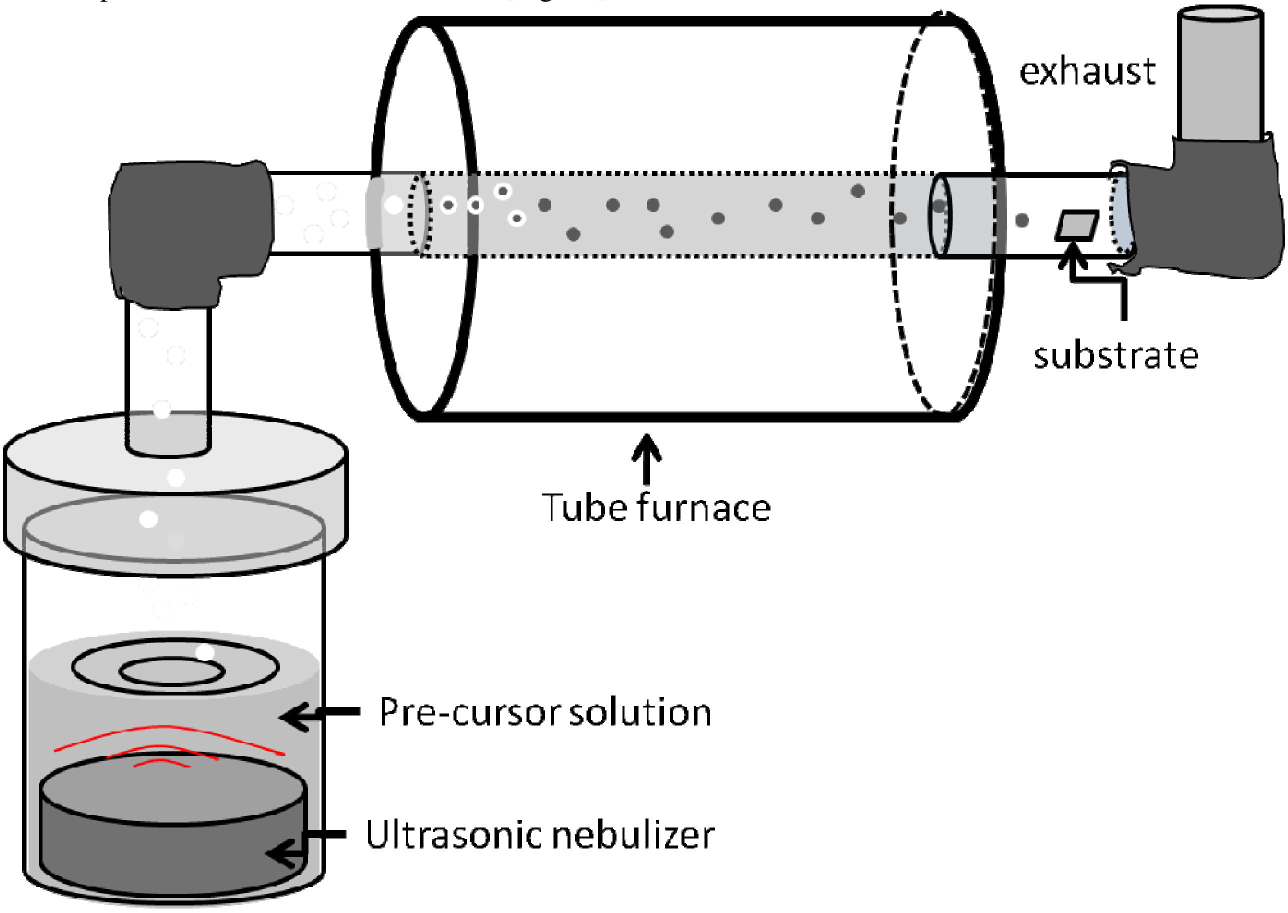

Fig. 10 Simple schematic of ultrasonic spray pyrolysis showing an ultrasonic nebulizer immersed in the precursor solution where droplets are generated and transport into a tube furnace for eventual pyrolysis and deposition onto substrates.

The theory of transitions of the liquid precursor droplet of initial diameter, *D*, in the heat field and the consequent transformation into a new material particle of diameter *d* is simple. During the preparation of the precursor solution suitable for spray pyrolysis, a precursor material of mass m_{pr} is dissolved in a solvent so that if the concentration of this precursor in the solvent is c_{pr} then

$$m_{pr} - c_{pr}\frac{4}{3}\pi D^3 \tag{42}$$

D is dependent on both frequency of the sound and the concentration of the precursor [348] as shown in the previous sections and as illustrated in Fig. 11 with data taken from Gurmen's group [348,353,391]

