# Multifrequency Ultrasonic Actuators with Special Application to Ultrasonic Cleaning in Liquid and Supercritical CO<sub>2</sub>

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## **Abstract**

The collaboration between ECO<sub>2</sub> SA and MPI resulted in the realization of an industrial plant for the precision cleaning of mechanic, micro mechanic, electronic and microelectronic industry parts, based on the solvent-power of supercritical CO<sub>2</sub> and on the cleaning power of ultrasonic energy propagated in liquid CO<sub>2</sub>. The plant, already productive, performs the cleaning of 15 kg/h of precision writing balls, and it has been successfully tested for other industrial applications. Substitution of CFC, chlorinated solvents and detergents, absence of contaminated solutions and vapor emissions and improvement of the surface cleanness, are the most important results. The propagation of ultrasonic energy in the homogenous and pressurized medium like liquid CO<sub>2</sub> (T< 32°C; P> 60 bars) is realized using a novel ultrasonic structural, multifrequency actuator, able to initiate ringing and relaxing, multimode mechanical oscillations (harmonics and sub harmonics) in an autoclave with very thick walls, producing pulse-repetitive, phase, frequency and amplitude-modulated bulk-wave-excitation. Such ultrasonic driving is creating uniform and homogenous distribution of acoustical activity on a surface and inside of the vibrating system, while avoiding creation of stationary and standing waves structure, making that the complete vibrating system (autoclave) is fully agitated.

## **Introduction**

The liquid, supercritical  $CO_2$  precision cleaning project proposed by Swiss company ECO<sub>2</sub> SA was recognized and encouraged by the Swiss Federal Bureau for environmental protection (BUWAL) because of its potential to substitute chlorinated and fluorinated solvents (CFCs) with inert and non toxic carbon dioxide. The project was developed around a specific industrial application - cleaning of precision steel and tungsten carbide balls, for ballpoint pens (these balls, with diameters from 0.5 to 1 mm, are contaminated during production by lubricant oils, metal powders and very fine abrasive powders, and must be cleaned to a very high degree, verified by SEM, using 5000 to 10000 magnification). An industrial ultrasonic-CO<sub>2</sub> pilot unit has been built for this purpose, and is also applicable to other products. The plant is currently operating well and has the capacity to clean small solid balls at the rate of 15 kg/h. ECO<sub>2</sub> SA is a Swiss company, active in Supercritical Fluid Technology for cleaning, liquid processing and exploitation of fine chemicals. MPI, also a Swiss company, develops challenging projects in Ultrasonics.

In this new cleaning process, the oils are removed from the surface by the detergent action and solvent-power of the supercritical liquid  $CO_2$ , while the solid residues are dislodged by sonic and ultrasonic wave agitation. Conventional ultrasonic baths, using water or other conventional solvents, operate at or close to atmospheric pressure. In the case described here, the liquid ( $CO_2$ ) is homogeneous, pressurized to at least 60 bars and must be kept at a temperature of no more than 31°C. Under these conditions conventional high power ultrasonic systems are ineffective. In this project a novel multifrequency and multimode (sweeping frequency) ultrasonic source was developed for optimal propagation of ultrasound into the internal cleaning reactor space (Patent EP 1 060 798 A1; -see more about <u>Multifrequency</u> <u>Ultrasonic Structural Actuators</u> in the second part of this paper).

2. Cleaning System

A basket containing the small solid balls is placed in a pressurized autoclave that has very thick stainless steel walls (approx. 20 to 25 mm). Inside this pressurized autoclave (or ultrasonic cleaning reactor) the basket is rotated by an external motor at a programmable rate between 10 and 150 rpm. The ultrasonic energy is applied for a period of 4-10 minutes during the washing phase with liquid CO<sub>2</sub>. Cleaning residuals and remaining powders are transported out of the autoclave by a continuous current of liquid CO<sub>2</sub>, generated by a diaphragm pump with a flow rate of 30-100 Kg/h. At the end of the cleaning cycle the liquid and/or supercritical CO<sub>2</sub> is depressurized and vaporized. This dramatically reduces its solvent power for complex and heavy oils so that all cleaning residuals, such as oil-remains and powders, are deposited in the separator-unit. To complete the cycle, the vaporized CO<sub>2</sub> is filtered, condensed and reintroduced to the autoclave.

