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From
Physics World
April 1996

© IOP Publishing Ltd 2007

ISSN: 0953-8585

Institute of Physics Publishing
Bristol and Philadelphia

Downloaded on Thu Jun 28 08:11:53 BST 2007 [131.211.208.145]

Spherical particles suspended in a liquid behave like large idealized atoms in many ways, but they also show a range of fascinating new phenomena

Colloids in suspense

WILSON POON, PETER PUSEY AND HENK LEKKERKERKER

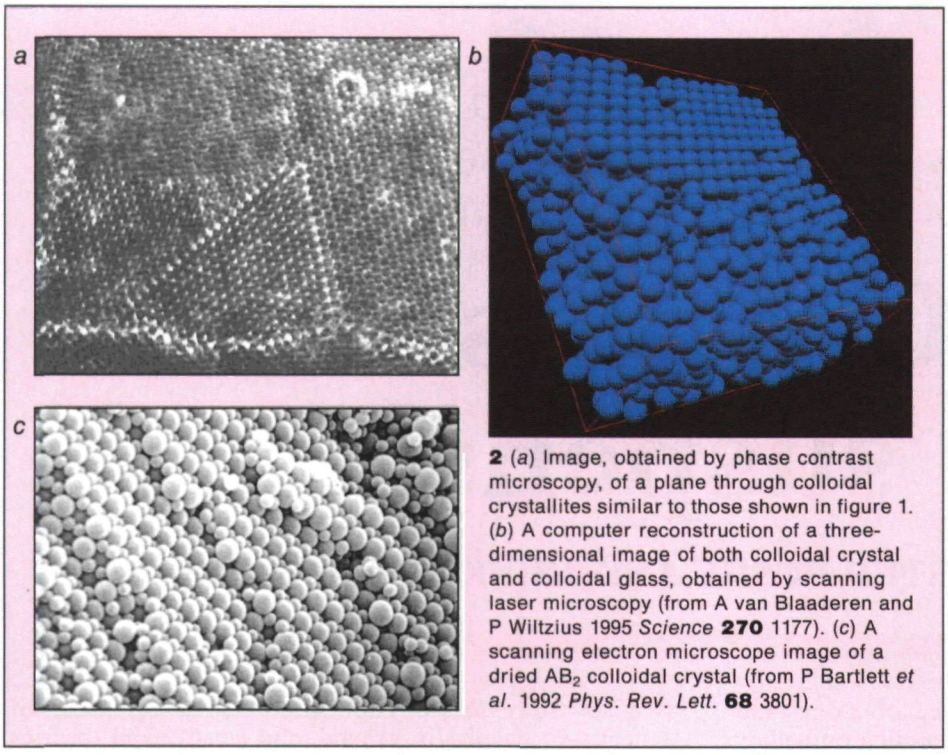
TODAY we do not doubt the existence of atoms and molecules – we can "see" them using scanning tunnelling and atomic force microscopes. At the beginning of this century, however, the reality of atoms was still a matter for debate. With simple yet brilliant experiments performed between 1907 and 1913, Jean Perrin resolved the issue by studying small particles suspended in solution. Using an ordinary light microscope he studied the behaviour of the smallest objects he could see – spherical colloidal particles of resin just 1 μm across, suspended in water. (A colloidal system contains small particles of one type of material in a continuous matrix of another type. They can be solid in liquid, like paint; liquid in liquid, like milk; and even gas in liquid, such as an aerosol.)

First, Perrin verified that the concentration of colloidal particles in dilute suspension varied exponentially with height when allowed to reach dynamic equilibrium in the Earth's gravitational field. Then he observed the random diffusion of individual particles and showed that their Brownian motions are just large-scale manifestations of the thermal motions of atoms. These experiments verified that the particles obey the equipartition of energy, each particle having an average kinetic energy of $^3/_2 k_B T$, where k_B is Boltzmann's constant and T is temperature. Perrin won the 1926 Nobel Prize for Physics for this work.

Today, Perrin's view of colloids as atoms lives again. Whereas Perrin studied dilute suspensions – the analogue of an ideal gas of atoms – the interest today is focused on concentrated systems, in which the particles can occupy 50% or more of the total volume of the suspension. Here we find that colloidal systems show analogues of all the states of atomic systems: gas, liquid, crystal, alloy and glass (figure 1).

