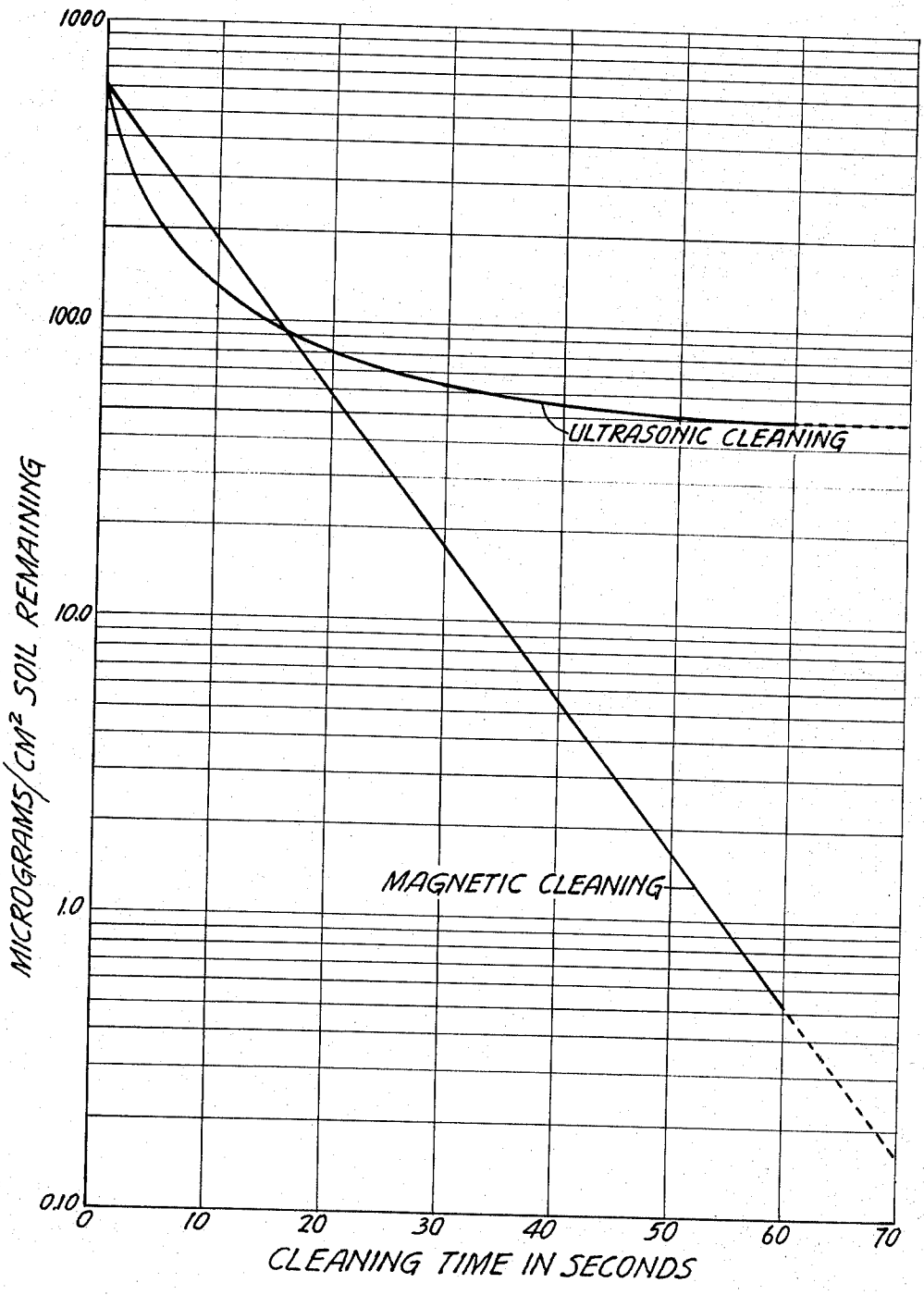
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MAGNETIC CLEANING

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MAGNETIC CLEANING

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ABSTRACT OF THE DISCLOSURE

Articles are cleaned by immersion in a moving magnetic fluid containing a cleaner, the movement being induced by an alternating gradient electro-magnetic field acting upon magnetic particles.

BACKGROUND OF THE INVENTION

This invention relates to cleaning articles.