A complete washing cycle takes 40 min. The volume of the autoclave in question is 10 liters, which allows a batch of approx. 15 kg of small tungsten carbide or metal balls.

## 3. Experimental Results

Production and experimental batch testing has shown that this technology allows an improvement of the industrial cleaning quality standard (degree of cleanness of balls' surfaces), an increase in productivity, an increase in operators safety (elimination of solvent vapor emission), and it becomes environment friendly (there isn't soil contamination trough solvent-vapor diffusion), including other benefits such as: no-need for detergents ands solvents, water cannot be contaminated, there isn't waist of cleaning oils.

Figure 1 shows the CO<sub>2</sub> cleaning-process block-diagram, Figure 2 presents the block diagram of a multifrequency structural actuator, while figures 3 and 4

are photos of the industrial pilot unit developed. Figures are shown at the end of this paper.

The plant has been successfully tested for precision balls used in ball-pens, for surgical instruments, for medical implants (screws and prostheses), jewelry, for electronic and microelectronic parts and products, for car and aeronautics industry etc. The cleaning degree and quality of the surface processing was checked by means of electronic microscopy (see Figures 5, 6 and 7).

# 4. MULTIFREQUENCY ULTRASONIC STRUCTURAL ACTUATORS

The high power ultrasonic system used in this process generates multimode mechanical oscillations in the cleaning reactor, or autoclave over a wide frequency range. This is in contrast to conventional power ultrasonic systems, which operate at a single frequency. In addition the method of driving these transducers is optimized.

Every elastic mechanical system has many vibration modes, plus harmonics and sub harmonics, both in low and ultrasonic frequency domains. Many of vibrating modes could be acoustically and/or mechanically coupled, and others would stay relatively independent. In our multimode transducer we have the potential to synchronously excite many vibrating modes (including harmonics and sub harmonics), producing a uniform and homogenous repetition of high intensity vibrations.

In our system we insure that the oscillations are not random - rather they follow a consistent pulse-repetitive pattern, frequency and amplitude-modulated by the control system. This avoids the creation of stationary or standing waves (typically produced by traditional ultrasonic systems operating at a single frequency) that generate regions of high and low activity.

This technique (multimode excitation) is beneficial in many other applications, e.g. Liquid processing, fluid atomization, powders production, artificial aging of solids and liquids, accelerated stress relief, advanced ultrasonic cleaning, liquid metal treatment, surface coating, accelerated electrolysis, mixing and homogenizing of any fluid, waste water treatment, water sterilization, accelerated heat exchange...

A multifrequency ultrasonic structural actuator (see Fig. 2) consist of:

- A) Sweeping-frequency Ultrasonic Power Supply (including all regulations, controls and protections),
- B) High Power Ultrasonic Converter (see Patent EP 1 060 798 A1),
- C) Acoustical Wave-guide (metal bar, aluminum, titanium), which connects ultrasonic transducer with an acoustic load, oscillating body, resonator...
- D) Acoustical Load (mechanical resonating body, sonoreactor, radiating ultrasonic tool, sonotrode, test specimen, vibrating tube, vibrating sphere, a mold, solid or fluid media, autoclave...),

E) Sensors of acoustical activity fixed on/in/at an Acoustical Load (accelerometers, ultrasonic flux meters, cavitation detectors, laser vibrometer/s...), which are creating regulation-feedback between the Acoustical Load and Ultrasonic Power Supply.

A strong mechanical coupling of high power ultrasonic converter (B) to the test specimen or acoustical load (D) is realized using acoustic-wave guide metal bar (C). Ultrasonic converter (B) is electrically connected to the ultrasonic power supply (A), or ultrasonic multimode generator. Acoustic activity sensors (E) are realizing feedback (for the purpose of automatic process control) between Acoustical Load (D) and Ultrasonic Power Supply (A).