The renewed interest in colloid physics, which comprises the phase behaviour, structure and dynamics of colloidal systems, is due to a combination of factors. A range of "model" systems, in which the particles have known shapes, sizes and interactions, has been developed, and techniques such as optical microscopy and the scattering of X-rays, neutrons and light are used to study them. The continued development of statistical mechanics in general, and advances in theories of simple atomic liquids in particular, have created powerful theoretical concepts and tools. Fast computers are used to simulate many-body physics. Indeed, many recent advances in colloid physics are built on the fruitful interaction of experiment, theory and simulation. Furthermore, many industrial products, such as paints, glues, polishes, lubricants, foods and pharmaceuticals, are colloids. Many biological materials are also colloidal in nature. Both industrial and biological colloids are generally complex, many-component systems. However,

1 Suspensions of sub-micron perspex spheres exhibit a range behaviour – fluid-like, crystalline and glassy – as the concentration by volume, ϕ, is increased (from left to right). The suspension is almost transparent because the spheres and the liquid in which they are suspended have nearly the same refractive indices. The suspensions have been shaken thoroughly and left undisturbed for a day. The most dilute sample, $\phi = 0.48$, remains in a colloidal fluid state. In the next three samples, $0.50 < \phi < 0.55$, small crystallites of particles have nucleated spontaneously, grown and settled under gravity to leave well-defined boundaries between coexisting colloidal fluid and colloidal crystal phases. Some of the crystallites have orientations that fulfil the Bragg condition for light of a particular wavelength and therefore appear coloured. The next two samples, $0.55 < \phi < 0.58$, are fully polycrystalline, whereas, in sample 7, crystallization is nucleated mainly at the surfaces of the sample cell. In the two most concentrated samples, $0.58 < \phi < 0.61$, the particles are packed so tightly that they are unable to move far enough to form nuclei for crystallization. These are metastable colloid glasses.



2 (*a*) Image, obtained by phase contrast microscopy, of a plane through colloidal crystallites similar to those shown in figure 1. (*b*) A computer reconstruction of a three-dimensional image of both colloidal crystal and colloidal glass, obtained by scanning laser microscopy (from A van Blaaderen and P Wiltzius 1995 *Science* **270** 1177). (*c*) A scanning electron microscope image of a dried AB_2 colloidal crystal (from P Bartlett *et al.* 1992 *Phys. Rev. Lett.* **68** 3801).

industry is realizing that the fundamental physics of model colloids provides a basis on which to build an understanding of more complex systems.

Why study colloidal systems if they simply mimic the behaviour of atoms? One answer is that they have special properties that allow experiments that would be difficult, if not impossible, with atomic systems. For example, while atomic glasses can only be formed by cooling a liquid extremely rapidly, colloidal glasses are much more controllable and thus easier to study. Colloidal solids are mechanically weak – for example, when a sample containing crystals is shaken, the crystals are destroyed by shearing forces and the resulting metastable fluid takes minutes or even hours to recrystallize. Colloids can thus be used for detailed studies of fundamental processes of broad relevance, such as crystallization and glass formation.

Furthermore, techniques such as phase-contrast and confocal scanning laser microscopy have made it possible to resolve individual colloidal particles deep in the bulk of samples and so to construct three-dimensional images of colloidal structures (figures 2*a* and 2*b*). Finally, the interactions between colloidal particles can be adjusted or "tailored". This lets us investigate experimentally how the behaviour of an assembly of particles depends on their interactions. With atoms, one is simply stuck with the interaction dictated by their electronic structure.

Colloidal crystals

Conceptually, the simplest interaction between particles is that between hard spheres, which feel an infinite repulsive force when they touch but no force otherwise. Assemblies of hard spheres are vital models in both the theory and the computer simulation of fluids and solids. Of course hard-sphere atoms do not exist, but good approximations to hard-sphere colloids do. Solid cores of amorphous polymethylmethacrylate (PMMA or perspex) with thin layers of a flexible polymer grafted onto their surfaces come close to being hard spheres. When two particles touch, their polymer coatings are compressed and a repulsive elastic force acts between them. With thin coatings, this force increases rapidly with a small decrease in interparticle separation, thus approximating the infinite hard-sphere repulsion. Moreover, in these experiments the particles have a refractive index that is similar to that of the suspension liquid, making van der Waals attractions negligible.

An atomic material such as argon has an interatomic potential of the form shown in figure 3*a* and can exist as a single state – a gas, liquid or crystal – or coexist in two or three of these phases (figure 3*b*). The critical temperature, T_C, is approximately the temperature at which the thermal energy of the atoms is equal to the energy associated with the minimum in the interatomic potential, $\varepsilon \sim k_B T_C$. Liquid is only found between the critical temperature and the triple-point temperature, T_t, the temperature at which all three phases coexist. Above the critical temperature there is no distinction between gas and liquid – the material is fluid.