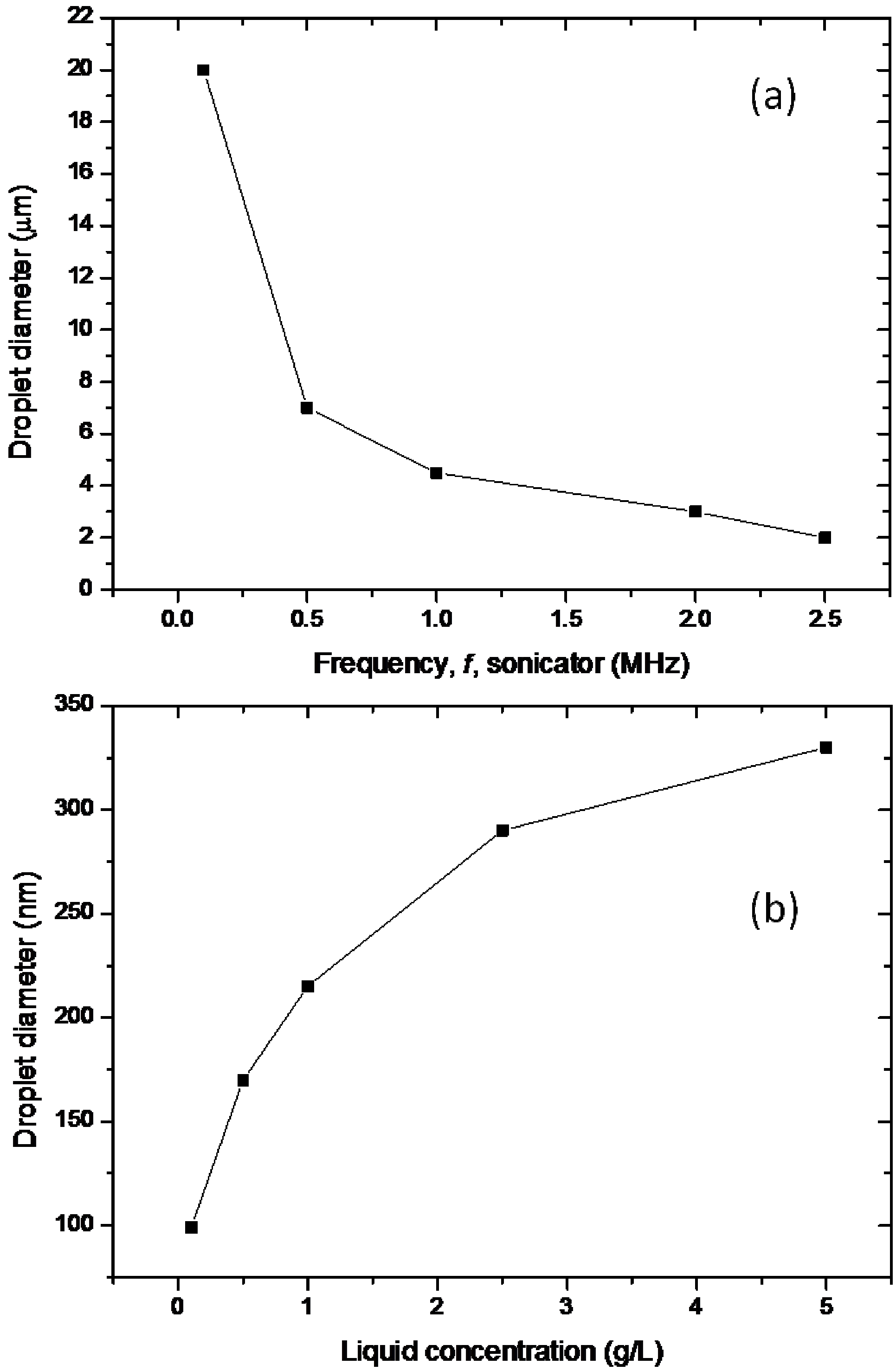

Fig. 11 Experimental observation of the dependency of droplet and hence particle size on nebuliser frequency (a) and precursor concentration (b). Data taken from S. Gurmen *et al.*, Mater. Res. Bull. (2006)

After pyrolysis–dissociation and decomposition – the precursor material, a remnant of evaporation, is further reduced to the final particulate of mass of m_p plus other species that mostly are in gaseous state and hence evaporate off without depositing. The particulate mass after assembly can be written as

$$m_p = \frac{M_p}{M_{pr}} m_{pr} \qquad (43)$$

M_p and M_{pr} are the molecular masses of the final particle and the precursor material respectively. Assuming that the initial liquid precursor droplets and the final solid particles are spherical Eq. 42 and Eq. 43 can be combined to give

$$\tfrac{4}{3}\pi\rho_p d^3 = \frac{M_p}{M_{pr}} c_{pr} \cdot \tfrac{4}{3}\pi D^3 \qquad (44)$$

d is the final particle diameter. This simplifies to the following equation

$$d = D\left(\frac{c_{pr} M_p}{\rho_p M_{pr}}\right)^{1/3} \qquad (45)$$

Eq. 43 has been widely used by a number authors employing ultrasonic spray pyrolysis in production of nano-particles to predict the final particle sizes.

5. Popularity of Ultrasonic Spray Pyrolysis

USP as an application of the ultrasonic droplet generation phenomenon has attractive features, like the traditional spray pyrolysis, of simplicity, economic viability, high deposition rate, possibility of coating over large areas and continuous operation [55]. But unlike other commonly known pneumatic atomizers, it has been described to possess the advantages of "… chemical purity and stoichiometry" and allows a narrow distribution of particle sizes. A large proportion of the droplets is below 20 µm and these are produced with low in-flight speed. This prevents the droplets from being removed from the gas phase by impact onto the walls of the reactor and through droplet-to-droplet collisions and consequent coalescence. The major disadvantages are potential for hollow structure or fractured particles which could be good for other applications and that the droplet production rate is typically low and highly dependent on the throughput of the nebulizer.

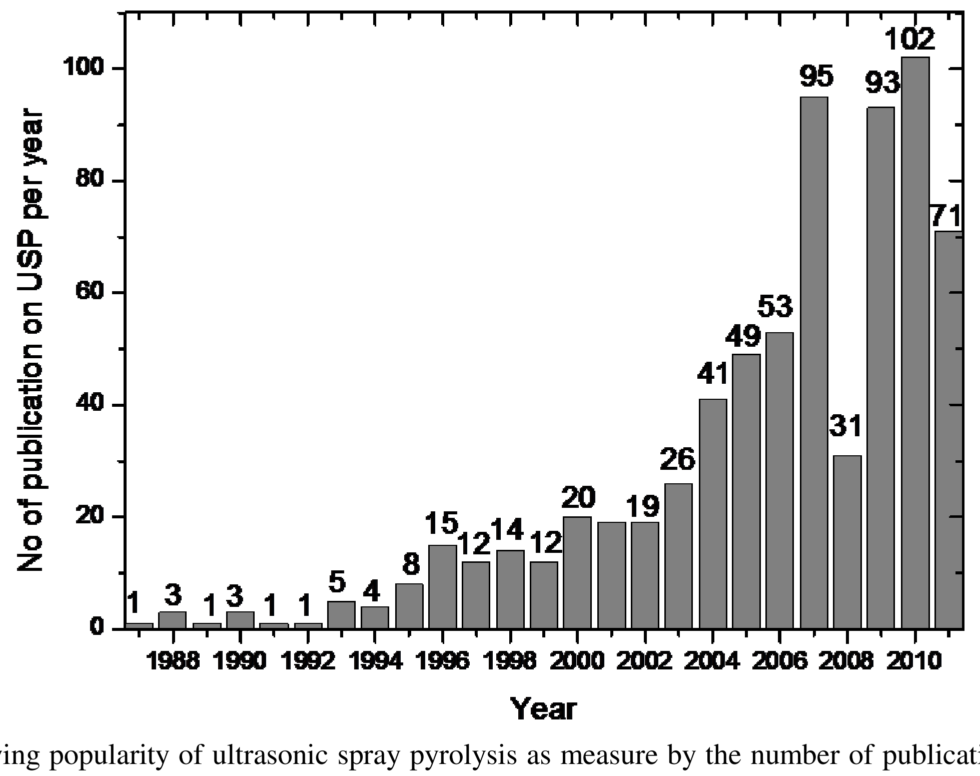

Fig. 12 Growing popularity of ultrasonic spray pyrolysis as measure by the number of publications (journal papers and conference proceedings) released per year since 1988 [96-512].