Articles made of metal, plastic, wood, ceramic, etc., have in the past been cleaned by various techniques including soaking in tanks containing solvent or detergent solution, mechanical stirring often being utilized to aid the cleaning process; vapor degreasing and rotary barrel tumbling with detergent solutions or solvents; ultrasonic methods wherein cavitation is induced in a liquid by means of sound waves; and electro-cleaning wherein electro-conductive articles are immersed in a conductive liquid connected as the anode of a direct current circuit. Additionally, aqueous solutions containing detergent have been used in ultrasonic cleaning as exemplified by U.S. Patent 3,402,075.

One particular need for better cleaning is with ceramic-gold integrated circuit holders to obtain low electrical leakage between leads. Presently available ultrasonic cleaning techniques are only about 98% effective in reducing electrical leakage to an acceptable level.

SUMMARY

The invention provides an efficient means of cleaning articles made of metals, plastics, wood, ceramic, etc. in a more rapid manner than that provided by soak tank cleaning, vapor degreasing, barrel tumbling, electro-cleaning, or ultrasonic cleaning, often at lower temperatures, all without physical or chemical change of the surface of the article being cleaned. It is possible to clean irregularly shaped articles such as ceramic-gold integrated circuit holders so efficiently that electrical leakage is less than $\frac{1}{10}$ that obtained by ultrasonic cleaning, all in a shorter time and at a lower temperature than is possible by ultrasonic techniques.

In accordance with the invention, articles are cleaned by immersion in a moving fluid comprising surfactant coated magnetic particles, chemical cleaner, and carrier fluid, said movement being induced by an alternating gradient electro-magnetic field.

BRIEF DESCRIPTION OF THE DRAWING

The drawing graphically illustrates the efficiency of magnetic cleaning and is a plot of the amount of soil remaining on aluminum tags after cleaning by magnetic and ultrasonic techniques for various time periods. Details of the procedures used in arriving at the plot are set forth in Example 1.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The previously mentioned magnetic fluid comprises surfactant coated permanent magnetic particles in a carrier liquid. Each particle is an individual permanent magnet which may be influenced by an electro-magnetic field. The permanent magnetic particles are such as gamma iron oxide (Fe_2O_3), hard barium ferrite, ($BaO \cdot 6Fe_2O_3$), particulate aluminum-nickel-cobalt alloys, or mixture thereof. Suitable magnetic particles have been found to have a magnetization (M) in excess of 10 gauss per gram, magnetization being a measure of the magnetic field intensity of the material from which the particles are prepared. Hard $BaO \cdot 6Fe_2O_3$ has a magnetization of about 70 gauss/gm. and gamma Fe_2O_3 has a magnetization of about 50 gauss/gm. Also, it has been found that suitable magnetic particles should have a magnetic coercivity (defined as the opposite sign field necessary to reduce the magnetization (M) to zero) greater than the alternating-gradient electro-magnetic field (H) applied to cause physical movement of the particle. Electro-magnetic fields of about 100 to about 1000 oersteds and higher have been used to move the particles. Hard $BaO \cdot 6Fe_2O_3$ has a magnetic coercivity of about 3000 oersteds and gamma Fe_2O_3 has a magnetic coercivity of about 300 oersteds. Magnetic particles having a magnetic coercivity less than about 100 oersteds have been found to not be particularly suited for use in the invention because application of external electro-magnetic fields sufficiently strong to move the particles causes demagnetization. Application of an electro-magnetic field to the permanently magnetic particles causes them to experience a torque tending to rotate them to an alignment parallel to the field. Variation of the direction from which the magnetic field is applied causes constant movement or rotation of the particles. It has been found that for effective use in the invention, the magnetic particles should be about 0.1 to about 20 microns in diameter, preferably about 1 to about 5 microns.

The previously discussed magnetic particles to be effective in the invention must be precoated with a surfactant. The surfactant aids in keeping the particles dispersed in the carrier liquid, prevents agglomeration of particles and aids in keeping particles from adhering to the articles being cleaned. It has also been found that the particles do not effectively clean articles if not coated with surfactant. The surfactants are effectively coated on the particles by ball milling. Suitable surfactants are those which best suspend the magnetic particles and which are compatible with and do not react with the cleaner composition utilized, said cleaners being hereinafter described. Preferred surfactants are the anionics such as sodium naphthalene sulfonate, and non-ionics such as octylphenoxy-tetra-ethyleneoxyethanol, octylphenoxy-octoethyleneoxyethanol, octylphenoxy-undeca-ethyleneoxyethanol, sec-pentadecanoxy-tetraethyleneoxyethanol, sec-pentadecanoxy-undeca-ethyleneoxyethanol.