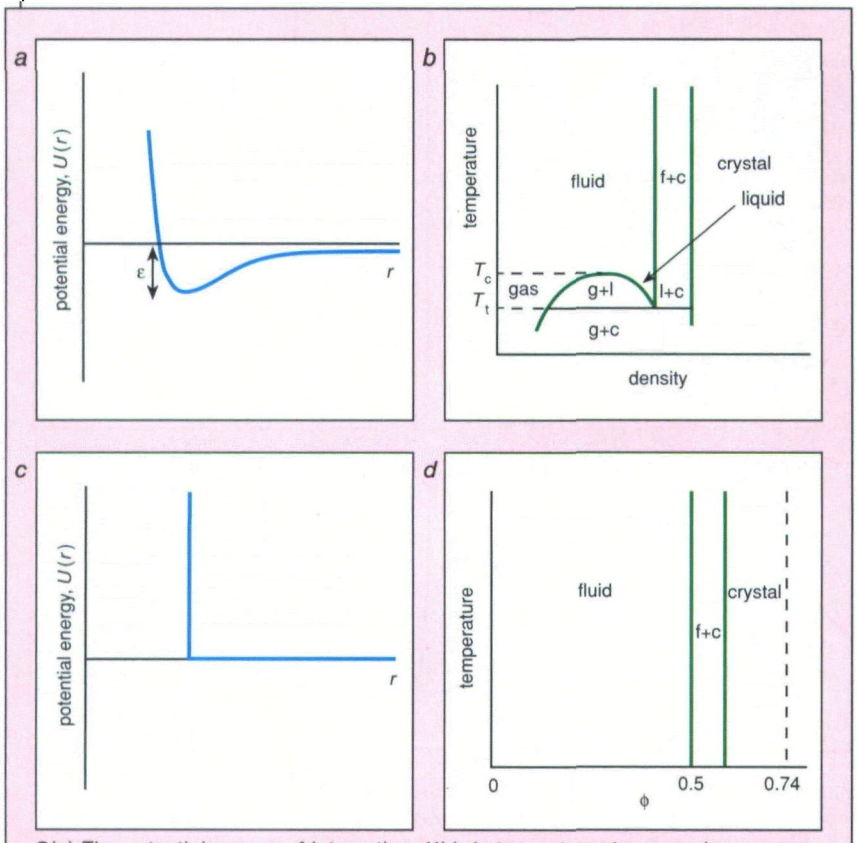
The absence of interparticle attraction in a system of hard spheres implies that the critical temperature is zero. The phase behaviour of hard spheres is therefore expected to be similar to that of a simple atomic substance above its critical temperature. The low-density fluid should freeze to a high-density crystalline solid without passing through a liquid state (figure 3*d*).

At first sight the crystallization of hard spheres seems to counter the intuition that attractive forces are needed to form crystals. In fact, some 30 years ago there was vigorous debate, stimulated by the computer simulations of Berni Alder and Tom Wainwright at the Lawrence Livermore National Laboratory, California, as to whether hard spheres should exhibit a freezing transition. Now the matter has been resolved by further simulations that located the transition precisely, by "density functional theory" and by the observation of colloidal crystals in suspensions of "microscopic billiard balls" (figure 1). It turns out that the crystallization transition of hard spheres is driven by entropy. Paradoxically, the particles of a metastable fluid have less freedom to move than in a crystal of the same density, so the entropy of the system is greater in the crystal than in the metastable fluid (*Physics World* February 1993 p24).

Colloidal crystals can also contain particles of different sizes. For example, the binary colloidal alloy, AB_2, can be grown from a suspension containing two species of hard-sphere particles, one of which is 1.72 times the size of the other (figure 2*c*). This complex structure forms spontaneously from a disordered mixture of particles moving in Brownian motion. Entropy is again the driving force. AB_2 is an efficient packing of spheres at this size ratio – particles in this structure have considerable freedom of local movement, relative to the disordered state, when they are packed at lower than the maximum density. Remarkably, colloidal alloys of this kind were first discovered in naturally occurring gem opals by John Sanders of the CSIRO laboratory in Melbourne, Australia, in 1978. Common opals are dried colloidal crystals of equal-sized silica spheres. The coloration, which gives them gem

quality, arises from the Bragg reflection of light by the lattice planes. Alloys of particles of different materials – for example, plastic and semiconductor or metal – may one day be engineered to have interesting and useful optical, dielectric or magnetic properties.