Since there are numerous publications, in the period from 1988 to the present (more than 730) [96-512], on materials processed using ultrasonic pyrolysis, it was seen as convenient to plot a time series graph as illustrated in Fig. 12.

6. Parameter Optimization in USP: Droplet Residence Time

One of the most important parameters for optimization of ultrasonic spray pyrolysis is flow rate of the precursor droplet. At extremely low flow rates, the throughput of the USP system is small at the benefit of obtaining truly nano-sized, nano-structured and completely-decomposed targeted materials. At extremely high flow rates, yield is high but complete decomposition of the precursor is compromized as the residence time of the precursor in the heated zone is small. An optimum flow rate is therefore necessary to obtain both high yield and pure materials. The relationship for residence time can be easily shown to be *d* and *L* are the diameter and length of the reactor respectively and Q_0 is the flow rate of the precursor assuming that the velocity of the carrier gas is the same as the velocity of the carried precursor droplets.

$$t_{residence} = \frac{\pi d^2 L}{4Q_0} \tag{46}$$

In the real case where the above assumption does not apply, temperature, T, and pressure, p, of the system are taken into account. In this case then the expression for residence time is given by C. Michel *et al.* (2006) [191] as

$$t_{residence} = \frac{\pi d^2 L}{4Q_0}\left(\frac{P_0}{P}\right)\left(\frac{T_0}{T}\right) \tag{47}$$

T_0 is room temperature, P_0 is the atmospheric pressure. In order to maximize t_{res} one can increase *L* to the maximum possible length. Increasing *L* has the disadvantage of an uneven temperature profile over a long distance.

One then needs to have several short heating zones whose temperature profiles are constant and manageable. Since the nature of products from USP depends in part on the control of the furnace temperature, Taniguchi and co-workers [137, 142,164,179,182,187,250,263,303,310,327,387] have made an elaborate setup with a furnace having several heating zones. A typical example of such multi-zone furnaces was well illustrated by Taniguchi's group [137]. This is illustrated in Fig. 13

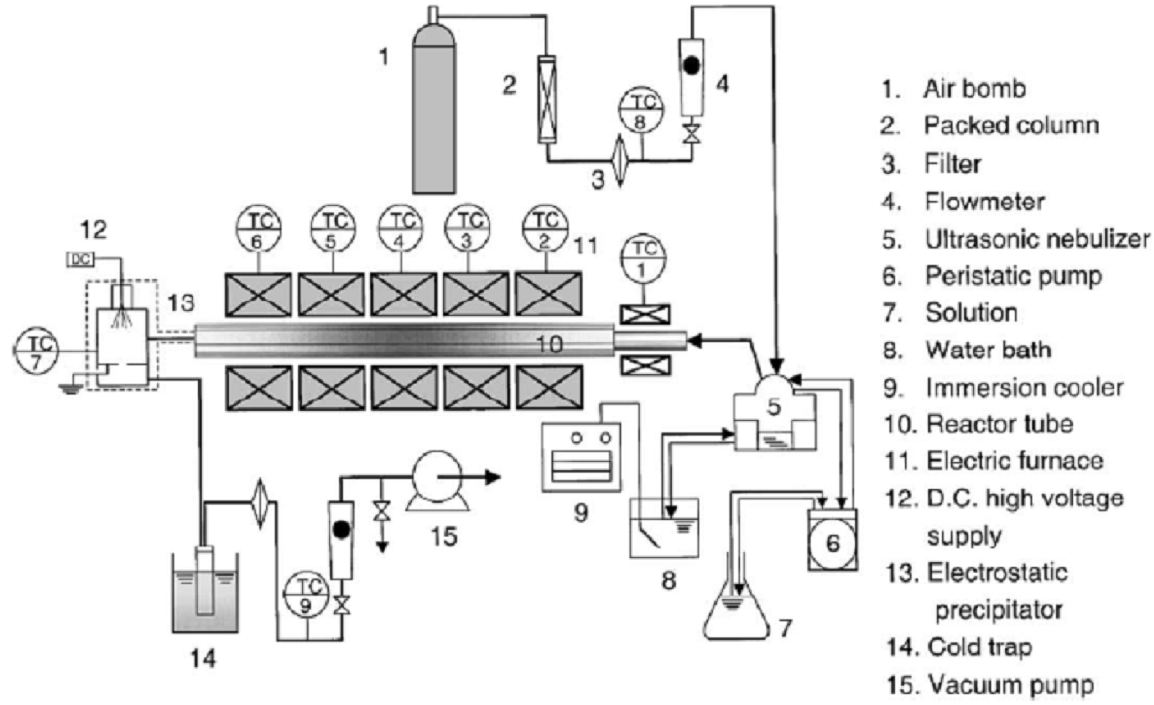

Fig. 13 A typical example of an ultrasonic spray pyrolysis employing a multi-zone furnace for control of product shape, particle size and other parameters. From Taniguchi's group.

7. Various Forms of USP

Worth noting are a few USP set-ups that have attracted attention through the years and the novel nano-structured materials they have produced. A setup by CNR Rao's group illustrated in Fig 14 had an ingenious provision for constant precursor liquid level apart from the usual USP components [97,99].

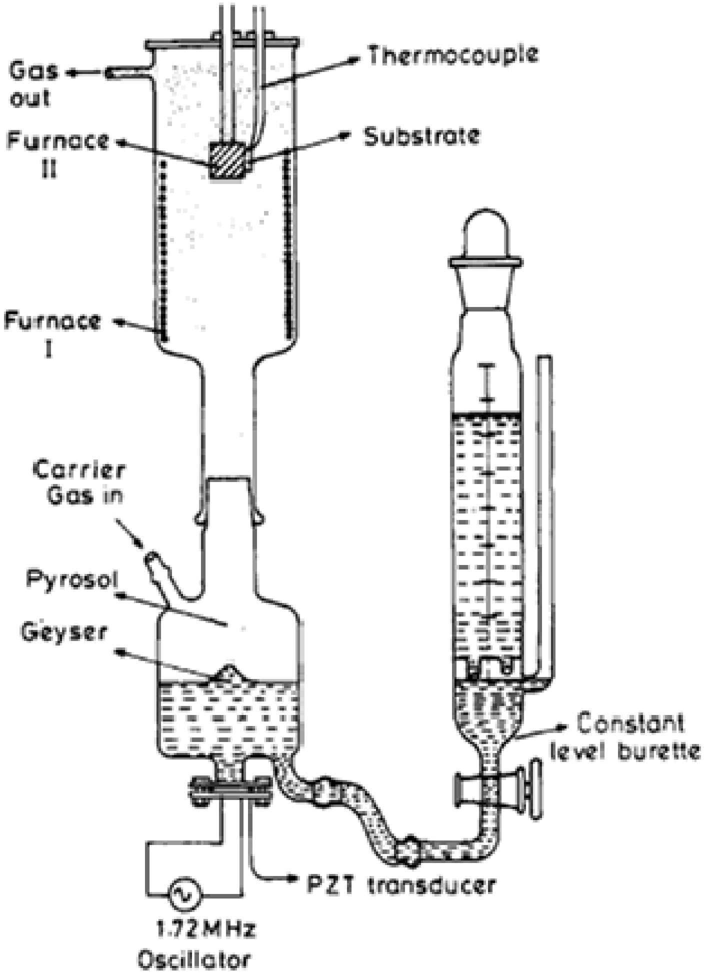

Fig. 14 A nebulised spray pyrolysis by C.N.R. Rao's group (from Ref. 97,99)

