

# Chapter 21

## High-Power Ultrasound in Surface Cleaning and Decontamination

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High-power ultrasound is being widely utilized for decontamination in different industrial applications. The same technology is also being investigated as an effective tool for cleaning of components in the decontamination of produce. An understanding of the basic technology and how it works in cleaning various industrial parts should help in applying it on a large scale in the food industry. The technology has evolved throughout the past four decades. Different frequencies were developed and are now industrially available. The frequency range is from 20 kHz to 1 MHz. Current sound technology provides a uniform ultrasonic activity throughout the cleaning vessel, which was a major disadvantage in the earlier technology. The two main driving forces that affect cleaning of surfaces are cavitation and acoustic streaming. Both are generated as a result of the direct interaction of high-frequency sound waves with fluids. The intensity of each force varies with the frequency used. In addition to cleaning of industrial components, other known applications of high-power ultrasound are in the areas of spray drying, emulsification, degassing, flavor extraction, homogenization, meat tenderization, and removal of bio-contaminants. Very high-frequency ultrasound with moderate to very low power is being used in sonar, therapeutic, and medical diagnostics.

### 1 High-Power Ultrasound

A minimum level of ultrasound energy is needed to generate cavitation in a liquid. The threshold was found to be about 0.3 and 0.5 W/cm<sup>2</sup> at the radiating surface when the frequencies of the ultrasound waves are 20 and 40 kHz, respectively.

Currently available high-power ultrasound frequency range is from 20 kHz to 1 MHz. A frequency that is good for one application may be not good for another. The basic reason for this is that every application is unique in its nature with respect to material of construction, contaminants, and required cleanliness level. For

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example, cleaning of very thin fragile wafers requires higher frequencies, while cleaning of automotive components requires lower frequency.

At the low end of 20–60 kHz, cavitation implosion energy is the main scrubbing force, while at the high end of 1 MHz, micro-streaming of the fluid molecules is the main force in fine cleaning. It is important to note that both types of forces exist at every frequency. Cavitation implosion energy is good for the removal of heavy contaminants, while micro-streaming is good for the removal of nano-size particulates. There is a linear relationship between the applied frequency and the generation of cavitation and micro-streaming in fluids.

## 2 Ultrasonic Cleaning Mechanism

### 2.1 Cavitation and Micro-streaming

A unique phenomenon takes place when high-energy ultrasonic waves [20 kHz to about 500 kHz (at about 0.3–1 W/cm<sup>2</sup>)] travel in a liquid or in a solution. The waves interact with the liquid medium to generate a highly dynamic agitated solution. In the process, high-intensity ultrasonic waves create micro-vapor/vacuum bubbles in the liquid medium, which grow to a maximum size proportional to the applied ultrasonic frequency and then implode, releasing their energies. This phenomenon is known as cavitation implosion. The higher the frequency, the smaller the cavity size, with lower implosion energy. The high-intensity ultrasonics can grow cavities to a maximum in the course of a single cycle. At 20 kHz, the bubble size is roughly 170 μm in diameter (Fig. 21.1). At a higher frequency of 68 kHz, the total time from nucleation to implosion is estimated to be about one-third of that at 25 kHz. The vacuum bubble size becomes smaller at higher frequencies as a function of the wavelength. For example, at 132 kHz the bubble size is estimated to be about half of that generated at 68 kHz, and the size will be even much smaller at 200 kHz.