Colloidal crystals were first observed in the laboratory in aqueous suspensions of charged spherical viruses by Aaron Klug and his colleagues at Birkbeck College, London, in 1959. Much work has been done subsequently with model charged spheres of polystyrene where, amazingly, crystallization can occur when less than 0.1% of the volume is occupied by the spheres. This results from the fact that the screened Coulombic repulsion between the charged particles can have a range of 10 or more particle diameters. To borrow a concept from solid-state physics, we can say that the basic unit in such a suspension is an "effective" hard sphere or "quasiparticle" consisting of the charged particle and its "atmosphere" of small ions (see p35). Crystallization occurs at a quasiparticle concentration of about 50%, which is similar to the crystallization concentration of hard spheres (figure 3*d*).



3(*a*) The potential energy of interaction, $U(r)$, between two atoms, such as argon, separated by a distance r. (*b*) Temperature–density projection of the corresponding phase diagram. (*c*) Hard-sphere potential – the energy of the interaction between two spheres is infinite when the particles touch, and zero otherwise. (*d*) Hard-sphere phase diagram, showing fluid at low concentrations and crystal at high concentrations. There is no liquid state.

Tenuous liquids

The liquid state is mysterious and subtle – it flows like a gas but occupies a particular volume and does not expand to fill a container. If liquids were not common in everyday life, it is quite possible that theory would not yet have predicted their existence. The liquid state of a simple atomic material such as argon is found only in a small region of the temperature–density diagram (figure 3*b*). While there have been great advances in our understanding of the properties of liquids in the last 30 years, much less attention has been paid to the question of what conditions are necessary for the liquid state to occur in the first place.

It is clear that for a liquid to be found there must be an attraction between the particles to provide the cohesive energy. Until quite recently it was widely believed that any kind of attraction – added to a shorter-ranged repulsion – would lead to the formation of a stable liquid at some density and temperature. Calculations on colloid–polymer mixtures (see below) by Alice Gast and colleagues at Princeton University in the US showed that this is not true. In fact, for a stable liquid phase to form the interparticle attraction must have a sufficiently large spatial range.

These and more recent calculations show that when the range of the interparticle attraction is reduced, the gap between the critical and the triple-point temperatures becomes smaller (figure 4*d*). When the range of the attraction is about one-third of the range of the repulsion, the critical and the triple-point temperatures coincide and the liquid state is squeezed out of existence. If the range of the attraction is further reduced, only fluid and crystal phases are found (figure 4*c*). Daan Frenkel of the FOM Institute for Atomic and Molecular Physics, Amsterdam, and colleagues have recently predicted that, for very short-range attractions, although no liquid is found, a novel crystal–crystal phase transition should occur at high densities.

These predictions can be tested experimentally by using a suspension of hard-sphere colloids to which polymer molecules that cannot penetrate their surfaces are added. The polymers are excluded, or "depleted", from the region between the surfaces of two particles when they are closer than the diameter of a polymer molecule (figure 4*a*). The resulting "depletion force" pushes the particles together (figure 4*b*). The range and strength of the "depletion potential" can be adjusted by changing the size (molecular weight) and concentration of the polymer respectively.

At the University of Edinburgh we have found that when the added polymer is small relative to the hard-sphere colloid, only colloidal fluid and crystal states are observed (figure 5*a*). However, when the polymer is large enough, colloidal liquid is observed together with triple and critical points (figure 5*b*). Samples containing all three phases were only observed when the size of the polymer molecules, and thus the range of the depletion attraction, was greater than about 25% of the colloidal particles' radius. This confirms that a sufficiently long-range attraction is necessary for the liquid state to exist. Similar experiments by Jerome Bibette and co-workers at the Centre de Recherche Paul Pascal, Pessac, France, in 1993, have reached the same conclusion.

The close link between the existence of a liquid state and the relative range of the interparticle attraction to the hard-core repulsion has been confirmed by a number of recent calculations and simulations. Theoretical work by Marc Baus and colleagues at Université Libre de Bruxelles in 1994 illustrates the fragile presence of liquids. A hard-sphere potential followed by an inverse power-law attraction, $V(r) \propto -r^{-n}$, gives no liquid state for values of n greater than 7–8. In nature, $n = 6$, corresponding to the

van der Waals attraction, which just allows the ubiquitous but tenuous existence of liquids in the world of atomic materials.