## **Operating Principles**

Ultrasonic Converter (B), driven by Power Supply (A), is producing a sufficiently strong pulse-repetitive multifrequency train of mechanical oscillations or pulses. Acoustical load (D), driven by incoming frequency and amplitude modulated pulse-train starts producing its own vibration and transient response, oscillating in one or more of its vibration modes or harmonics. As the excitation changes, following the programmed pattern of the pulse train, the amplitude in these modes will undergo exponential decay while other modes are excited.

A simplified analogy is a single pulsed excitation of a metal bell that will continue oscillating (ringing) on several resonant frequencies for a long time after the pulse is over. How long each resonant mode will continue to oscillate after a pulse depends on mechanical quality factor in that mode.

Every mechanical system (in this case the components B, C and D) has many resonant modes (axial, radial, bending, torsional, ...) and all of them have higher frequency harmonics. Some of resonant modes are well separated and mutually isolated, some of them are separated on a frequency scale but acoustically coupled, and some will overlap each other over a frequency range - these will tend to couple particularly well.

Since the acoustical load (D) is connected to an ultrasonic converter (B) by an acoustical wave-guide (C), acoustical relaxing and ringing oscillations are traveling back and forth between the load (D) and ultrasonic converter (B), interfering mutually along a path of propagation. The best operating frequency of ultrasonic converter (B) is found by adjustment when maximum traveling-wave amplitude is reached, and when a relatively stable oscillating regime is found. The acoustical load (D) and ultrasonic converter (B) are creating a "Ping-Pong Acoustical-Echo System", like two acoustical mirrors generating and reflecting waves between them. For easier conceptual visualization of this process we can also imagine multiple reflection of a laser beam between two optical mirrors. We should not forget that the ultrasonic converter (B) is initially creating a relatively low pulse frequency mechanical excitation, and that the back-and-forth traveling waves can have a much higher frequency.

In order to achieve optimal and automatic process control, it is necessary to install an amplitude sensor (E) of any convenient type (e.g. accelerometer, ultrasonic flux sensor) on the Acoustical Load (D). The sensor is connected by a feedback line to the control system of Ultrasonic Power Supply (A).

There is another important effect related to the ringing resonant system described above. Both the ultrasonic source (B) and its load (D) are presenting active (vibrating) acoustic elements, when the complete system starts resonating. The back-forth traveling-waves are being perpetually reflected between two oscillating acoustical mirrors, (B) and (D). An immanent (self-generated) multifrequency Doppler effect (additional frequency shift, or frequency and phase modulation of traveling waves) is created, since acoustical mirrors, (B) and (D), cannot be considered as stable infinite-mass solid-plates. This self-generated and multifrequency Doppler effect is able to initiate different acoustic effects in the load (D), for instance to excite several vibrating modes in the same time or successively, producing uniform amplitude distribution of acoustic waves in acoustic load (D), etc. For the same reasons, we also have permanent phase modulation of ultrasonic traveling waves (since opposite-ends acoustic mirrors are also vibrating). We should strongly underline that the oscillating system described here is very different from the typical and traditional half-wave, ultrasonic resonating system, where the total axial length of the ultrasonic system consists of integer number of half-wavelengths. Generally speaking, here we do not care too much about the ultrasonic system geometry and its axial (or any other) dimensions. Electronic multimode excitation continuously (and automatically) searches for the most convenient signal shapes in order to excite many vibration modes at the same time, and to make any mechanical system vibrate and resonate uniformly.

In addition to the effects described above, the ultrasonic power supply (A) is also able to produce variable frequency-sweeping oscillations around its central operating frequency (with a high sweep rate), and has an amplitudemodulated output signal (where the frequency of amplitude modulation follows sub harmonic low frequency vibrating modes). This way, the ultrasonic power supply (A) is also contributing to the multi-mode ringing response (and selfgenerated multifrequency Doppler effect) of an acoustical load (D). The ultrasonic system described here can drive an acoustic load (D) of almost any irregular shape and size. In operation, when the system oscillates we cannot find stable nodal zones, because they are permanently moving as a result of the specific signal modulations coming from Ultrasonic Power Supply (A)).

