How clean is clean?

Glenn Greenlees, managing director of Standard Industrial Cleaning Systems, looks for some definitive answers .

Of course "it" – the workpiece or component – has to be as clean as possible, but what is acceptable? Indeed, what is essential to achieve the quality requirements of the next process or perhaps the end customer?

Those involved in microprocessor or semiconductor manufacture will be seeking a level of cleanliness which would seem incredible to the average machine shop – anybody watching the *Discovery Channel* will see satellite assembly undertaken by staff dressed as though they already belong in space. This is not a fashion statement! In these circumstances, the elimination of even nanometre scale particulate contamination is essential to maintain operational integrity, and the appropriate level of cleanroom is required to provide the filtered, temperature and humidity controlled air needed in this "super-clean" environment.

At the other end of the scale, the re-manufacture of automotive parts or the maintenance of railway rolling stock will require that gross contamination, such as heavy oils and road dirt, be removed even before work can begin. Typically, manufacturing processes like casting, pressing, punching, machining, moulding, soldering, polishing and lapping, will each require a particular level of cleanliness to be achieved – perhaps the next step will be assembly, coating, bonding or testing such as crack detection or other NDT operations.

Surface cleanliness is frequently measured in terms of residual contamination in weight per given surface area of substrate. The automotive industries now utilise extremely high gloss wet paints, which rely upon a very clean substrate in order to give the desired adhesion and mirror finish. Residual carbon levels of 5mg / m^2 are generally required to achieve the desired adhesion of these high performance products.

In addition, a high degree of mechanical filtration is also essential, as even sub micron particulate contamination will render the item cosmetically unacceptable. In powder coating the substrate requirement is not quite so stringent and values can generally be higher with specifications often quoting < 50 mg/m^2 carbon.

The above scenarios differ significantly but all still require the component parts to be appropriately "clean".

Increasing cleanliness requirements

In almost all areas of manufacture, surface cleanliness requirements are constantly increasing, in line with greater manufacturing parameters such as tighter machining tolerances and demands for greater adhesion and durability of coatings and bonding agents.

This forms (only) part of the dilemma facing those now legally required to invest in new or replacement cleaning and degreasing technologies.

The implementation of *1999/13/EC - the European Solvent Emissions Directive* and the *Solvent Emissions (England & Wales) Regulations 2004* has certainly focused the attention of most on the need to replace open solvent vapour systems with a commercially viable and legally long term compliant process, which can actually deliver the required quality on a consistent basis.

In fact, most of us have relied so much upon vapour cleaning in tanks of trichloroethylene liquor/vapour that we have rarely even had to consider the levels of cleanliness that we have been demanding or, indeed, consistently achieving.

We now see so many major corporations dictating to subcontractors what "approved" cleaning chemicals must be utilised in order to maintain their supplier status. It must be assumed that the various products have been nominated based upon such criteria as evaluated performance and chemical compatibility.

However, rarely will one find an actual process description which details how the various products should be utilised. In addition, many seem to be playing a Health & Safety or environmental card as a misguided PR exercise. The cleanliness specification requirements will often quote "no oil residues" or "no particles".

So many times the writer has been in discussion with clients when the question of required cleanliness has prompted responses such as "very clean" or even "clinically clean"– this to be achieved in a manufacturing factory environment with the often associated oily floors and contamination laden atmosphere!

It is also common to see component cleanliness criteria based upon the levels of contamination present in the process wash liquid.

In progressing an aqueous application, I was recently presented with a document requiring that "the facility should maintain a wash fluid cleanliness of 3.75 mg/litre or ISO ....".

When applied to a simple "dunk tank" immersion process (is in this case) how can this indicate the final levels of surface cleanliness of the components placed therein? If we are considering aqueous cleaning, surely the wash water must become dirty, indicating that soils have been removed from the parts? In fact, with aqueous processes we will (nearly) always be washing with dirty water.

Depending upon actual requirements, aqueous cleaning will generally be a progressive multi-step operation – the ultimate component cleanliness largely being dependant upon final rinse water quality whereas, for instance, with hermetically sealed solvent technology soils will be removed from the components and subsequently be separated from the solvent by distillation. In most cases oils and greases have a far higher boiling point than the cleaning solvent.