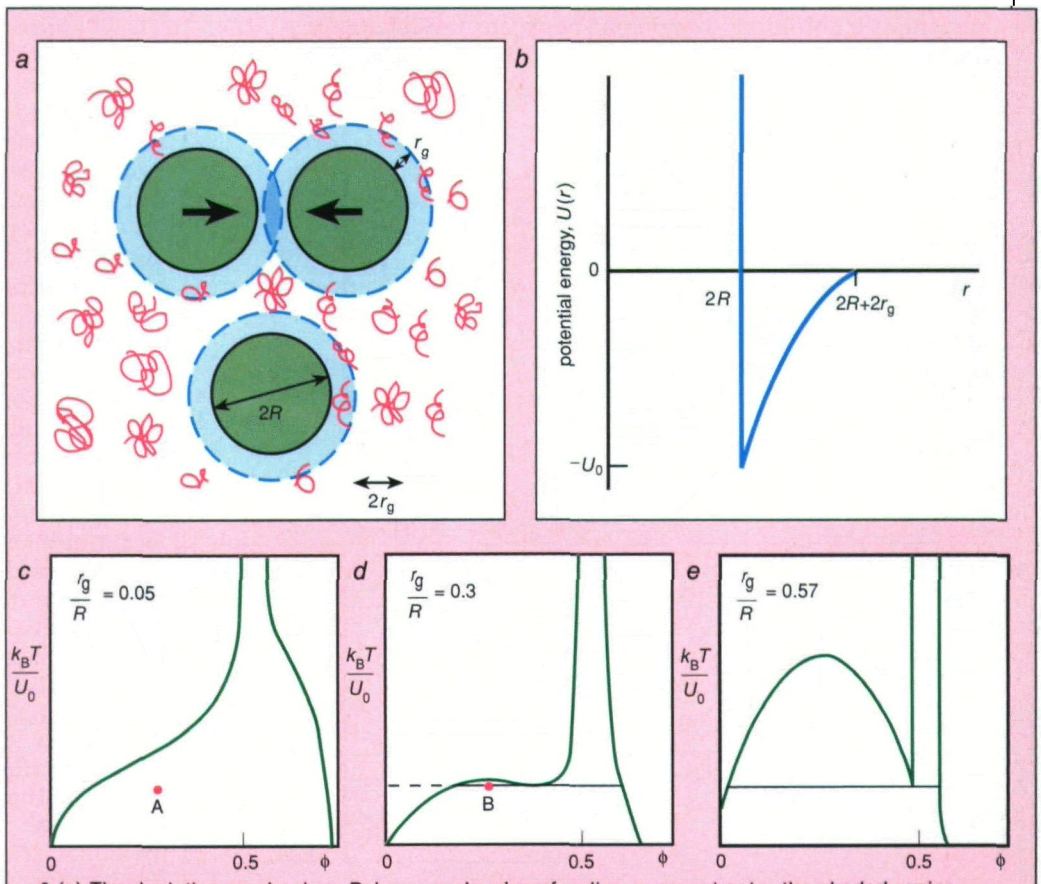
Metastable glasses

Colloidal suspensions are slow to reach equilibrium from a metastable state. The time taken for particles to reach equilibrium is set by the relaxation time, τ_R, the time taken by a particle to move a distance equal to its radius through Brownian motion. The relaxation time depends on the radius of the particle cubed, so a suspension of 1 μm particles is some 10^{12} times slower to relax than a liquid of 1 Å atoms. Thus, while the nucleation of crystals in an atomic liquid may take 10^{-9} s, the same process in a colloidal suspension can take minutes or hours. This characteristic, together with the possibility of direct visual observation using optical microscopy, have combined to make the study of the phase transition kinetics and metastability of colloidal systems active areas of research.

Arguably the simplest metastable state found in colloids is "hard-sphere glass". For a volume fraction, ϕ, greater than 0.545, a collection of hard spheres is crystalline at equilibrium. This is true all the way up to close-packing at $\phi = 0.740$ (figure 3*d*). However, in disordered suspensions of hard-sphere colloids above $\phi_g \approx 0.58$ the particles become so tightly trapped, or "caged", by their neighbours that they are unable to move far enough to nucleate crystallization. Instead, long-lived amorphous states called colloidal "glasses" are obtained. The precise way in which the diffusive dynamics of the particles slow down in metastable colloidal fluids as the concentration is increased towards ϕ_g has been studied in detail since 1990 using dynamic light scattering by Bill van Megen and Sylvia Underwood at the Royal Melbourne Institute of Technology in Australia.

In this technique, laser light is directed into the suspension. The scattered light forms a random diffraction pattern of bright and dark speckles, which fluctuates as the particles move in Brownian motion. The rate of fluctuation reflects the rate of particle diffusion. By processing the randomly fluctuating intensity of a single speckle with a computer, the dynamic scattering function $f(\tau)$ is constructed (figure 6).