7.1. Asynchronous Pulse USP

The asynchronous-pulse ultrasonic spray pyrolysis (APUSP) is another interesting design suitable for growth of stacked films or controlled doping and development of superlattices [106,157,158,160]. In APUSP, two more ultrasonicator-containing chambers are harnessed. Each chamber contains the appropriate precursor solutions that are to be deposited – the host and the dopant etc. The "sonicators" in these chambers are controlled by a pulse generator one at a time in an asynchronous manner. The period of each chamber's pulse determines the level of doping, or the thickness of the layers in the superlattices.

In a typical APUSP (Michel Lopez and Zea 2006 Ref. 191) an inert gas is first introduced to the reaction chamber at relatively low and steady flow rate for about 30 minutes to drive the air out. The nebulised solutions – precursor and dopant are delivered to the substrates in pulses through the nozzles.

Each spray lasts 5 seconds for both but after the spray of the dopant is conducted, a delay of 2-4 seconds was employed to ensure that the introduced dopant was completely decomposed before conveying a pulse spray of the host. The deposition is carried out by repeatedly performing these spray processes. It took 12-14 s for each cycle and the deposition time lasted for 15 -30 mins for the preparation of one sample.

An appropriate interval between the pulse spray of dopant and host solutions play an important role in depositing high crystallinity films. There are cases where the precursor liquid to be sonicated passes through the ultrasonicator and introduced from the top rather than from the bottom.

This design has the advantage of high yield of final desired product. However, introducing droplets from below has the advantage of selecting on the small droplets with most of the large one returning to the precursor under gravity. In both the Lee *et al.* (1998) [106,157] set up and that of Patil & Patil (2000) [73,104,120,124,374,375,430,439,492] the substrate has its own special heater apart from the standard furnace. Contrary to heating substrates, Kang & Park (1999) [113,114,118,145,161,170,176,178,198,239,262], realised that subjecting the particle collector to coolants such as liquid nitrogen helped prevent Ag nano-particle agglomeration and they become well dispersed in ZnO.

Note that in their USP design, they included a temperature controlling water bath around the precursor container to prevent changes in temperature and pressure which in turn have an effect on droplet size as shown in the previous sections.

7.2. Electrostatic Nebulizer USP

Recently, another innovation to USP [Zaouk *et al* Ref. 206, Chen *et al* Ref 240, 249, Chang & Hwang, Ref. 304, Bin *et al* Ref. 332, Lee *et al*, Ref. 458 and Min *et al* Ref 502, 503] has been the manner in which droplets are produced from the precursor liquid. Apart from spraying with ultrasonic nebulisers, it has been realized from the days of Lord Kelvin that by applying a high potential difference to the liquid surface makes such a surface erupt into liquid droplets. In electrostatic assisted USP (EAUSP), a high tension is applied between the liquid and the substrate. There are cases where the precursor liquid to be sonicated passes through the ultra-sonicator and introduced from the top rather than from the bottom

This design has the advantage of high yield of final desired product. However, introducing droplets from below has the advantage of selecting on the small droplets with most of the large one returning to the precursor under gravity. If a liquid is forced to flow through a small nozzle which is subjected to an electric field, the liquid will exit the outlet in different forms or modes as a result of different electrodynamic mechanisms. These modes include among others dripping mode, cone mode, cone-jet mode and spindle mode [Zaouk *et al.*, 2000 Ref. 206]. The kind of mode the spray displays depends on the electrical potential applied to the nozzle, the flow rate, the conductivity and the surface tension.

For film deposition, the cone-jet mode is the most suitable, it is a continuous mode and the formation of a homogeneous fine spray is possible. In this mode, there exists the so-called "Taylor cone" with 49.3^o half angle at the apex of the cone (see Taylor instability in the previous section). This cone is extended by a jet which breaks up into spray droplets to generate an aerosol of the precursor liquid. Chen *et al.* [140] final particle morphology has large particles and large flakes. This could be due to agglomeration of droplet as they descend.

The best way to select only the small droplets is to have the substrate above the spray. The large ones cannot make it to the substrate and therefore are forced to return to the ultrasonic nebuliser. As for Zaouk *et al.*, the potentials need to be optimized for self assembly.