The carrier liquid used with the previously described magnetic particles to provide the magnetic fluid may be water or an organic liquid such as kerosene, carbon tetrachloride, acetone, benzene, toluene, perchloroethylene, etc. The carrier liquid must be chosen such that it will not react with the magnetic particles being used or the articles being cleaned and may additionally contain such other components as, for example, abrasive particles, bactericides, pH regulators, alkaline builders, buffering agents softeners surfactants, etc.

The excellent cleaning obtained by practice of the invention requires incorporation of a cleaner in the previously described magnetic fluid. Several types of cleaner compositions have been found effective. The specific cleaner composition preferably utilized is the one which would normally be used to clean the articles to be cleaned and which is compatible with the carrier liquid and with the surfactant coated on the magnetic particles. Oil based cleaners are generally used with organic solvent carrier liquids and surfactants are generally used

with aqueous carrier liquids. The pH of the magnetic fluid and cleaner should not be excessively high or low because strongly acidic or alkaline solutions may attack the particles or articles being cleaned. The cleaner composition should not contain a surfactant of ionic charge opposite that of the surfactant coated on the magnetic particles, or agglomeration of the particles may result causing poor cleaning and difficulty in removing the particles from the articles being cleaned. It is, in some instances, possible to utilize non-ionic surfactants with either anionic or cationic surfactants. It has been found that the magnetic particles will settle in the carrier liquid if the composition is allowed to remain still for an extended period of time; however, they readily re-suspend when exposed to an alternating gradient electro-magnetic field. If desired, abrasive particles may be incorporated in the carrier liquid to obtain enhanced cleaning.

It has been found that the magnetic fluid which comprises carrier liquid, magnetic particles, cleaner, and any additional components should have a viscosity of about 10 to 7500 cps., preferably about 100 to 1500 cps. at 72° F. as measured on a Model LVF Brookfield Viscometer using a No. 2 spindle at 6 r.p.m. These fluids have been found to exhibit a thixotropic behavior and have a thixotropic index of about 7. For example, a fluid having a viscosity of 152 cps. when measured with a No. 2 spindle at 60 r.p.m. exhibits a viscosity of 1,050 cps. when measured at 6 r.p.m. with the same spindle. Fluids having a viscosity higher than about 7500 cps. have not been found to be effective because particle movement is not feasible at reasonable H fields. For effectve cleaning, the volume ratio of particulate, whether magnetic particles or abrasive particles, to liquid should be from about 1:100 to 30:100, preferably about 15:100 to 25:100.

Compositions comprising magnetic and abrasive particles in a liquid and moved by a magnetic field have been disclosed for polishing and abrading articles, as shown for example, by U.S. Patents 2,735,231; 2,735,232; 2,787,-854; and 3,423,880.

The previously described cleaning of soiled articles is preferably achieved by placing the articles, magnetic fluid and cleaner in a non-magnetic container and placing that container within a field that will cause movement of the particles. An alternating gradient electro-magnetic field is preferred and may be generated by means of oscillators, oscillator/amplifier combinations, solid-state pulsating divices, motor generators, and mechanical vibrators. It is also possible to move the particles by means of revolving permanent magnets and by rotation of the magnetic fluid in a fixed direct current field. The electromagnetic field may be applied to the magnetic fluid by means of air or metal core coils, stator devices, etc., and may be applied from any direction individually or simultaneously.

A particularly effective way of obtaining rapid efficient cleaning is by placing the magnetic fluid, cleaner, and articles to be cleaned into a non-metallic container and placing that container within an electric motor stator having field coils wound with a basket mesh weave. Such a stator is conveniently run off of readily available 60 cycle current. Operation at higher frequencies provides greater cleaning efficiency.

Effectiveness of cleaning in the afore-described manner is readily determined by cleaning articles containing a pre-determined amount of synthetic soil, cleaning the articles and determining the amount of residual soil. Specifically, aluminum tags were polished to a surface roughness of RMS–12 micro-inches, thoroughly cleaned in a soak tank, and synthetic soil applied. The synthetic soil comprised 0.2 part by weight anhydrous lanolin, 5 parts C^{14} tagged 1 micron carbon black particles, 4 parts magnesium silicate, 20 parts kerosene, and 5 parts lubricating oil, the soil being cured at 200° F. for 48 hours following application. The concentration of tagged soil was determined by beta particle counting with a gas Geiger counter. The tags were separated into groups, each group containing about 1875±50 micrograms of soil. The tags are then subjected to the various cleaning procedures hereinafter described, the radioactivity again counted, and the amount of residual soil, expressed as a percent of the original amount of soil or as micrograms per square centimeter was calculated.