Roughly speaking the characteristic time taken for this function to decay can be identified with the relaxation time, τ_R. A fast decay indicates a short relaxation and a slow decay indicates a long relaxation. The experiments show that the relaxation time lengthens dramatically when the volume fraction, ϕ, is increased. At high concentrations, $\phi = 0.587$, the dynamic scattering function does not decay completely. This implies that the particles are permanently trapped in the cages formed by their neighbours – this is a colloidal glass. The small-amplitude initial decay of $f(\tau)$ at $\phi = 0.587$ reflects the fact that, even in the glass, the particles have some freedom for local motions inside their cages. This indication of the glass transition from particle dynamics on the microscopic scale nicely parallels macroscopic observations of the samples, since, above $\phi = 0.587$, crystallization is suppressed. Data on colloidal systems have provided some of the most direct tests of modern mode-coupling theories of the glass transition, pioneered in the mid-1980s by Wolfgang Götze at the Technical University of Munich and co-workers (*Physics World* December 1991 p32).



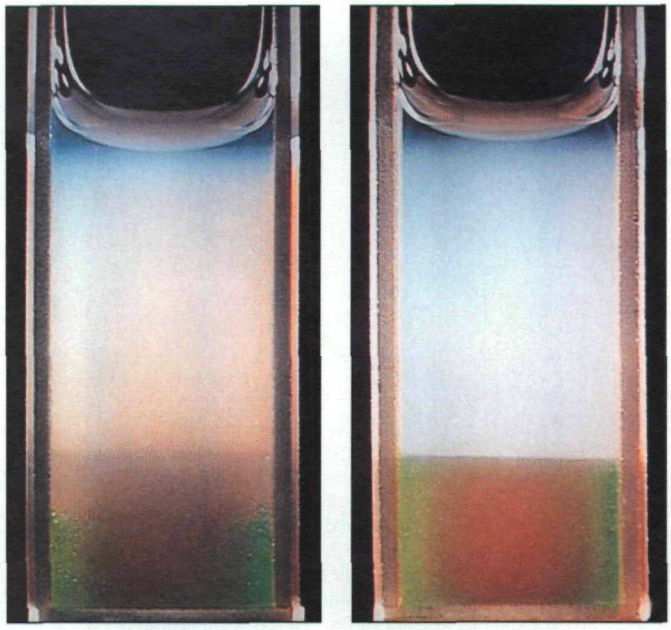
4 (*a*) The depletion mechanism. Polymer molecules of radius r_g cannot enter the shaded region between the closely spaced particle of radius R. The unbalanced osmotic pressure exerted on the particle surfaces by the polymers results in an attractive force. (*b*) The potential energy associated with the depletion force. (*c*)–(*e*) Predicted phase diagrams for different ratios of polymer radius of gyration, r_g, to particle radius, R. At large size ratio (e) the diagram resembles that of an atomic material such as that shown in **3***b*. As r_g/R gets smaller, the critical and triple-point temperatures get closer (*d*), and for small size ratios (*c*) the liquid region ceases to exist.

Aggregation and gelation

Attractive forces between colloidal particles – van der Waals forces, for example – tend to be short ranged compared with the size of the particles. (The depletion attraction caused by large polymers, discussed above, is an exception to this rule.) When a short-ranged potential is strong enough, diffusing colloidal particles stick to one another, forming clusters, in a process known as non-equilibrium aggregation. In fact, these clusters were some of the first examples of fractal structures studied experimentally by David Weitz and colleagues at Exxon Research and Engineering in New Jersey.

However, the formation of many branched clusters in an

aggregating colloidal suspension cannot continue forever. The average density of a cluster gets smaller as the cluster grows. Eventually, whatever the initial concentration of the suspension, the clusters will touch to form a space-spanning structure or gel. The study of this process of "kinetic gelation", using light scattering and computer simulation, has produced a somewhat surprising result in a number of laboratories – including the authors' and those of Marzio Giglio at the University of Milan, Italy, and Weitz at Exxon. Instead of showing a random diffraction pattern, the scattered laser light forms a speckled ring on the screen. In time, the ring brightens and collapses but eventually becomes "frozen" at gelation. This result suggests that the aggregating system is spontaneously developing a characteristic lengthscale, which becomes "stuck" at the gel point. While in some cases this length can be associated with the typical size of a cluster, its detailed origins are still a matter for debate.