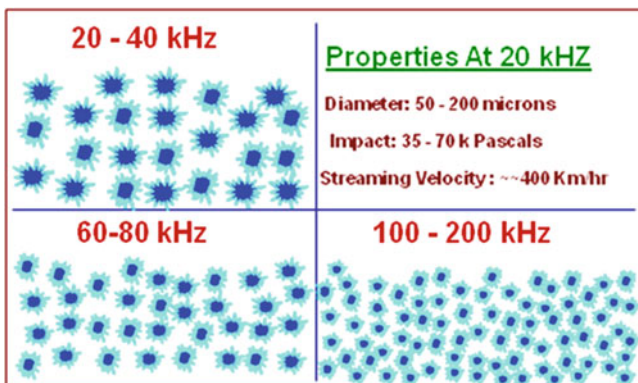


Fig. 21.1 Ultrasound frequency and cavitation size and population

Meanwhile, at higher frequencies, the minimum amount of energy required to produce ultrasonic cavities is higher and must be above the cavitation threshold. In other words, the ultrasonic waves must have enough pressure amplitude to overcome the natural molecular bonding forces and the natural elasticity of the liquid medium in order to grow the cavities. For water, at ambient temperature, the minimum amount of energy needed to be above the threshold was found to be about 0.3 and 0.5 W/cm<sup>2</sup> per tank radiating surface for 20 and 40 kHz, respectively. The energy released from implosions is expressed in new forces, namely shock wave, micro-streaming, shear forces, and miniature eddy currents.

## 2.2 Ultrasonic Generation

The transducers most commonly used for generating ultrasonic vibrations are piezoelectric, magnetostrictive, electromagnetic, pneumatic, and other mechanical devices. The piezoelectric transducer (PZT) assembly (Fig. 21.2) is the most widely used configuration in cleaning and plastic welding applications. Lead zirconate titanate is used interchangeably for PZT (but is not discussed in this chapter). The PZT assembly can generate a wide range of frequencies from about 20 kHz to the megasonic range.

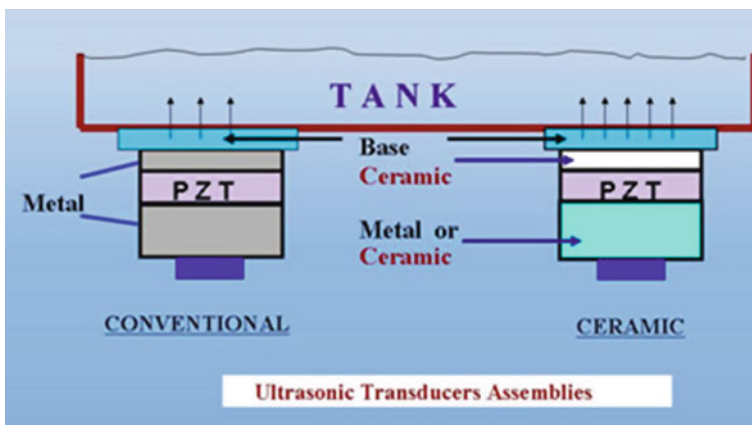


Fig. 21.2 Ultrasonic PZT transducer assembly bonded to the bottom of a tank

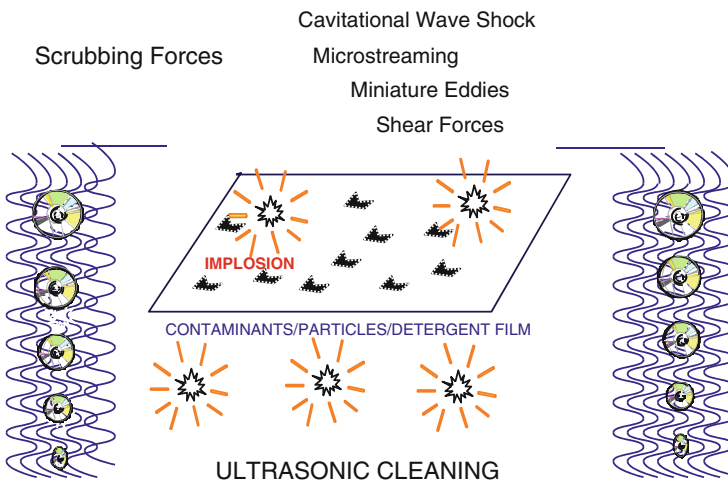
Typical piezoelectric (PZT) transducers are normally mounted on the bottom and/or sides of the cleaning tanks. The transducers can be mounted onto various designs and sizes that are sealed in stainless steel containers or immersed in the cleaning fluid (immersibles).

Another design for immersibles (manufactured by Martin Walter Co., a Crest Tech company) is known as the push-pull transducer. It is an immersible transducer. The push-pull is made of two PZT transducers mounted on the ends of a titanium rod with different lengths and diameters. The ultrasonic waves generated along the

rod axis propagate perpendicularly to the resonating surface. The waves interact with the fluid medium to generate cavitation implosions.