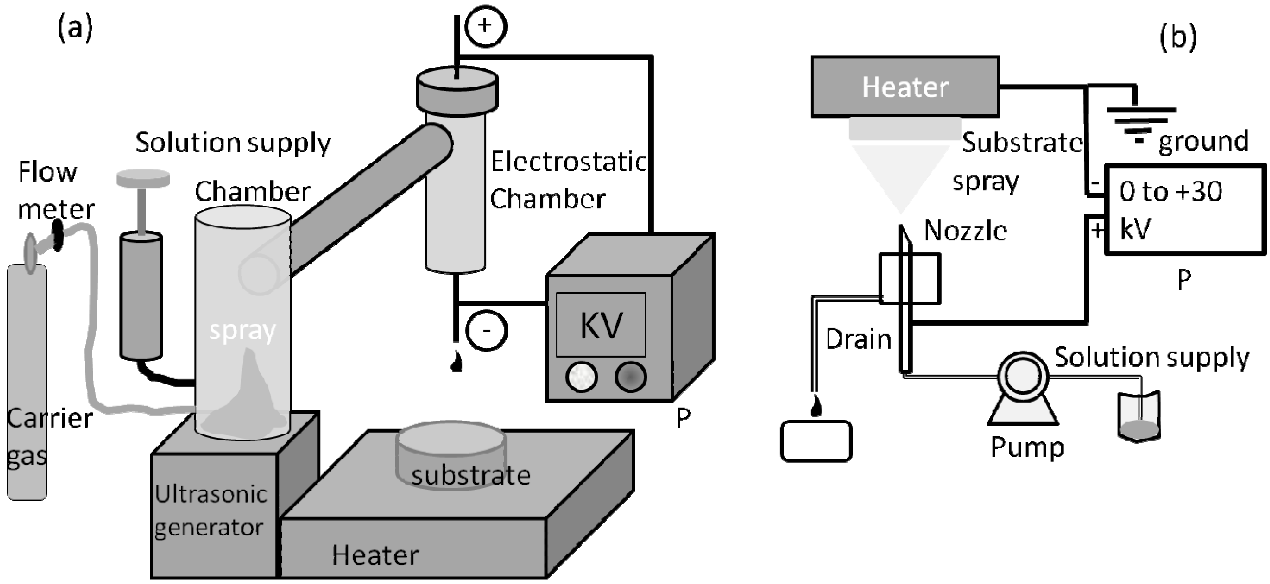

Fig. 15 (a) Electrostatic assisted USP (EAUSP), note that aerosol are directed onto the substrate from the top (Redrawn from Chen *et al.*, Mater. Res. Bul, 2007) (b) Electrostatic spray deposition (ESD); note that deposition is from bottom to top (Adapted from Zaouk *et al.*)

7.3. Infrared USP

An interesting USP system employing a novel heating source was reported by Matsuzaki and co-workers [117] when synthesizing yttria stabilized zirconia thin films. Their substrates temperatures were controlled by heating a "susceptor" with an infrared radiation heater.

The substrate temperature could be tuned from 873 K to 1023 K. It is interesting to note that grain size increases with substrate temperature, the Arrhenius plot shows that the activation energy for yttria stabilized zirconia is about 68 kJ mol^{-1} and grain size increases with increase in deposition rate. The particles obtained by this work were rather large in general. This could be due to (1) agglomeration at higher substrate temperatures an effect known as the Oswald's ripening (2) spraying from the top as alluded to before.

The Ostwald's ripening observed here should be distinguished from the opposite effect which was observed recently and reported in Mwakikunga *et al.* [55,243-247,333,511].

Mwakikunga *et al.* found spheres of WO_3 obtained from USP to shrink in diameter as the furnace temperature was increased without heating the substrates where the perfect sphere would land. In the case of shrinkage in diameter as a function of surrounding temperature, it was found that the data was in agreement with the Tiller equation given as

$$d_c = \frac{4\sigma_e \Omega_M}{RT \ln\left(p/p^*\right)} \quad (48)$$

σ_e is the interfacial energy between the nucleating materials and the surrounding environment and Ω_M is the molar volume of the nucleating material. A number of authors have used this equation in explaining the growth of nano-wires by chemical vapour deposition [Tan *et al.*, APL (2003) Ref. 148]

7.4. Flame-Assisted USP

An interesting kind of pyrolysis is called flame assisted USP (FAUSP) [108,130,159,208,209,241,344]. FAUSP was developed in the 1980's. It operates by injecting the spray of a precursor solution obtained from an aerosol generator into a combustion chamber where the individual droplets are rapidly combusted. Fuel such as natural gas or hydrogen is introduced in order to generate the appropriate high temperature. In some case, instead of external fuels, flammable alcoholic solutions are used as precursors.

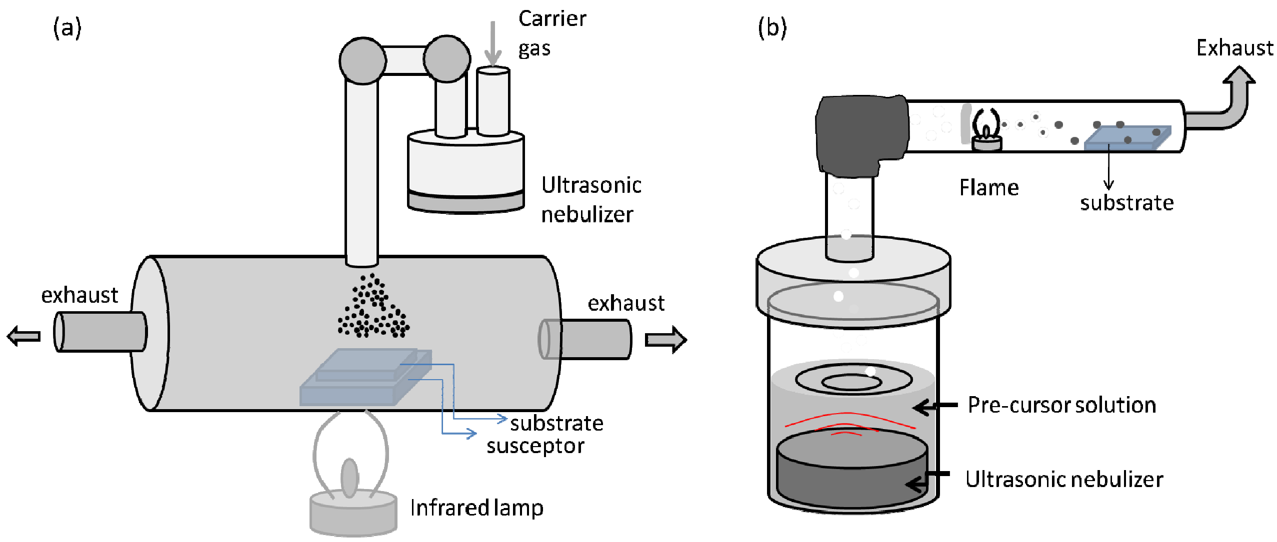

Fig. 16 (a) Infrared USP and (b) Flame assisted USP (FAUSP) Redrawn from: Chen *et al.*, Eur. J. Solid State & Inorg. Chem. (1998)

8. Morphology, Structural and Other Properties of Materials Obtained by USP

8.1. Solid and Hollow Spheres

The spherical shape of the particles definitely comes from the spherical droplets from the precursor liquid. When scanning electron microscopy is performed on these particles one can see the manifestation of spherical daughter particles from mother spherical liquid droplets. The particle size may be less than 3 µm [see Fig. 18 from Oh *et al* Refs. 169,176,184,198,199,202,227,239,432,438] from the four SEM micrographs (a) but at higher magnification with TEM (bottom right) the morphology changes to one showing that the spheres are composed of numerous crystallites whose size as determined by the Scherrer equation from X ray diffraction shows they are nano-crystalline. The crystallite size increases as the calcinations temperature is increased. Bucko, Ref. 209]. This is equivalent to increasing substrate temperature and thereby increasing particle and crystallites sizes as seen above. However, this is to be contrasted from the in-situ furnace temperature increase which has the reverse effect of decreasing the particle and crystallite size as shown in Fig. 20 [from M Yuan *et al.* (1998) [Refs 108, 112] also found hollow spheres when preparing zirconia and yttria-stabilized- zirconia (YSZ) fine powders by flame- assisted ultrasonic spray pyrolysis. This was attributed to the presence of nitrates in the precursor.