Cleanliness acceptance criteria should be based upon quoting a specific measurable level of component cleanliness (eg. in mg / m^2) whilst, of course, insisting that any processes adopted are fully compliant with all of the relevant legislation and standards.

The confused subcontracting manufacturer now finds himself between a rock and a hard place, he must clean with what the customer demands but, of course, the parts must meet the required standard.

For a moment let us forget the ongoing aqueous/solvent arguments and – in the true style of so many shampoo advertisements – I would suggest: "here comes the science". One dictionary defines cleanliness as "free from dirt: pure: neat: complete" – not really a great a help in establishing a practical criteria in today's ever more demanding manufacturing environments.

How do we measure cleanliness?

Surface cleanliness can be either arbitrarily ascertained or critically measured. How do we measure it? In theory very simply. We can take a "clean" item, measure it's various characteristics and then repeat the process after contamination.

Measurement can be either direct or indirect. Direct methods require the surface of the actual component part to be analysed in some way whilst indirect methods such as non-volatile residue analysis (NVR) utilise a solvent of some kind to remove residual contamination from the part which is, itself, subsequently analysed to establish its contents.

Direct methods can be simple, relying upon a variety of visual inspection techniques, magnified or otherwise, including techniques such as ultraviolet photoelectric emission, which can cause any residual contamination to fluoresce. White rags can be rubbed onto the surface of the cleaned part and the results interpreted accordingly. Of course these techniques are highly subjective.

Water itself can be used to test the surface – the principal being that many contaminants are actively hydrophobic ie. they are not readily wettable and noticeably repel any applied water.

The simple water break test requires water to be flowed over the surface. If the water "breaks" cleanly off the surface leaving the part dry it will give an indication that the part is demonstrating a high degree of wettability and is clean. If the water forms beads or globules it will indicate that residual contamination is present. A practical demonstration experienced most weekends when our beloved automobile is rained upon after some hours of washing and buffing. The car surface is, is fact, highly contaminated with surface wax deposits.

To be effective as an indication of surface cleanliness the water used in the test must, itself, be free from contamination which would alter its flow characteristics. Again this test is rather subjective but the principal can be extended to give a significantly more quantifiable indication in a contact angle test where a measured amount of pure water is strategically placed upon the flat surface and allowed to settle.

The angle formed by the drop and the surface substrate can then be measured. A clean surface will give a low contact angle whilst the presence of contamination will cause the drop to "cling" to the surface giving a high angle of contact.

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About the author . . .

The author of this article, Glenn Greenlees, is marketing director of Standard Industrial Cleaning Systems, an independent distributor “dedicated to offering the world’s best in both aqueous and solvent cleaning technologies.” Glenn believes that with the many changes in both environmental legislation and solvent classification it is vital that potential investors are given the facts regarding the technologies available. Standard Industrial CleaningSystems is the exclusive UK distributor for the Italian mannufacturer Firbimatic Spa, a European market leader in the design and manufacture of hermetically sealed solvent based degreasing systems. The Firbimatic range includes machines for use with trichloroethylene, perchloroethylene, A111 hydrocarbons and modified alcohols. Special applications are addressed with bespoke systems.

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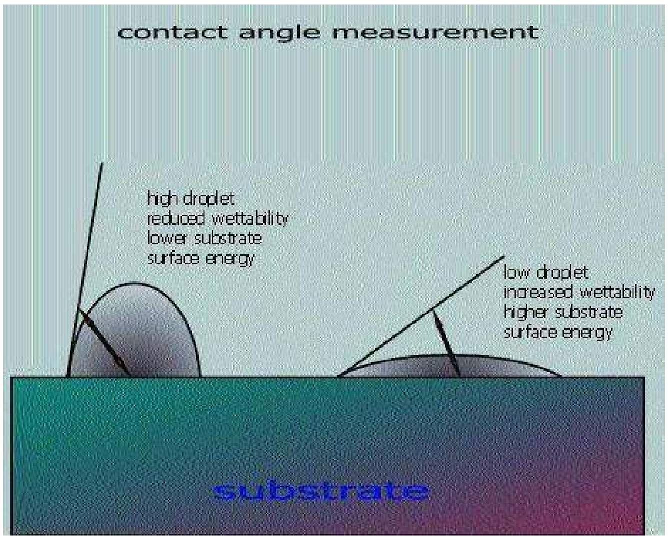

The test *(see above illustration)* can be simplified and “brought out of the laboratory” by replacing the angle measurement with an optical comparison of the size of the droplet – large diameter and flat being an improvement over small diameter and tall for a measured volume of water.