It is important to know that by exciting an acoustical load (D) we could produce relatively stable and stationary oscillations and resonant effects at certain frequency intervals, but also dangerous and self-destructive system response could be generated at other frequencies. Everything depends on the choice of the central operating frequency, sweeping-frequency interval and ultrasonic signal amplitudes from the ultrasonic power supply (A). Because of the complex mechanical nature of different acoustic loads (D), we must test carefully and find the best operating regimes of the ultrasonic system (B, C, D), starting with very low driving signals (i.e. with very low ultrasonic power). Therefore an initial test phase is required to select the best operating conditions, using a resistive attenuating dummy load in serial connection with the ultrasonic converter (A). This minimizes the acoustic power produced by ultrasonic converter, and can also dissipate accidental resonant power. When the best driving regime is found, we disconnect the dummy load and introduce full electrical power into ultrasonic converter. The best operating ultrasonic regimes are those that produce very strong mechanical oscillations (or high and stable vibrating, mechanical amplitudes) with moderate output (electric) power from the ultrasonic power supply. The second criterion is that thermal power dissipation on the total mechanical system continuously operating in air (with no additional system loading) is minimal. Differently formulated, low thermal dissipation on mechanical system (B, C, D) means that the ultrasonic power supply (A) is driving the ultrasonic converter (B) with limited current and sufficiently high voltage, delivering only the active or real power to a load. The multifrequency ultrasonic concept described here is a kind of "Maximum Active Power Tracking System", which combines several PLL and PWM loops. The actual size and geometry of acoustical load are not directly and linearly proportional to delivered ultrasonic driving-power. It can happen that with very low input-ultrasonic-power, a bulky mechanical system (B, C, D) can be very strongly driven (in air, so there is no additional load), if the proper oscillating regime is found.

## **Other Applications of Multifrequency Actuators**

The spectrum of various imaginable applications related to above described multifrequency structural ultrasonic actuators could be illustrated by the following list:

## 1. Ultrasonic liquid processing

-mixing and homogenization
-atomization, fine spray production
-surface spray coating
-metal powders production and surface coating with powders

- 2. Sonochemical reactors
- 3. Water sterilization
- 4. Heavy duty ultrasonic cleaning
- 5. Pulped paper activation (paper production technology)

6. Liquid degassing, or liquid gasifying (depending of how sonotrode is introduced in liquid)

- 7. De-polymerization (recycling in a very high intensity ultrasound)
- 8. Accelerated polymerization or solidification (adhesives, plastics...)

9. High intensity atomizers (cold spay and vapor sources). Metal atomizers.

10. Profound surface hardening, impregnation and coating
 -surface hardening (implementation of hard particles)
 -capillary surface sealing
 -impregnation of aluminum oxide after aluminum anodizing

-surface transformation, activation, protection

11. **Material aging and stress release on cold** -Shock testing. 3-D random excitation

# 12. Complex vibration testing (NDT, Structural defects detection, Acoustic noise...)

-accelerated 3-dimensional vibration test in liquids -leakage and sealing test -structural stability testing of Solids -unscrewing bolts testing

# 13. Post-thermal treatment of hardened steels (cold ultrasonic treatment)

-elimination of oxides and ceramic composites from a surface -profound surface cleaning -residual stress release, artificial aging, mechanical stabilization

14. **Ultrasonic replacement for thermal treatment.** Accelerated thermal treatment of metal and ceramic parts in extremely high intensity ultrasonic field in liquids.

## 15. Surface etching

-abrasive and liquid treatment-active liquids (slightly aggressive)-combination of active liquids and abrasives

# 16. Surface transformation and polishing

-combination of abrasives and active liquid solutions -electro-polishing and ultrasonic treatment

- 17. Extrusion (of plastics and metals) assisted by ultrasonic vibrations -special ultrasonic transducers in a direct contact with extruder
- Founding and casting (of metals and plastics) assisted by ultrasound

   vacuum casting, homogenization, degassing
   micro-crystallization, alloying, mixing of different liquid masses

# 19. Adhesive testing

-aging test -accelerated mechanical resistance testing -accelerated moisture and humidity testing