The following examples, in which all parts are by weight unless otherwise indicated, illustrate preparation of the magnetic particles, magnetic fluids and cleaner compositions of the invention, without limiting the scope thereof.

Example 1

This example compares the effectiveness of cleaning by the magnetic techniques of the invention and of cleaning by ultrasonic techniques. Aluminum tags precoated with synthetic soil in the previously described manner were magnetically cleaned as hereinafter described for varying periods of time. Similarly soiled tags were cleaned for the same time periods by ultrasonic methods. Comparative results of the residual soil are clearly shown in the drawing.

A magnetic fluid of the following composition was prepared:

	Parts
$BaO \cdot 6Fe_2O_3$ particles (1 micron average diameter)	20
Silicon carbide (500 mesh and finer)	20
Sec-pentadecanoxy-tetra - ethylenoxyethanol	5
Sec-pentadecanoxy-tetradeca-ethylenoxyethanol	5
Triethanol amine	3

The above components were combined in a ball mill with 500 parts of water and milled for 30 hours at 24 r.p.m. using ½ inch diameter glass balls. The surfactants also functioned as the cleaner. Aluminum tags synthetically soiled with 1875±50 micrograms of the previously described soil were cleaned for 15 seconds. The cleaning was accomplished by placing the magnetic fluid at 72° F., and aluminum tags in a 400 ml. glass beaker, placing the beaker inside a 36 segment stator having a basket mesh weave spanning about 9 segments, (obtained by rewinding a ¼ horsepower single phase electric motor), applying 60 cycle 110 volt current for 15 seconds, removing the tags, rinsing them in deionized water, and determining the residual soil by means of a Geiger counter.

The same procedure was repeated with similarly contaminated aluminum tags cleaned for 30 seconds, 45 seconds, and 60 seconds. The results of each cleaning experiment are shown in the drawing with the amount of residual soil being expressed as micrograms per square centimeter of tag surface area.

Similarly contaminated aluminum tags were cleaned for the same time periods as used above by ultrasonic techniques. The same concentration of surfactant and cleaner were used at the same temperature in an ultrasonic cleaning unit having the same volume and geometry as that used for magnetic cleaning. The magnetic and abrasive particles were omitted. The soil residual found after cleaning separate groups of tags for 15 seconds, 30 seconds, 45 seconds, and 60 seconds are plotted in the drawing, the dramatic difference between magnetic and ultrasonic cleaning being quite clear.

Example 2

As controls, mild steel discs containing a known quantity of radioactive synthetic soil applied as previously described were (1) cleaned in a water soak tank with mechanical agitation (three blade paddle of 2 inch diameter at 1000 r.p.m.) (2) cleaned in a soak tank containing detergent solution without mechanical agitation, and (3) in a soak tank using mechanical agitation and detergent solution.

In each instance, the solution temperature was maintained at 180° F. and the steel discs cleaned for 3 minutes.

The controls containing detergent solution had 8 ounces per gallon of an alkaline cleaner comprising approximately equal amounts of sodium meta silicate and tetra sodium pyrophosphate together with a small proportion of sodium alkyl aryl sulfonate.

The amount of residual soil (1) with plain water and mechanical agitation was found to be 44 percent, (2) with detergent and no mechanical agitation, the residual soil was 11 percent, and (3) with detergent and mechanical agitation, the residual soil was 3.1 percent.

Example 3

This example compares cleaning (1) with cleaner alone, (2) with magnetic fluid alone, and (3) with magnetic fluid containing cleaners.

A magnetic fluid of the following composition was prepared:

	Parts
($BaO \cdot 6Fe_2O_3$)-1 micron ave. diameter	30
Silicon carbide (500 mesh and finer)	30
Sec-pentadecanoxy-undeca-ethylenoxyethanol	1
Sec-pentadecanoxy-tetra-ethylenoxyethanol	1
Triethanol amine	0.25

The above components were combined in a ball mill with two liters of water and milled for four hours at 24 r.p.m. using ½ inch diameter glass balls. After milling, the mixture was filtered to remove the water and 38 parts of the alkaline cleaner solution used in Example 2 at 8 oz./gallon added.