As an alternative, the water can be replaced with liquids which are calibrated as test inks – the surface tension (critical energy) of the liquid is known (in mN/m or Dynes/Cm) and when matched with the component surface, indicated by a continuous unbroken line, the cleanliness accordingly noted. The surface energy of metals is much higher than that of surface contaminants; thus, the higher the dyne level, the cleaner the part is. These are semi-quantitive techniques which are extremely useful for on site monitoring of surface integrity or indeed, when evaluating cleaning processes – for instance the typical requirement for subsequent vacuum processes being an equivalent surface energy of > 40 mN/m whilst preparation for printing with solvent based inks will require 38 mN/m and many adhesives will require substrate displaying > 44 mN/m to ensure satisfactory bonding.

Of course these wettability tests are an indication of the relative surface energies of test liquid and substrate and problems can arise due to the formation of passive layers which will restrict the achievable surface energy . These passive layers may, of course, have been left as residual contamination due to insufficient cleaning but can also be generated as a result of the chemistry of an aqueous cleaning process itself. Furthermore, many materials are fundamentally restricted in the levels of surface energy achievable in practical tests. Whilst, for instance aluminium has a natural (high) surface energy of 660 mN/m it will be unable to demonstrate satisfactory wetting with tap water (at 72 mN/m) due to the formation of surface oxides - this must not be confused with a lack of surface cleanliness i.e. the absence of oil or grease residues. Similarly, many plastics possess inherently low surface energies and will be readily wettable by such test liquids (the value for PTFE is < 20 mN/m).

Evaluation of surface cleanliness using relative substrate surface energies with wettability techniques is simple, inexpensive, mobile and provides instant readings providing the operator is familiar with the natural limitations of the materials under test.

There are many other surface cleanliness test procedures which require equipment ranging in sophistication and user expertise. These include:

Evaluating substrate surface energy to indicate cleanliness.

- ***Gravimetric Analysis****– this can be either a direct or indirect technique and requires scales accurate to 1 milligram (or better). The direct method requires the component sample (which must be small) to be measured before and following the cleaning process – the difference is attributed to the levels of residual contamination. For an indirect approach, the remaining soils are removed by an appropriate solvent which is then filtered/evaporated to leave the residue available for weighing.*
- ***Scanning Electron Microscopy (SEM)****– a beam of electrons is passed over the surface of a sample, which scatters, allowing surface topography to be inspected.*
- ***X-Ray Photoelectron Spectroscopy (XPS)*** *– a highly sophisticated and expensive process which uses special equipment to bombard the surface of interest with x-rays under vacuum conditions, causing electrons to be released from the surface. Since each type of element (ie. carbon, oxygen, etc.) releases a unique amount of electrons under these conditions the actual elemental composition of the surface can be quantified.*
- ***Secondary Ion Mass Spectroscopy (SIMS)*** *– the sample surface is bombarded by ions which partly ionises the eroded surface material. The ionised component is analysed in a mass spectrometer.*
- ***Gas Chromatography/Mass Spectrometry*** *– residual contamination is extracted using a solvent and subsequently analysed.*
- ***Total Organic Carbon (TOC)****– TOC analysis is used to quantify the amounts of organics residues on a substrate surface.*

Here in the UK it is apparent that, for the majority of applications, the selection of appropriate cleaning and degreasing techniques is still very much a hit and miss affair. Many companies still spend months or years in “evaluating” the processes available.

In fact, this R & D is very often a time consuming and costly trial and error approach, often wasting resources with products and processes which are chemically and physically incapable of achieving the desired results on a long term and compliant basis. It is also common to find the same tests being repeated within many divisions of the same organisation.

In so many areas of manufacture the specific surface cleanliness requirements have already been fully researched and evaluated and the appropriate required process nominated and implemented. With the demise of the traditional double lidded “tank” type solvent vapour cleaning perhaps it is time now to give the question of surface cleanliness the serious attention it deserves.***Tel: 0151 326 2314.***