Prior to this study, Messing *et al.* [96,98] had studied the spray pyrolysis of nitrate solutions and proposed a mechanism to explain the particle morphology. During the pyrolysis of spray droplets in the flame, the evaporation of the solvent and the reaction/decomposition of the solute proceed successively from the outer part to the inner part of the droplets. When a nitrate solute with a relatively low melting point is present, it melts to fill the pores of the structure. The molten salt will inhibit the removal of the trapped solvent in the inner parts of the droplets as a result of the reduction of the gas permeation. This leads to a build-up of internal gas pressure and, finally, explosion or foaming of the particles to form hollow particles or particle fragmentation with a broad size distribution.

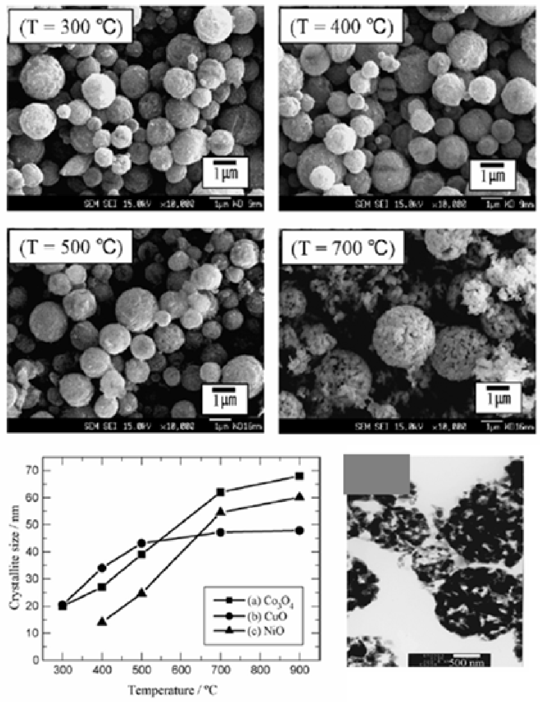

Fig. 17 The figure shows the effect of calcination temperature on morphology and crystallinity of Co_3O_4, CuO and NiO. From S. W. Oh *et al.*

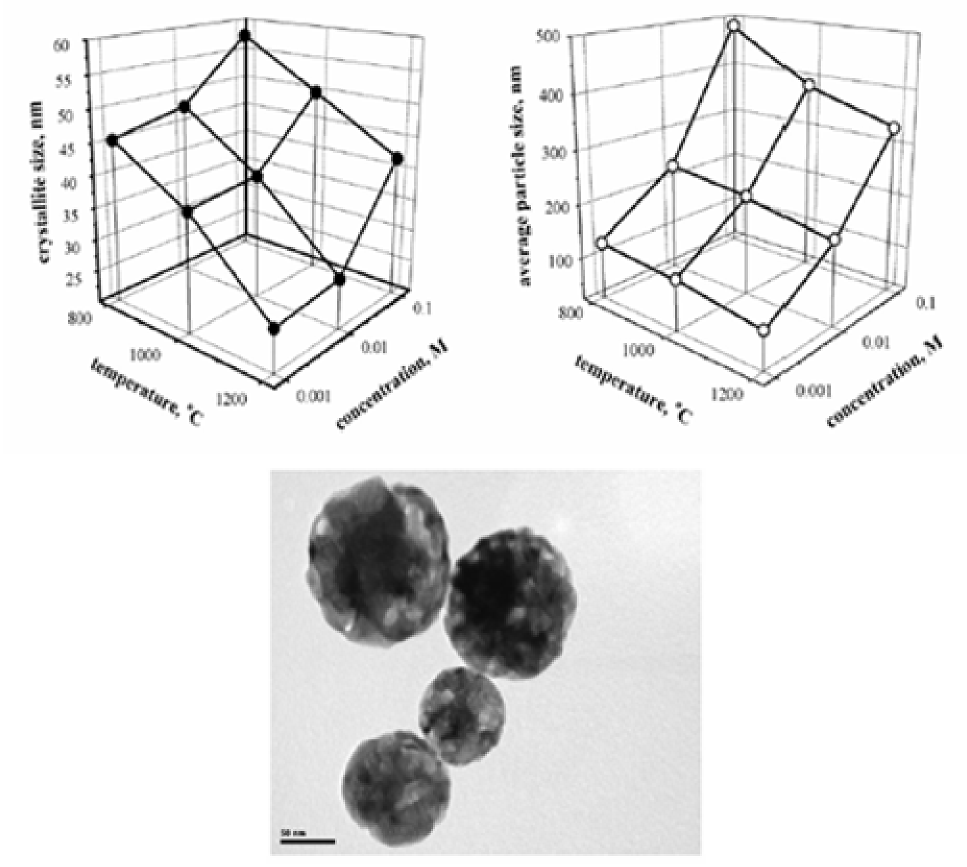

Fig. 18 Effect of furnace temperature during synthesis on the morphology and crystallinity of $BZrO_3$ nanopowders by USP (from M.M. Bucko & J. Obłąkowski, *J Eur Ceram Soc.*, (2007) [Ref. 209])

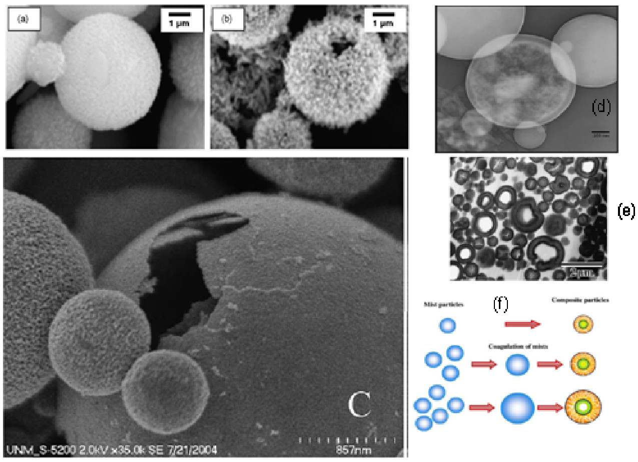

Fig. 19 (a) and (b) SEM images of hydroxyapatite (Hap) powders by the USP/SAD method showing the gaping hole in one of the spheres in (b) an indication of the possibly hollow nature of these spheres [From G.-H. An *et al.*, Mater. Sci. Eng. (2007) Ref. 163] (c) More vivid proof of hollow NiO–$Sm_{0.2}Ce_{0.8}O_{1.9}$ composite spheres [S. Suda *et al.*, Solid State Ionics (2006) Ref. 196] (d) and (e) HRTEM image of LiFePO4/C composite prepared at 450°C showing a shell structure and the intersection of the shells of other spheres [From M. R. Yang, J. Power Sources (2006) Ref. 201,204] (f) A conceptual model proposed by Yang *et al.* (2006) on how the hollow LiFePO4/C composites form with or without voids.