5 (*a*) Colloid–polymer mixture showing gas–crystal coexistence (point A of **4***c*). (*b*) Colloid–polymer mixture showing three-phase coexistence of gas, liquid and crystal (point B of **4***d*).

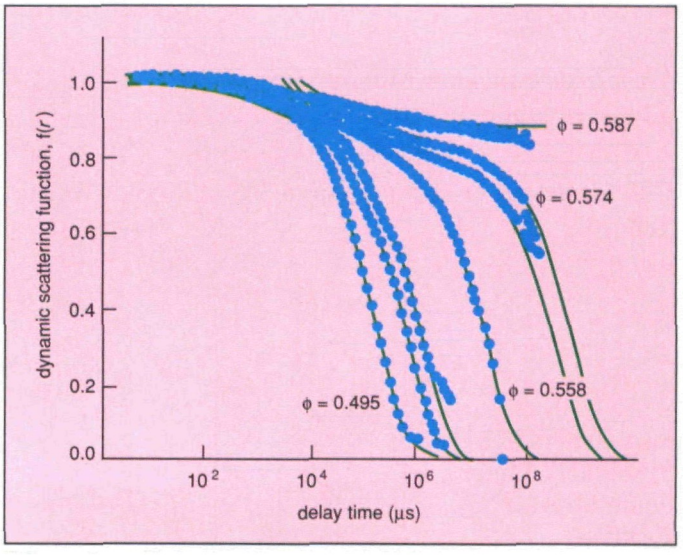
The equilibrium state of a collection of particles with a strong, short-range attraction is fluid–crystal coexistence (figure 4*c*). The "fractal gel" must therefore be a non-equilibrium state. Over enough time, thermal energy should break up the gel and crystals should nucleate. In many previous studies of colloidal aggregation the strength of the interparticle attraction has been many tens of k_BT, so such thermal rearrangements were highly unlikely. However, by carefully tuning the strength of the narrow depletion attraction in colloid–polymer mixtures, we have now studied the initial formation of a non-equilibrium gel state – "transient gelation" – which subsequently falls apart due to thermal rearrangement.

This possibility was first suggested from computer simulations by Kurt Binder at the University of Mainz, Germany, and colleagues. Recent experiments suggest that such non-equilibrium aggregation is "switched on" by a metastable liquid–gas phase boundary hidden inside the equilibrium fluid–crystal coexistence region. Further study of similar problems is likely to provide important insights into the nature of metastability, the understanding of which has vital industrial relevance – many products are sold in metastable states, the lifetime of which translates as "shelf life".

Gravity

So far, we have ignored the effects of gravity. Unless the density of colloidal particles exactly matches that of the liquid they are suspended in, the particles will sediment, or rise, in the Earth's gravitational field. The importance of gravity can be estimated from the ratio of the time a particle takes to diffuse a distance equal to its radius, R, to the time it takes to sediment the same distance. This ratio is given by the Peclet number, Pe. For spherical particles it is straightforward to show that $Pe = m_B gR/k_BT$, where m_B is the buoyant mass of the particles. Hence, Pe is essentially the ratio of gravitational potential energy to thermal energy. In the experiments discussed so far, $Pe << 1$, so the particles have plenty of time to explore configuration space by Brownian motion before the effect of gravity is felt. Thus, in the experiments of figure 1, colloidal crystallites nucleate under more or less equilibrium conditions. Because the crytallites are then denser than the colloidal fluid, they settle quickly enough to give the observed macroscopic phase separation. The size of the crystals is then limited by sedimentation since particles attempting to join them from the colloidal fluid are "brushed off" by the shear flow. Gravity can, of course, be eliminated by going into space. In the first of a planned series of colloid experiments in the space shuttle, Paul Chaikin and William Russel of Princeton University have recently repeated the experiments of figure 1. In contrast to the ground-based experiments, they found that the crystallites in the coexistence region grew large and dendritic, quite unlike the small, compact crystals in figure 1.