### 2.3 Cavitation Formation Mechanism

The ultrasonic cleaning illustration (Fig. 21.3) shows the generating cavitation through at least three steps: nucleation, growth and violent collapse, or implosion. The transient cavities (or vacuum bubbles or vapor voids), ranging from 50 to 150  $\mu\text{m}$  in diameter at 25 kHz, are produced during the sound waves' half cycles. During the rarefaction phase of the sound wave, the liquid molecules are extended outward against and beyond the liquid's natural physical elasticity/bonding/attraction forces, generating vacuum nuclei that continue to grow to a maximum. Then, violent collapse occurs during the compression phase of the wave. It is believed that the latter phase is augmented by the enthalpy of the medium and the degree of mobility of the molecules, as well as the hydrostatic pressure of the medium.



**Fig. 21.3** Micro-vacuum bubble formation and scrubbing forces released on implosion

Cavitations are generated in the order of microseconds. At 20 kHz frequency, it is estimated that the pressure is about 35–70 kPa and the transient localized temperatures are about 5,000°C, with the velocity of micro-streaming around 400 km/h (Table 21.1). Several factors have great influence on the cavitation intensity and abundance in a given medium. Among these factors are the ultrasonic wave form, its frequency, and the power amplitude. Other determining factors are the physical properties of the liquid medium including viscosity, surface tension, density, and vapor pressure; the medium temperature and the liquid flow, whether laminar or turbulent; and dissolved gases.

**Table 21.1** High-Power ultrasound in water

Frequency (kHz)	Cavitation size ( $\mu\text{m}$ ) and intensity	Microstreaming velocity (m/s)	Boundary layer ( $\mu\text{m}$ )
20 – 30	~ 125 High	~ 50	4.4
40	75 Medium	>100	2.32
68	42		
72, 80	Medium		
104			
132	23 Mild		
172			
192			1.6
400	Very mild		0.594
0.8 – 1 MHz	Extremely mild		

## 2.4 Frequency and Cavitation Abundance

At low frequencies of 20–30 kHz, a smaller number of cavitations with larger sizes and higher energy are generated. Much denser cavitations with moderate or lower energies are formed as frequency increases. Frequencies of 20–35 kHz are more appropriate for cleaning heavy and large size components, while a frequency of 60–80 kHz is recommended for cleaning delicate surfaces such as optics. Frequencies of 132 and 192 kHz are recommended for cleaning ultra-delicate materials such as thin films and tiny components. The latter two ranges are appropriate for use during rinsing steps with all applications. For example, at 60–80 kHz, the cavitation abundance is high enough and mild enough to remove detergent films and to remove submicron particles in the rinsing steps without inflicting damage on surfaces. The 35–45 kHz frequency range was found to be appropriate for a wide range of industrial heavy components such as automotive engines and steel molds.

Estimates of cavitation abundance at various ultrasonic frequencies have shown that cavitations are more abundant at higher frequencies. For example, about 60–70% more cavitation sites per unit volume of liquid are generated at 68 Hz

than at 40 kHz because the size of the micro-cavities decreases at higher frequencies (Awad, 1996). Therefore, one would expect that at the higher frequency, at a given energy level, the scrubbing intensity would be milder, particularly on soft and thin or delicate surfaces. In general, selecting the proper frequency for a particular application is critical and must be carefully investigated.

### 3 Precision Cleaning

Precision or critical cleaning of components or substrates is the complete removal of undesirable contaminants to a desired preset level, without introducing new contaminants in the process (Awad, 1996). The preset level is normally the minimum level at which no adverse effects take place in a subsequent operation. To achieve the desired cleanliness level, it is critical not to introduce new contaminant(s) into the cleaning process. For example, in an aqueous cleaning process, it is important to have high-quality rinse water and a minimum of two rinse steps. Otherwise, residual detergent and/or salts from the rinsing water will become new contaminants. Re-contamination of cleaned parts with out-gassed residues produced from packaging or storing materials is another source (Awad, 1996).