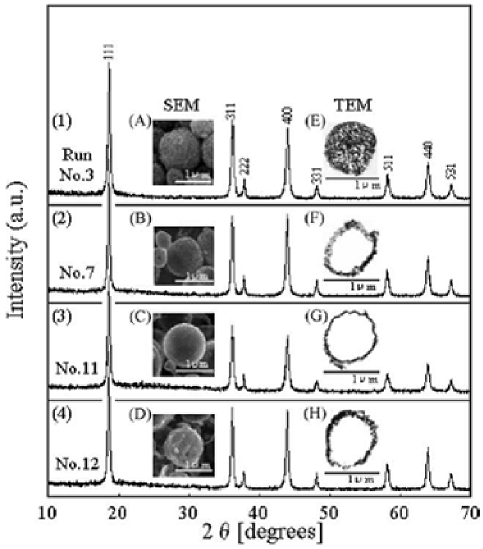

Fig. 20 Examples of XRD, SEM and TEM micrographs of $LiMn_2O_4$ particles prepared from various precursors (1) dense $LiMn_2O_4$ with porous surface structure (2) hollow $LiMn_2O_4$ particles with hybrid surface structure (3) hollow $LiMn_2O_4$ particles with smooth surface structure and (4) hollow $LiMn_2O_4$ with shrinkage surface structure. From Matsuda & Taniguchi, *Journal of Power Sources* (2004)

8.2. One-Dimensional Nanostructures from USP: Nanowires, Nanoribbons, Nanorods

Of interest, apart from the production of nano-particles by ultrasonic spray pyrolysis, has been the attainment of one-dimensional structures. Many of the one- dimensional structures have been micro-sized such as the ZnO nanorods grown almost at right angles to the substrate surface [212,214,233] as shown in Fig. 21. This one-dimensional growth only happens at specific conditions. Note that as furnace temperature is reduced that micro-rod diameter decreases.

Another interesting case of one dimensional growth by USP was observed by Htay *et al.* [242,254,317,367,477] who reported micro-sized platelets, wires and tips of ZnO obtained at controlled conditions. Temperature of synthesis was found to dictate the morphology of the micro and submicron-structures that they obtained. In this case different furnace temperatures yield different structures- rods, wires or platelets. One-dimensional growth from spheres of WO_3 which transform themselves into W_xO_y nanowires after thermal annealing at 500^{o}C in argon for 17 hours [Ref. 243-247,333,511] has been observed.

Recently dense one-dimensional nano-ribbons of VO_2 grown by USP at 700^{o}C in argon carrier gas without the need for thermal annealing [Fig. 24 and 25] were also observed [unpublished]. Their electronic transition temperature at 70^{o}C was confirmed using a four-point probe technique. It was found that for the same synthesis conditions, furnace temperature, precursor flow rate etc, vanadium oxides yielded mostly nanobelts, nanoribbons and sheets where tungsten oxides showed nanowires and nanorods.

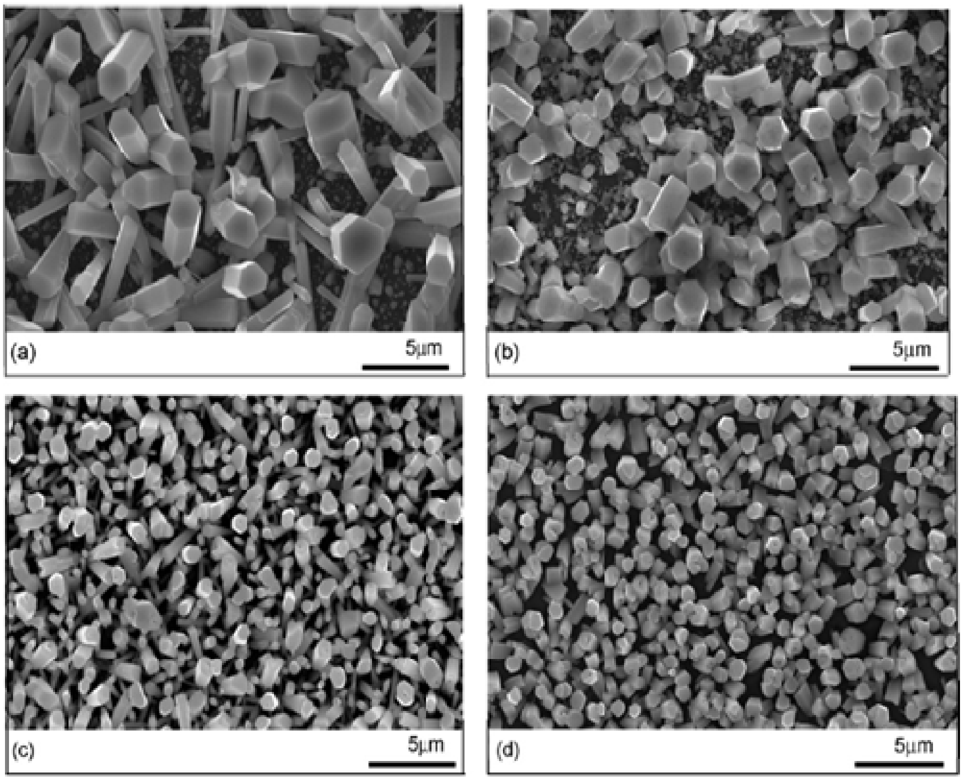

Fig. 21 SEM images of ZnO microrods deposited by USP at (a) 550^{o}C (b) 500^{o}C, (c) 450^{o}C and (d) 400^{o}C [From U. Alver *et al.*, Mater. Chem. Phys. (2007) Ref. 233]