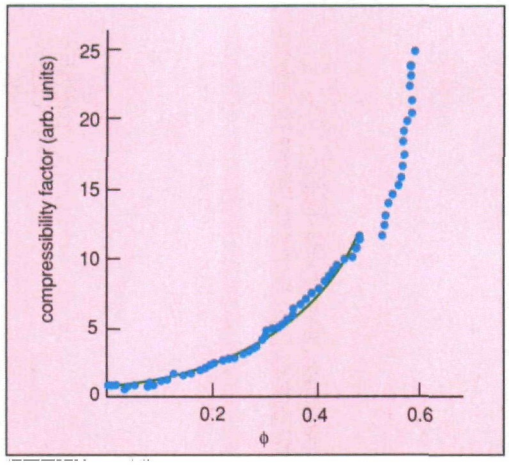
If a colloidal suspension is left undisturbed in a gravitational field for long enough it will eventually reach a stable, dynamic "sedimentation equilibrium". Here the concentration increases from top to bottom, and the downward flux of particles due to sedimentation is exactly balanced by the upward diffusion flux induced by the concentration gradient. From a macroscopic point of view, the associated gradient of the osmotic pressure supports the particles. One interpretation of Perrin's original experiment is that, in showing that a dilute colloidal suspension in sedimentation equilibrium has a Boltzmann density profile, he verified van't Hoff's law – the colloid



6 Dynamic scattering functions for colloidal glasses, showing dramatic lengthening of the relaxation time as the concentration of the glass transition is approached. Note that the *x*-axis is logarithmic. (From W van Megen and S M Underwood 1993 *Phys. Rev. Lett.* **70** 2766)

equivalent of the ideal gas equation.

Vittorio Degiorgio and colleagues at the University of Pavia in Italy have recently reported a beautiful extension of Perrin's experiment in which a concentrated suspension of hard-sphere colloids was allowed to come to sedimentation equilibrium. They measured the density profile from the intensity of depolarized light scattering and calculated the equation of state (figure 7). The beauty of this experiment is that the whole equation of state is obtained with a single measurement. This same idea has been exploited by Jean-Pierre Hansen at the Ecole Normale Supérieure at Lyon, and others, to obtain the equation of state of a system in a single computer simulation.



7 Equation of state of hard-sphere colloids deduced from the concentration profile measured for a suspension in sedimentation equilibrium. The compressibility factor, $\Pi/(Nk_BT)$, where Π is the osmotic pressure and N is the number of particles per unit volume, plotted against ϕ. A dilute colloidal fluid exists at the top of the sample cell, $\phi \rightarrow 0$, becoming crystalline at the bottom $\phi > 0.55$. The break in the data at $0.50 < \phi < 0.55$ indicates the fluid–crystal freezing transition. The solid line is the theoretical prediction for a fluid of hard spheres. (From R Piazza *et al.* 1993 *Phys. Rev. Lett.* **71** 4267)

Looking ahead

We have emphasized some recent advances that rely on our ability to tailor the interparticle potential to reveal new phase behaviour and metastable states. However, much other interesting physics and vitally important applications are contained in areas such as "dynamics" – a broad term encompassing the study of Brownian motion as well as behaviour under flow and deformation. Equally, we have concentrated on spherical particles, ignoring the recent increasing effort on rod-like particles that show liquid crystalline phases. Colloid physics is a vigorous and growing activity. Further fundamental discoveries can be expected, in parallel with increased "engineering" of colloidal systems to provide useful materials.

Further reading

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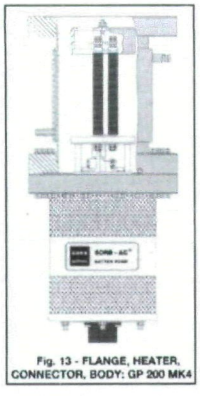
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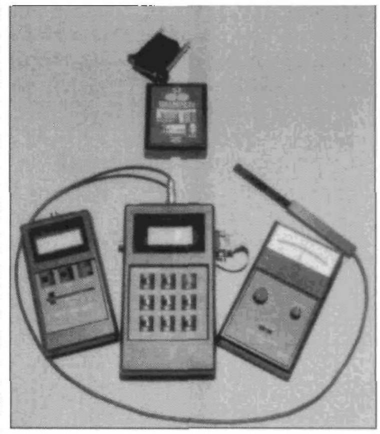

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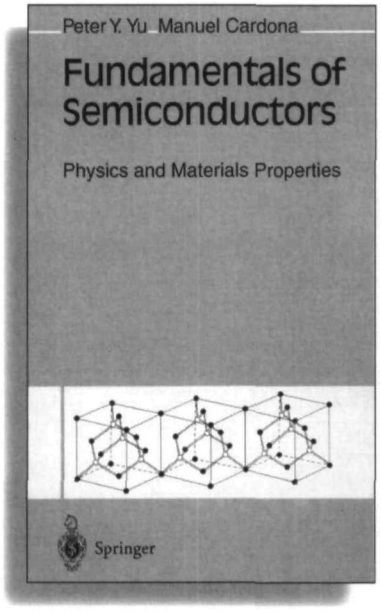

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