To meet production and quality demands, one must keep in mind that one essential element is choosing the appropriate cleaning chemistry/process. Moreover, rejected parts are the curse of the assembly line and improper cleaning methods are often to blame. Even beyond the factory floor, improper or inadequate cleaning of a component could directly affect warranty claims (Bhatt, 1998; Durkee, 1994).

#### 3.1 Ultrasonic Cavitations and Surface Cleaning

The energy released from an implosion in close vicinity to the surface collides with and fragments or disintegrates the contaminants, allowing the detergent or the cleaning solvent to displace it at a very fast rate. The implosion also produces dynamic pressure waves, which carry the fragments away from the surface. The implosion is also accompanied by high-speed micro-streaming currents of the liquid molecules. The cumulative effect of millions of continuous tiny implosions in a liquid medium provides the necessary mechanical energy to break physically bonded contaminants, speeds up the hydrolysis of chemically bonded contaminants, and enhances the solubilization of ionic contaminants. The chemical composition of the medium is an important factor in speeding the removal rate of various contaminants.

Cleaning with ultrasonics offers several advantages over other conventional methods. Ultrasonic waves generate and evenly distribute cavitation implosions in a liquid medium. The released energies reach and penetrate deep into crevices, blind holes, and areas that are inaccessible to other cleaning methods (Fuchs, 1997; O'Donoghue, 1984). The removal of contaminants is consistent and uniform, regardless of the complexity and the geometry of the substrates.

### 3.2 Power Requirements

The general ultrasonic power requirements for almost all cleaning applications, expressed in terms of electrical-input wattage to the transducers, is in the range of 7.15–9.15 W/cm<sup>2</sup> of transducer radiating surface.

## 4 Ultrasonic Cleaning Equipment

Ultrasonic aqueous batch cleaning equipment consists of at least four steps: ultrasonic wash tank, minimum of two ultrasonic separate (or reverse cascading) water rinse tanks, and heated recirculated clean air for drying. The last drying step is not included if the post-cleaning operation includes an aqueous bath, as in electro- or electroless plating. Ultrasonic transducers are bonded to the outside bottom surface (Fig. 21.2) or to the outside of the sidewalls or provided as immersibles inside the tanks. The latter is usually the preferred method when large size tanks are under consideration. Two types of immersibles (Fig. 21.4) are commercially available in various sizes and frequencies. The traditional sealed metal box contains a multi-transducer system and a cylindrical immersible, which is powered by two main transducers at both ends, known as push–pull immersibles (Weber and Walter, 1993).



Ultrasonic Tank



Ultrasonic Generators



Immiscible Transducers



**Fig. 21.4** Ultrasound units (courtesy of Crest Ultrasonics)

Prior to selecting equipment, it is imperative that an effective cleaning process first be developed, and then the number and size of the stations are subsequently determined based on the required yield, total process time, and space limitation.

The automation of ultrasonic cleaning systems is well established. Automation includes a computerized transport system able to run different processes for various parts simultaneously, along with data monitoring and acquisition. The advantages of automation are numerous, including consistency, achieving desired throughputs, and full control of process parameters (Awad, 2000).

Typical tank size ranges from 10 to 2,500 l, based on the size of the parts, production throughput, and required drying time. The entire machine can be enclosed to provide a clean room environment meeting class 10,000 down to class 100 clean room specifications. Process control and monitoring equipment consists of flow controls, chemical feed pumps, in-line particle count, TOC measurement, pH, turbidity, conductivity, refractive index, etc. The tanks are typically made of corrosion-resistant stainless steel or electro-polished stainless steel. Titanium nitride or a similar coating such as hard chrome or zirconium is used to extend the lifetime of the radiating surface in the tanks or the immersible transducers.

The number and the size of the stations are determined based on the required process time. A semi-aqueous cleaning system, for example, includes an extra station for solvent displacement, connected to a phase separation/recovery system. The entire machine can be enclosed to provide a clean room environment meeting class 10,000 down to class 100 clean room specifications.