## 20. Corrosion testing

-in different liquids -in corrosive liquid, vapor phase



Fig. 1 Liquid-CO<sub>2</sub> Ultrasonic Cleaning, Process Diagram



Fig. 2 Block Diagram of a Multifrequency Structural Actuator



Fig. 3 Liquid-CO<sub>2</sub> Ultrasonic Cleaning Industrial Pilot Unit



Fig. 4 Liquid-CO<sub>2</sub> Ultrasonic Cleaning Autoclave



Fig. 5 SEM image of the cleaned surface of a precision writing ball at 1860 magnification



Fig. 6 SEM image of the cleaned surface of a precision writing ball at 10000 magnification



# Fig. 7 SEM image of a traditionally cleaned surface of a precision writing ball (organic solvent/water/detergent-method)

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## A Disruptive Innovation

Making the Case for CO<sub>2</sub>

by David Jackson

Today  $CO_2$  is being used in many forms throughout industry to solve a variety of surface cleaning problems.  $CO_2$  serves as an alternative to many conventional surface immersion, extraction, spray and plasma cleaning and treatment processes in an array of manufactured products ranging from radial tire molds to guidance gyros, automobile fuel injectors to spacecraft materials, and aircraft engines to disk drive components.

However with all of this apparent capability,  $CO_2$  cleaning today remains entrenched in the cleaning industry as a "specialized" or "niche" cleaning process. However, more significantly,  $CO_2$  is a transformational innovation with respect to the cleaning industry.

 $CO_2$ 's introductory performance can be attributed to the lack of innovation in its business model rather than the limitations of the cleaning technology itself.  $CO_2$  has been applied as a cleaning solution across a broad base of markets and applications and has been used in ways not possible using "more general" cleaning technology – a quality that portends a transformational cleaning technology, or in other words, a "disruptive innovation".

## Transformational Technology

Four suppositions are offered as evidence that CO<sub>2</sub> is a transformational cleaning innovation:

**Tier I. The Environment**:  $CO_2$  technology provides numerous strategies for meeting interlocking environmental imperatives. **Tier II. Economics**:  $CO_2$  technology delivers economic power in the form of a very competitive cost of ownership and on-going operational cost reductions that go directly to the bottom line of an enterprise.

**Tier III. Utility**: CO<sub>2</sub> technology has the power to satisfy a diverse mix of manufacturing and assembly requirements. **Tier IV: New-Market Transformations**: CO<sub>2</sub> technology enables new-market transformation strategies – those that lie in the third dimension where formerly non-consumers become consumers.

Over the past 20 years transformational CO<sub>2</sub> cleaning technologies

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have been developed to address the environmental, economic and utility performance requirements. A foundation formed from a combination of superior environmental, health and safety benefits (Tier I), attractive economics (Tier II), and unmatched utility (Tier III) is the basis for innovative business strategies.

The focus today is the creation and implementation of transformational business strategies - new-market business transformations (Tier IV) that exploit the unique capabilities of this foundation. New-market transformations are defined here as business strategies that produce new performance metrics and create consumers in a market of formerly non-consumers of cleaning technology.

#### Tier I. The Environment

A beneficial feature of  $CO_2$  is that it is a recycled and renewable by-product of natural and industrial processes.  $CO_2$  is commercially obtained as a gaseous by-product from ammonia and petrochemical plants as well as natural  $CO_2$  gas wells.  $CO_2$  is non-toxic, non-flammable and is a renewable and recycled precision cleaning resource.

Carbon dioxide is supplied commercially as a liquid in cylinders, mini-bulk tanks and bulk storage tanks. This is the starting compound from which purified solid, liquid and supercritical fluid cleaning agents are produced.

The U.S. Environmental Protection Agency has determined that recycled  $CO_2$  is an environmentally benign alternative cleaning chemistry for industry because there is *no net increase* in global warming gases or environmental damage as a result of using  $CO_2$ . In addition, the Southern California Air Quality Management District (SCAQMD) does not consider carbon dioxide derived for use as a recycled cleaning agent a hazardous air pollutant.

Finally, using recycled carbon dioxide as a cleaning solvent eliminates the need to produce additional quantities of organic cleaning agents. This conserves our vital oil, natural gas and energy resources, and prevents additional air and water pollution from being created.