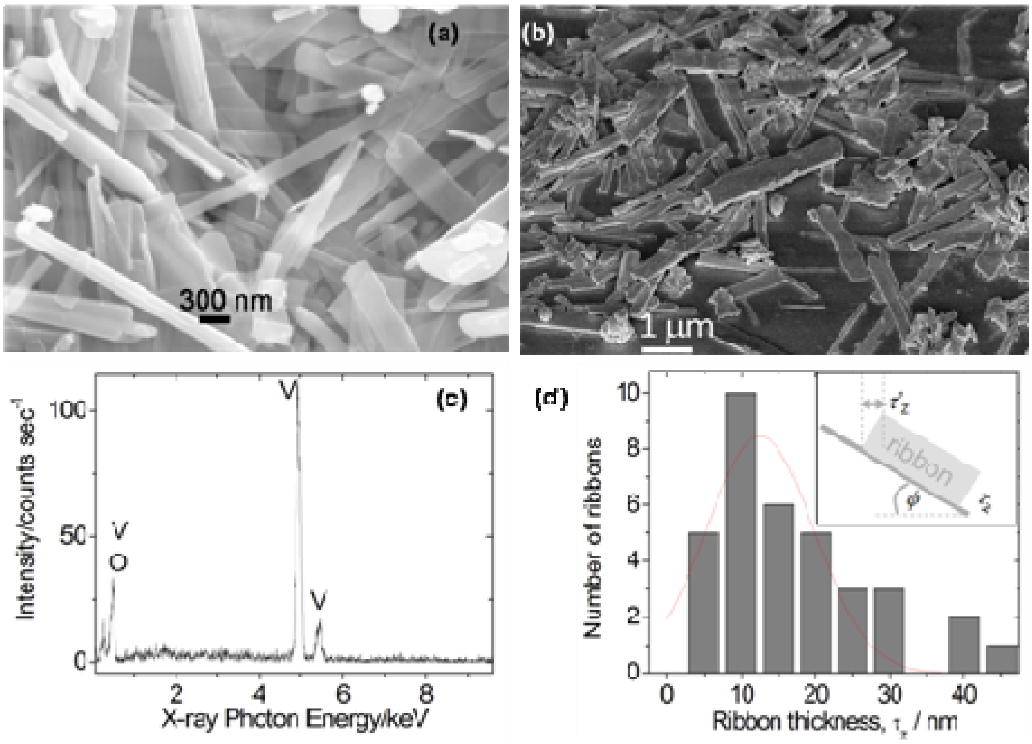

Fig. 22 (a) SEM micrograph of VO_2 nano-ribbons (b) tilted at $\phi = 54°$ (c) an EDS spectrum showing the V and O peaks on a carbon adhesive tape substrate and (d) size (thickness) distribution histogram (thickness determined from $\tau_z = \tau'_z/\sin\phi$ as illustrated in the inset of (d))

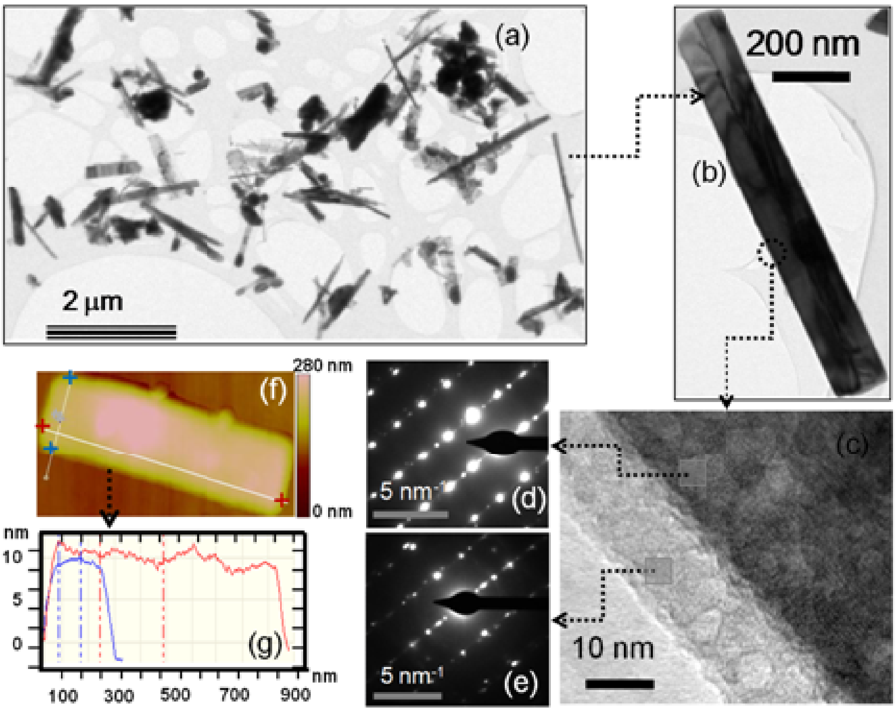

Fig. 23 Transmission electron microscopy (a) low resolution image (b) low resolution on a single ribbon (c) higher resolution on the edge of ribbon revealed bi-layered structure: V_2O_5 and VO_2 and in some ribbons a core-shell structure. (d) and (e) are SAED patterns for V_2O_5 and VO_2 regions respectively (f) AFM height image of a single VO_2 nano-ribbon. The profile (g) shows that the VO_2 ribbon is typically 10 nm thick.

9. Conclusion and Outlook

The review has shown the humble beginning of the ultrasonic spray pyrolysis: from the phenomenon of ejection of liquid droplets by high frequency sound waves called ultrasound since Michael Faraday to the highly sophisticated thin film and powder production technologies employing this phenomenon. Since then some theories and experiments have been performed to explain this phenomenon. One mechanism is the capillary wave mechanism where sound waves operate only on the liquid surface. Droplet size depends on surface tension, liquid density and the frequency of the sound. In the cavitation mechanism, sound waves may introduce turbulence in the bulk of the liquid leading to cavities which may also erupt to the surface in a random fashion but whose distribution is described by the Weber number, the Ohnesorge number and the so-called Intensity number. In this case, the liquid droplet size, apart from depending on surface tension, density and frequency of the ultrasound wave, also depends on the viscosity and the stated numbers.

We have also introduced the thermodynamics of how the droplet size should change when the temperature and pressure in the liquid changes in which case density, surface tension, density, viscosity and hence the Weber and other numbers also vary. The review also covers the applications of these phenomena which culminate into what has been branded ultrasonic spray pyrolysis. Publications reporting synthesis of various materials by this method are shown to increase very rapidly showing that there is this method is growing in popularity aroung the world. From the trend of publications per year, it has been demonstrated that USP will be a standard method in many labs in the next generations.

The success of any user of this method will depend on the understanding of the dynamics of particle generation from droplet formation to the deposited particles which is lacking in many texts.

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