## 5 Cleaning Chemistry

It is important to realize that the use of ultrasonics does not eliminate the need for proper cleaning chemicals and implementation and maintenance of the proper process parameters (Seelig, 1995).

Furthermore, the chemical composition of the cleaning medium (for aqueous cleaning or organic solvent cleaning) is a critical factor in achieving the complete removal of various contaminants without inflicting any damage to the components.

Cleaning with ultrasonics using only plain water is workable, but only for short time. The question, then, is how long ultrasonic cleaning is going to work before failing to clean. In fact, cleaning is more complex in nature than just extracting contaminants away from the surface. Soil loading and encapsulation/dispersion of contaminants are determining factors for both the effective lifetime of the cleaning medium and the cleaning results.

Reproducibility and consistency of the cleaning results are essential requirements for all successful cleaning processes. Cleaning chemistry, as part of the overall cleaning process, is a very crucial element in achieving such consistency (Kaneegsburg, 1999). Requirements for the selected chemistry are many. It must cavitate well with ultrasonics and be compatible with the components to be cleaned. Other properties such as wettability, stability, soil loading, oil separation, effectiveness, dispersion or encapsulation of solid residues, ability to rinse freely, and disposal are all important factors that must be addressed when deciding on the



appropriate chemistry. Clearly, an expert in the field is the best individual to make this decision.

Both aqueous and organic solvent cleaning methods have advantages and disadvantages. Aqueous cleaning is universal and achieves better cleaning results. Organic solvents are effective in removing organic contaminants, but short on removing inorganic salts. Drying and protection of steel components are valid concerns. However, the current available technologies offer effective ways to alleviate these concerns.

Power ultrasound enhances the effect of cleaning or decontaminating chemicals, in part due to the dislodging of general and bio-contaminants such as oils, protein, or bacterial clumps from surfaces, thus making them available to the chemicals (Mason, 1998).

The role of aqueous chemistry is to displace oil, to solubilize it or emulsify organic and bio-contaminants, to encapsulate particles, and to disperse and prevent redeposition of contaminants after cleaning. Special aqueous formulations assisted with ultrasonics are being used to decontaminate post-operation surgical instruments, dental and medical devices, and in food processing (Boucher, 1980). Decontamination of hatchery eggs was recently reported using a bactericide and ultrasound (Slapp, 1995). Some additives in cleaning chemicals are used to assist in the process of breaking chemical bonding, removal of oxides, preventing corrosion, or enhancing the physical properties of the surfactants or to enhance the surface finish. Post-cleaning, it is important to use deionized water (RO water) for rinsing off the aqueous chemical(s) to achieve spot-free surfaces. A minimum of two rinse steps is recommended.

### ***5.1 Selection of Cleaning Fluids***

Effective cleaning fluids are essentially selected on the basis of (1) the chemical and physical nature of the contaminants to be removed; (2) compatibility with the substrate material(s); (3) environmental considerations; and (4) required cleanliness specifications. Therefore, in precision or fine cleaning there is no one specific chemistry for all applications. Every case must be examined individually to determine the most effective and safe chemical.

## **6 Contaminants**

Three general classes of common contaminants are organic, inorganic, and particulate matter. Particles do not necessarily belong to a certain class and can be from any one class or a mixture thereof. Insoluble particulate contaminants can, for example, be divided into two groups: (1) water-wettable or hydrophilic particles, including metal particles, metal oxides, minerals, and inorganic dusts; and (2) nonwater-wettable or hydrophobic substances, including plastic particles, smoke and carbon particles, graphite dust, and organic chemical dusts. Substrate surfaces, too, can be divided into hydrophilic and hydrophobic groups.

Contaminants of any class can be water soluble or water insoluble. Organic contaminants in most cases will be hydrophobic in nature, such as oils, greases, waxes, polymers, paints, print, adhesives, or coatings.

Except in a very few cases, inorganic materials or salts are insoluble in solvents that are nonwater miscible. Water is the best universal solvent for organic or inorganic ionic materials. However, water-insoluble inorganics, such as polishing compounds made of oxides of aluminum, cerium, or zirconium, require a more elaborate cleaning process.