#### Tier II. Economics and CpC

Table 1 below gives an exemplary cost-per-clean (CpC) model for solid phase  $CO_2$  cleaning using a snow shear spray cleaning technology from a case study of a disk drive manufacturer. The cleaning system used with a 50 percent duty cycle in a 2000 hour manufacturing operation will produce 3,600,000 selective cleans per year at an annual  $CO_2$  cost of \$7,500. This yields a CpC value of \$0.002 per clean using ultrapure  $CO_2$ , which is very competitive with conventional cleaning alternatives and 58 percent less than conventional  $CO_2$  cleaning.

Table 1. Annualized Cost per Clean Model Using Snow Shear Technology		
Production Day		
Hours per day	8	
Days per week	5	
Weeks per year	50	
Cleaning Process		
Cleaning cycle on (sec)	2	

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Cleaning cycle off (sec)	2	
Duty cycle	50%	
Cleans per day	14,400	
Cleans per week	72,000	
Cleans per year	3,600,000	
Carbon Dioxide Usage		
Machine setting (lbs/hour)	15	
Usage @ duty cycle (lbs/hour)	7.5	
Usage per production day (lbs/day)	60	
Usage per production year (lbs/year)	15,000	
Consumables Cost		
(Ultrapure Carbon Dioxide Supply)	\$7,500	
Cost per Clean (CpC)	\$0.002	

#### III. Utility

Precision cleaning with  $CO_2$  enhances product yields, process performance and productivity, and improves environmental quality in the workplace. Following are a summary of environmental and economic benefits discussed herein for  $CO_2$  cleaning:

- Carbon dioxide is non-toxic, non-hazardous, non-corrosive, and non-flammable.
- Cost-of-Ownership and Cost-per-Clean for CO<sub>2</sub> cleaning is very competitive.
- Carbon dioxide cleaning produces no solvent waste, does not require wastewater treatment, and requires no drying.
- Carbon dioxide will not be regulated out of existence since it is a by-product of industrial and natural processes.
- Cleaning with carbon dioxide does not add to global warming – it helps reduce it.

 $\rm CO_2$  starts as a gaseous substance, obtained from a natural or industrial source at standard temperature and pressure (STP), carbon dioxide may be pressurized and condensed to a liquid phase.

From a liquid phase,  $CO_2$  may be further cooled and condensed as a solid phase or heated and pressurized to a supercritical state. Below are just a few examples of how  $CO_2$  can be applied within the industrial cleaning markets:

- General Cleaning; precision machined or fabricated metal parts.
- Precision Cleaning; microelectromechanical (MEMS) devices, semiconductors, fiberoptics and optoelectronic devices.
- Biomedical Cleaning; sterilization, disinfecting and surgical implant preparation.
- Textile Cleaning; fabric care and treatments
- Materials Cleaning; molded and extruded polymer device extraction processes.
- Surface Modification; preparation of surfaces for underfill, coating or plating.

#### **Critical Cleaning Performance**

Tremendous advances have been made in the areas of solid, liquid, and supercritical  $CO_2$  precision cleaning technologies over the past 20 years. Two of these innovations include snow shear

### and centrifugal shear CO<sub>2</sub> cleaning, respectively.

Compared to conventional  $CO_2$  snow guns developed in the 1980's, snow shear  $CO_2$  employs spray cleaning technology, which produces a lower localized humidity and higher localized temperatures. This translates into little or no atmospheric condensation during normal scan cleaning operations. Moreover, spray pressures as low as 30 psi to as high as 5000 psi may be generated - providing a full spectrum of surface spray cleaning energy. Additionally, additives may be applied to enhance the "chemistry" of the solid phase cleaning agent.

Snow Shear spray cleaning technology is able to meet the increasing cleaning challenges posed by microscopic contaminants on critical surfaces. These pictures demonstrate the removal of microscopic (sub-micron) particles and residues from the surfaces of contaminated magnetic crash stop latches used in hard disk drives using a short  $CO_2$  cleaning operation and can remove thin film contaminants (i.e., fingerprints) and particles smaller than 0.2 microns.

Centrifugal shear technology represents another advancement in  $CO_2$  cleaning technology. Liquid carbon dioxide (LCO2) or supercritical carbon dioxide (scCO2), which may contain trace amounts of gaseous or liquid chemical cleaning additives, are used in combination with bi-directional centrifugal shear cleaning and separation action.