Mild steel discs containing a known quantity of the previously discussed synthetic soil applied as previously described were cleaned by placing a 3 liter glass beaker containing 1 liter of the magnetic fluid cleaner composition at 180° F. inside an air coil. An electro-magnetic field of 150G at a frequency of 800 Hz. was applied for 3 minutes, and the parts thereafter rinsed in tap water at 140° F.

Similarly soiled steel discs were cleaned by the same electro-magnetic technique with the beaker containing only magnetic fluid without cleaner. Also, similarly soiled steel discs were cleaned with the cleaner without the aid of magnetic fluid.

The amount of residual soil (1) with the magnetic fluid-cleaner composition was found to be 0.07 percent, (2) with mechanical action alone the residual soil was found to be 19 percent, and (3) with cleaner alone without mechanical action the residual soil was found to be 11 percent.

The synergistic effect of the magnetic fluid-cleaner composition is evident from the amounts of residual soil.

Example 4

Magnetic particles prepared by the tcehnique described in Example 3 were utilized at 20 percent by weight in kerosene to provide a magnetic fluid. To the kerosene magnetic fluid was added 20 percent by weight of a cleaner comprising 80 parts kerosene, 15 parts anionic alkyl sulfonate detergent (commercially available from the Cincinnati Millacron Chemical Co. under the trade designation Carlisle Base 600), 1 part pine oil, and 4 parts SAE 50 non-detergent engine oil.

Mild steel discs containing a known quantity of the previously discussed synthetic soil applied as previously described were cleaned for three minutes at 180° F. in a glass beaker in an air coil by applying an electro-magnetic field of 150G at a frequency of 800 Hz. The discs were found to contain 0.71 percent residual soil, whereas using the same cleaner in a mechanically agitated soak tank was found to provide discs containing 5.42 percent residual soil.

Example 5

Magnetic particles prepared by the technique described in Example 3 were utilized at 20 percent by weight in water to provide a magnetic fluid. To the aqueous magnetic fluid was added 20 percent by weight of a cleaner comprising 40 parts kerosene, 15 parts of the anionic alkyl sulfonate detergent used in Example 4, 40 parts water, 2 parts pine oil, and 3 parts octylphenoxy-octo-ethylenoxyethanol (commercially available from the Rohm & Haas Company under the trade designation "Triton 100X").

Mild steel discs containing a known quantity of the previously discussed synthetic soil applied as previously described were cleaned by placing a 3 liter glass beaker containing 1 liter of the magnetic fluid-cleaner composition at 180° F. in an air coil. An electro-magnetic field of 150G at a frequency of 800 Hz. was applied for 3 minutes and the parts thereafter rinsed. The discs were found to contain 1.43 percent residual soil, whereas using the same cleaner in a mechanically agitated soak tank was found to provide discs containing 13.3 percent residual soil.

Example 6

Magnetic particles prepared by the technique described in Example 3 were utilized at 20 percent by weight in water to provide a magnetic fluid. To the aqueous magnetic fluid was added 20 percent by weight of a cleaner composition comprising 10 parts sodium hydroxide, 0.2 part octylphenoxy - undeca - ethylenoxyethanol (commercially available from the Rohm & Haas Company under the trade designation "Triton X102"), 44.8 parts sodium carbonate, 10 parts sodium phosphate, and 35 parts sodium silicate.

Mild steel discs containing a known quantitiy of the previously discussed synthetic soil applied as previously described were cleaned by placing a 3 liter beaker containing 1 liter of the magnetic fluid cleaner composition at 180° F. in an air coil. An electro-magnetic field of 150G at a freqeuncy of 800 Hz. was applied for 3 minutes and the parts thereafter rinsed. The cleaned discs were found to contain 0.66 percent residual soil, whereas using the same cleaner composition in a mechanically agitated soak tank was found to provide discs containing 1.81 percent residual soil.

Example 7

Magnetic particles prepared by the technique described in Example 3 were utilized at 20 percent by weight in water to provide a magnetic fluid. To the aqueous magnetic fluid was added 20 percent by weight of a cleaner composition comprising 5 parts octylphenoxy-hexa-ethylenoxyethanol (commercially available from the Rohm & Haas Company under the trade designation "Tergitol 15–S7"), 20 parts sodium sulfate, 5 parts butyl Cellosolve, and 70 parts sodium phosphate.