Organic contaminants can be classified into three general classes: long-chain, medium-chain, and short-chain molecules. The physical and chemical characteristics are related to their structure and geometry.

## 7 Mechanism of Cleaning

Two main steps take place in surface cleaning. The first step is contaminant removal and the second step is to keep those contaminants from re-adhering to the surface. The removal of various contaminants involves different mechanisms, based on the nature and/or the class of the contaminant.

Organic contaminants are removed by two main mechanisms. The first is by solubilization in an organic solvent. Degree of solubilization in various solvents is directly related to their molecular structure. The second mechanism is by displacement with a surfactant film followed by encapsulation and dispersion.

In aqueous cleaning, the detergent contains a single or mixture of surfactants. Surfactants are long-chain organic molecules with polar and non-polar sections in their chains. Surfactants can be ionic or nonionic in nature, based on the type of functional groups attached to or part of their chains. When diluted with water, surfactants form aggregates, called micelles, at a level above their critical micelle concentration (CMC). The micelles are composed of aggregates of hydrophilic moiety and hydrophobic portion of the surfactant molecules. They act as a solvent and encapsulate similar contaminants, thus preventing redeposition.

The mechanism of removal of organic contaminants by detergent involves wetting of the contaminant as well as the substrate. According to Young's equation, this will result in increasing the contact angle ( $\theta$ ) between the contaminant and the surface, thus decreasing the surface area wetted with the hydrophobe and reducing the scrubbing energy needed for removal:

$$\cos \theta = \frac{\gamma_{SB} - \lambda_{SO}}{\gamma_{OB}}$$

where  $\gamma_{SB}$  is substrate bath interfacial energy,  $\gamma_{OB}$  is soil bath interfacial energy, and  $\lambda_{SO}$  is substrate soil interfacial energy.

Ultrasonic cavitation plays an important role in initiating and completing the removal of hydrophobic contaminants (e.g., oils, soils). The shock wave (and the

micro-streaming currents) greatly speed up the breaking up of the hanging contaminants, thus enhancing displacement with the detergent film. The removed contaminants are then encapsulated in the micellic aggregates, thus preventing their redeposition. The net result is that ultrasonic cavitations accelerate the displacement of contaminants from the surface of the substrate and also facilitate their dispersion throughout the cleaning medium.

## ***7.1 Particle Removal***

Particles, in general, have irregular shapes. All the adhesion forces, i.e., van der Waals, electrical double layer, capillary, and electrostatic, are in theory directly proportional in magnitude to the size of the particle. One would expect that the energy of detachment would decrease with the size of particle. However, smaller particles are always more difficult to detach. This is mainly due to the lodging effect. Smaller particles tend to get trapped in the valleys of a rough surface.

## ***7.2 Particle Removal Mechanism***

The mechanism of particle removal involves shifting the free energy of detachment to be near or smaller than zero, according to Gibbs adsorption equation. Surfactants play a very important role in decreasing interfacial tension by adsorption at particle and substrate interface with the bath. The interfacial tension  $\gamma_{OB}$  and  $\gamma_{SB}$  will decrease and, accordingly, the force needed to detach the particles will decrease, when  $\Delta G$  is greater than zero or a positive value:

$$\Delta G = \gamma_{SB} + \gamma_{OB} - \gamma_{SO}$$

where  $\gamma_{SB}$  is substrate bath interfacial energy,  $\gamma_{OB}$  is soil bath interfacial energy,  $\gamma_{SO}$  is substrate soil interfacial energy.

The wettability of the surface plays an important role in achieving this step. The ultrasonic cavitation role is to provide the necessary energy for the detachment (i.e., the removal force). At high-frequency (60–70 kHz) ultrasonics, the detachment or the removal efficiency of very small size particles of 1  $\mu\text{m}$ , measured in deionized water, was found to be 95% versus 88% at 40 kHz. This is to be expected in light of the fact that cavitation size is smaller at higher frequencies and can reach deeper into the surface valleys. One would then anticipate that by using a combination of high-frequency ultrasonics at 65–68 kHz and the appropriate chemistry, the removal efficiency of various particles could be further optimized (Busnaina et al., 1994).