A centrifugal shear  $CO_2$  cleaning system comprises two modules: a dense fluid centrifuge and a  $CO_2$  cleaning fluids recovery system. Dirty substrates (e.g., aircraft bearings) are placed into a centrifugal cleaning chamber and fluidized with dense phase  $CO_2$ . During bi-directional centrifugal cleaning operations the substrates are continuously contacted with clean fluid while contaminated  $CO_2$ , wrung from the surfaces during immersion spray cleaning operations, is transferred from the cleaning chamber and drag-out management operations.

At the end of the cleaning cycle, clean dry substrates are removed from the centrifugal cleaning chamber. Centrifugal  $CO_2$  cleaning times may range from 10 to 60 minutes per cleaning cycle for general liquid  $CO_2$  degreasing applications and typically 30 to 120 minutes per extraction cycle for supercritical  $CO_2$  extraction applications. The recovery system economically recovers about 95 percent of the  $CO_2$  for reuse.

Contaminant recovery and  $CO_2$  make-up is pre-determined and managed through a scheduled drag-out and re-charge sequence, respectively, based upon contaminant loading and economic considerations. Centrifugal shear  $CO_2$  cleaning process parameters include temperature and pressure, rotational speed, ramp/brake sequences, among several other parameters specific to the centrifugal cleaning technology. The Centrifugal shear cleaning technology can meet a variety of surface cleanliness requirements for non-volatile residue, gross particulate and extractable content.

#### **Emerging Opportunities**

Only recently have compelling applications for CO<sub>2</sub> cleaning technology in the semiconductor markets emerged. This is due

primarily to a convergence of shrinking device geometries and package scale with an increasing demand for improved cleaning performance in high tech markets.

The microelectromechanical systems (MEMS), optical and integrated circuit (IC) wafer manufacturing market segments exemplify this convergence. Manufacturing, automation, and yield challenges within these market segments create almost limitless opportunities for  $CO_2$  cleaning technology. A technology gap is developing wherein current wet surface cleaning technology is reaching an upper limit of performance and applicability in these high tech market segments. After over 20 years of incubation, technological and socioeconomic factors have converged to create new high tech entrance opportunities for  $CO_2$  cleaning technology.

The advances made in technology over the last two decades shows an increasing trend toward miniaturization of devices. Many new semiconductor, microelectronic, and micro-mechanical devices are emerging and they are getting smaller. Circuit designs and mechanical features present on these devices are presently 750 times smaller than the width of a human hair. One important aspect of this trend is that as the device shrinks the cleaning requirement becomes more stringent and number of cleaning steps during manufacturing and assembly operations increases.

For example, a "killer defect" is considered to be one half the size of the device features or linewidth. In the late 1980's, this was generally in the range of 0.5 micrometers whereas today it is in the range of 0.05 micrometers as linewidths approach 0.07 micrometers. Moreover, surface cleaning is involved in 40 percent of the production operations in today's typical wafer fabrication process.

Almost all manufactured products are cleaned during production, sometimes many times and by many different processes. All products cleaned are cleaned to meet a variety of cleanliness standards from visually clean to microscopically clean. High technology products require critical cleaning. These products include sensors, integrated circuits, MEMS, disk drive components, fiber optics, precision automotive parts, implantable devices and many others across many market segments. Contaminants (dirts, oils and particles) must be removed from these products during or following manufacturing operations to prevent defects, which result in yield loss and lost revenue.

#### A Developing Technology Gap

Today's high tech cleaning requirements are much more complex characterized by microscopic scale, new surface structures, and new manufacturing materials. For example, microelectromechanical (MEMS) devices are shrinking in size but increasing in mechanical complexity. For this application, critical cleaning and drying technology must be developed to meet the increasingly stringent cleanliness and performance standards for such devices during manufacturing and assembly operations.

Cleaning and drying surface features such as microscopic beams, mirrors, gears, levers, trenches, pores, and other microstructures, while preventing new microscopic scale cleaning phenomenon such as "stiction" and "capillary collapse", cannot continue to be addressed using conventional wet cleaning technology. Wet technology cannot be used indefinitely because of the increasingly negative physical and chemical phenomenon associated with using these conventional cleaning and drying chemistries on microscopic features on miniature manufactured surfaces.