Mild steel discs containing a known quantity of the previously discussed synthetic soil applied as previously described were cleaned for 3 minutes at 180° F., by the technique used in Example 6, the discs being found to contain 0.01 percent residual soil. Similarly contaminated steel discs cleaned with the same cleaner in a mechanically agitated soak tank at 180° F. for three minutes were found to contain 0.25 percent residual soil.

Example 8

Magnetic particles prepared by the technique described in Example 3 were utilized at 20 percent by weight in water to provide a magnetic fluid. To the aqueous magnetic fluid was added a cleaner composition comprising a 20 percent by weight aqueous solution of trisodium phosphate.

Mild steel discs containing a known quantity of the previously described synthetic soil were cleaned for three minutes at 180° F. by the electro-magnetic technique used in Example 6 and were found to contain 6.9 percent residual soil. Similarly contaminated steel discs cleaned in a soak tank containing the same cleaning composition were found to contain 13 percent by weight residual soil.

Example 9

The effectiveness of magnetic cleaning is readily shown by cleaning 40 lead ceramic-gold integrated circuit holders. After cleaning, the electrical leakage between any two leads must be less than 1×10^{-9} amps at 100 volt D.C.

Conventional celaning of these parts consists of cleaning in an ultrasonic cleaner for 10 minutes with 6 oz./gal. of detergent at 160° F., rinsing, and drying. Approximately 98% of the holders are cleaned sufficiently well to pass the electrical leakage test.

For magnetic cleaning, an aqueous cleaning solution comprising 20 percent by weight of magnetic particles prepared as described in Example 3 with 5 percent by weight of a cleaner comprising 56 parts water, 20 parts silicon carbide particles (500 mesh and finer), 2.3 parts of a mixture of surfactants which were ethoxylated C_{11-15} secondary aliphatic alcohols containing 5 ethylene oxide units, 1.5 parts of a second surfactant mixture comprising ethoxylated secondary C_{11-15} aliphatic alcohols containing 12 ethylene oxide units and 0.5 part triethanol amine. Circuit holders were cleaned for 30 seconds at 72° F. using an electro-magnetic field of 150G at a frequency of 800 Hz. One hundred percent of the cleaned circuit holders had an electrical leakage of less than 1×10^{-10} amps at 100 volts D.C.

I claim:

1. A cleaner composition activatable by an alternating gradient electro-magnetic field, said composition comprising carrier liquid, surfactant coated magnetic particles, and cleaner, said magnetic particles having a coercivity of not less than about 100 oersteds.

2. The composition of claim 1 wherein the carrier liquid is water.

3. The composition of claim 1 wherein the carrier liquid is a non-aqueous solvent.

4. The composition of claim 1 wherein the volume ratio of particulate matter to liquid is from about 1:100 to 30:100.

5. The composition of claim 4 wherein the magnetic particles have a magnetization not less than about 10 gauss per gram.

6. The composition of claim 5 wherein the average diameter of the magnetic particles is on the order of about 0.1 to about 20 microns.

7. A cleaner composition activatable by an alternating gradient electro-magnetic field, said composition comprising aqueous carrier liquid, barium ferrite magnetic particles coated with non-ionic surfactant, and cleaner, said mangetic particles having a coercivity of not less than about 100 oersteds, a magnetization of not less than about 10 gauss per gram, an average diameter of about 1 to about 5 microns, the volume ratio of particulate matter to liquid matter being about 1:100 to 30:100.

8. The composition of claim 7 wherein said cleaner is an alkaline detergent.

9. A method of cleaning an article of manufacture comprising:

(A) providing a readily pourable liquid cleaning medium which includes
  a chemical cleaning composition,
  a carrier liquid, and
  surfactant-coated magnetic particles having a coercivity of not less than about 100 oersteds and an average diameter of about 0.1 to 20 microns;
(B) placing said cleaning medium in a non-magnetic container,
(C) immersing said article in said medium,
(D) imposing an alternating frequency electro-magnetic field on said cleaning medium for a time sufficient to achieve the desired cleaning result,
(E) removing the article from the cleaning medium, and
(F) rinsing the article to remove the excess cleaning medium.

10. Method of claim 9 wherein said particles are barium ferrite.

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