### 7.3 Prevention of Redeposition

Redeposition of contaminants is inhibited by another mechanism that forms a barrier between the removed contaminant and the cleaned surface. In solvent cleaning, the adsorbed solvent layers on the surface provide a film barrier. In aqueous cleaning, a good surfactant system is capable of encapsulating contaminants inside their micellar structure, as depicted in (Fig. 21.5). Thus, redeposition of the encapsulated contaminants (soils) onto an adsorbed surfactant film on the surface is prevented via steric hindrance for nonionic surfactants, while anionic surfactants prevent redeposition via an electrical repulsive barrier (Fig. 21.6).

Encapsulation can be permanent or transient, based on the nature of the used surfactants. Transient encapsulation is superior to emulsification, as it allows better

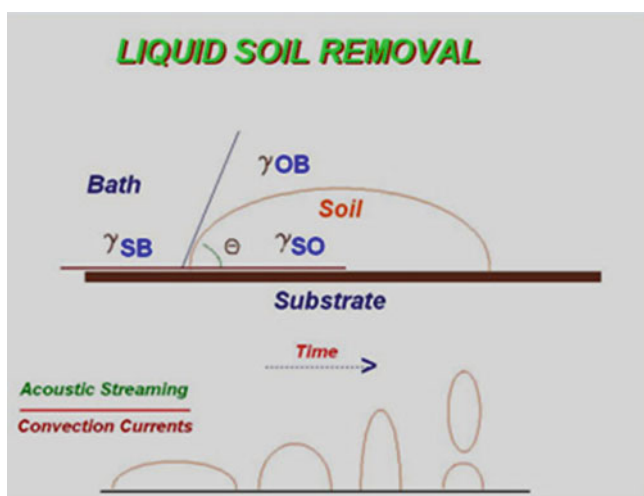


Fig. 21.5 Soil removal

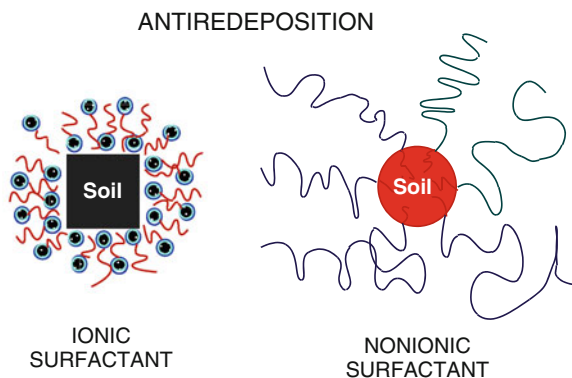


Fig. 21.6 Soil encapsulation

filtration and/or phase separation of contaminants. The potential of reversing the redeposition step by the application of sonic shock waves on loaded micelles results in partial re-adhesion. Therefore, allowing an increase in the soil load in a cleaning solution to reach saturation point, without good filtration, will result in a significant decrease in the detergent cleaning efficiency, at which point the cleaning action may cease. To ensure steady cleaning efficiency, the dispersed contaminants must be removed by means of continuous filtration or separation of contaminants, along with maintaining the recommended concentration of the cleaning chemical.

### 7.4 Cleaning Chemistry and Particles

A crucial element in small particle removal is prevention of redeposition. Cleaning chemistry plays this crucial role. The cleaning fluid should be capable of encapsulating the removed particles and thus preventing their redeposition on surfaces. Also, the cleaning fluid, whether aqueous or solvent, must displace the insoluble contaminants and form an insulating boundary film on the surface. The physical nature of the substrate and the degree of its surface finish are important factors in sub-micron particle removal (Awad, 1999; Mittal, 1995). For example, a silicon wafer surface is different from that of an aluminum disk, regarding their physical makeup, topography, and finish. Plastics are another challenge when dealing with submicron particles because of the inherent static electricity charges.

## 8 Conclusion

Cleaning with the assistance of ultrasonic cavitation has numerous advantages, most importantly a high level of decontamination and cleanliness and consistency in results. For the best cleaning results, selection of ultrasonic frequency and the cleaning chemistry (organic solvent or aqueous) is specific per application.

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