A technology gap is developing between the cleaning needs of high-tech manufacturing companies and the sustaining performance of current cleaning technology. This gap must be filled with transformational cleaning technology to sustain the growth trajectories and profitability of existing and emerging high tech manufacturing industries. Gap filling will entail the implementation of new cleaning and manufacturing performance metrics (lower right axes).

Moreover, high tech markets have already been introduced to or have just begun to test  $CO_2$  in various cleaning applications and the results are very promising.  $CO_2$  is being used in a variety of precision cleaning applications in the disk drive, optical and microelectronic device manufacturing industries. The disk drive industry has identified  $CO_2$  technology as providing reproducible and effective precision cleaning solutions for micro disk drive microcontamination issues.

Carbon dioxide will be an enabling cleaning option for the IC, MEMS and Optical wafer manufacturers at some point in the future. Confronted by finer architectures and higher aspect ratios with high sensitivity to contaminating residue that require new lowk (photoresists having very low dielectric constants) materials, traditional techniques are giving way to alternative or new ones.

The trend towards miniaturization of silicon, germanium and gallium arsenide microprocessors in the electronics industry and the emergence of new MEMS manufacturing, which use much the same microprocessor manufacturing technology, is creating new material and process challenges. Conventional cleaning, drying, etching, and deposition technologies are being pushed as contamination removal issues become more and more important with each new device generation.

#### Tier IV. New-Market Transformations

In many manufacturing operations a product is cleaned prior to or following a particular assembly process, sometimes many times through the production cycle. Historically, parts cleaning has been performed as an independent operation using, for example, a spray cleaner, vapor degreaser or ultrasonic cleaning system. Segregation of the cleaning process has been required due to the inherent chemical and physical incompatibilities between conventional cleaning operations and most assembly tools. Manufacturing and assembly operations requiring a cleaning or surface treatment process (before, during, or after) may include pick and place, cutting, drilling, trimming, micro-machining, adhesive bonding, dicing, abrasive finishing, singulation, polishing, electroplating, soldering, potting, coating, encapsulation, stamping, and welding, among many other operations.

The Clean-Assembly concept involves the integration of  $CO_2$  dry cleaning technology into various types of common assembly tools to produce new forms of new  $CO_2$ -enabled manufacturing tools. Clean-Assembly tools are much more productive because two or more assembly processes can be performed simultaneously within the same work cell. Products don't have to be removed, cleaned (inspected) and returned to the production line - resulting in reduced human interaction, higher throughput and decreased cost-of-ownership.

For example, a new robotic surface treatment and inspection platform (Clean-Inspect) is being developed that utilizes advanced carbon dioxide cleaning technology and optically stimulated electron emission analysis (OSEE). This new Clean-Inspect platform can "selectively" clean, treat and analyze critical surfaces in preparation for manufacturing applications such as bonding, painting, plating and coating.

In the traditional manufacturing model, parts cleaning is not normally considered a value-added operation. The Clean-Assembly model changes the value network for all types of manufacturing operations by incorporating or hybridizing the cleaning process with the (always) value-added assembly operations. This enhances both product yield and tool productivity.

Carbon dioxide cleaning technology has advanced considerably over the past 20 years. The next phase of development for  $CO_2$ lies in the third dimension of the transformational innovation model. The third dimension is where previously non-consumers are turned into consumers and where new contexts of consumption and competition are created.

Innovative business strategies promise to create new value networks for the cleaning industry. The result will be that over time, traditional customers will have the option to migrate into the networks created by  $CO_2$ .

#### About the Author

David Jackson has been a pioneer in the  $CO_2$  cleaning development field since 1984 at Hughes Aircraft, where he was a precision cleaning and contamination control group head. Mr. Jackson holds a Bachelors Degree in Chemistry from California State University and is the VP and CTO for Pur  $CO_2$ , a new joint venture company between Cool Clean, Inc. and Deflex Corporation. He can be reached at 661-775-7691 or <u>djackson@deflex.com</u>.

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