
Win Labuda

My professional life, which now in 1998 has lasted almost 45 years, has offered me - in spite of the technologically vital time in which we live - a fascinating chance: to do research in an area to which only a few others worldwide have devoted themselves: cleaning by wiping to produce ultra-clean surfaces. It may be that the usefulness that one expected to gain in this area seemed limited or also that the multitude of physical and chemical parameters involved seemed to make systematic research complicated: seldom were enthusiastic companions in the HiTech land of ultraclean wiping and its measurement to be found. That is actually incomprehensible when one considers that the global market for cleanroom wipers is now worth about one-fourth billion DM and that cleaning by wiping is a time-consuming procedure which has a lot of time-/cost-saving potential. However, wise and friendly advice, the support of experienced friends, and conducive circumstances assisted me on my way. So today I can look back, not without pride, on twenty years of successful work, in which the once little-appreciated „cleaning rag“ has become a respectable HiTech product.

The Research into Cleaning by Wiping

a personal retrospective of 20 years of research, 1978-1998

Win Labuda
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On the occasion of my 60th birthday I have tried to write a retrospective for my friends, but also for my own orientation - a retrospective back to the time when cleaning by wiping technology had no name in Germany, when microchips had a memory of 64 k-bit, and most wipers in cleanrooms consisted of white cellulose.

Acknowledgements to Friends

Some of you deserve in the context of our research activities the expression of my deep gratitude for your valuable support and advice:

Edward Paley, the American pioneer in cleaning technology, gave me the first insights into the subject area of technologically-sound cleaning. He told me in 1973:

„The structures of some HiTech processes will become smaller, but the spatial extent of the contaminators remains in principle unchanged. Out of that a new great industry will develop.“

His foresight changed my life. After working as a representative in Germany for his company, *The Texwipe Corp.* for six years I founded the *Clear & Clean GmbH* in Lübeck/Germany in 1979. At that time there were still no cleanrooms in Europe in the contemporary sense, but it became apparent that it would come, and so I devoted myself intensively and exclusively to *cleaning technology.*

When the first large cleanroom at Siemens AG came into being in Germany in 1985, it was the young physicist Lodevicus Hermans who first urged me to observe more exactly the phenomenon of particle, fibre, and ion generation from wiping materials and other cleanroom supplies and to write these observations down. Thanks is due to him for the first impulse to build up systematic research and for a host of stimulating suggestions.

In 1987 I met *Yuko* - today my wife, who took on the physical and chemical analysis in our - at that time - still very small laboratory with a dedication that almost only a Japanese is capable of. She made further studies in chemistry and later contributed considerably to the good reputation and growth of the C&C labs.

Even today I often think in gratitude of my friend from youth - now deceased - *Hans Zerle* one of the „forefathers“ of Clear & Clean GmbH. In 1990 he figured out how to acquire an electron scanning microscope, at a bargain and by adventurous means, from the arsenal of a large German electric company. This instrument soon became the centre of our research and has remained so until today.

In 1985 I was asked by my friend *K.G. Müller* to work in the VDI (Association of German Engineers) on national guidelines for surface cleanness; a project to which *Dr. Peter Ehrler* from the Textile Institute in Denkendorf, *Prof. Heinz Fissan* from the University in Duisburg and *Mr. Willibald Poesch*, at that time chief engineer of IBM Deutschland GmbH, contributed definitively. They all impressed and motivated me in different ways to continually deepen my knowledge of the chosen subject area and to be open to public discussion. From *Dr. Peter Ehrler* I learned to honor the questioner and dissentor in the field, not to refuse to wish him well, to quote his work, and to build up human relationships in spite of differing opinions. *Professor Heinz Fissan* and *Willibald Poesch* stimulated me through their thoughtful lectures and essays, presented in beautiful clarity. They also stimulated me to emulate them in diction and in focus, although until now I have not been as successful in that way. At a conference for textile technology in 1993 I met *Professor Eckhard Schollmeyer*, a well-known textile researcher from Krefeld, who has since become a friend. He undertook to integrate us into the activities of his institute in many ways. Countless discussions, a joint lecture, and several of my lectures at textile research conferences were the fruits of our cooperation till now. The intellectual closeness of a research institute stimulates a small enterprise like Clear & Clean to make efforts that perhaps otherwise might not have been possible.

In the exchange of ideas with *Prof. Schollmeyer* and his excellent colleagues in the German Textile Research Centre in Krefeld in 1995 the most intensive phase of our work to the present began: the research of the micro-mechanisms of cleaning by wiping, which had been unnoticed until then. There the goal is always the creation of ultrapure surfaces in the subnanometer realm with the aid of textile wiping materials. For that we not only brought in the possiblities of *atomic-force microscopy*, but *microgravitometry* and e*llipsometric profilometry* as well.

An unceasingly committed and stimulating friend and sponsor to me has been and is *Klaus Schöttle*, the pugnacious Swabian and experienced mechanical engineer. Not only do I owe him discussions, fruitful criticism and many constructive ideas which concern the measuring of particle release from textile materials, but also valuable advice on the construction of production facilities and many holidays sacrificed out of pure friendship.

It would have been much harder or not at all possible for me to grasp much of this, if I had not had my friend *Dr. Ulrich Heim*, the physicist, researcher, and walking store of information, who knows the answer to any scientific question in the shortest of times or who at least knows someone who knows an answer or can contribute pages and pages of litera-

ture to the topic. He has led me into the most beautiful realm of epistemological dreams and provided me with marvellous and unforgettable reading.

Successful research is often not possible without the support of friends. Perhaps they are not directly involved in the research work, but they nevertheless work in the background by showing confidence in the matter. Even when doubts seemed to prevail everywhere; there were friends who quietly saw to it that there were enough contracts to pay for the research in good and also in bad times. Today I would like to express a special thanks to them even if they naturally would not want to be mentioned by name here.

The Analysis of the wiping procedure

Since the arrival of cleaning by wiping in the cleanrooms with their submicron particles and thinnest of contamination layers, the desire has existed for a simple method to compare and classify the suitability of the wiping materials of various manufacturers for the tasks of precision cleaning. The greatest problem in this context is understanding the multitude of parameters dependent on each other during a wiping procedure (Fig. 7). This concerns surface-edge phenomena, strength and attrition parameters, the laws of particle generation and adhesion, the capillary structure and distribution and - not least - the chemical and also triboelectric parameters. Cleaning by wiping in the micro-spectrum is a complicated physical domain, which demands a high degree of technical insight. Many engineers and physicists do not want to subject themselves to the work of getting thoroughly acquainted. At the same time it seems natural to most of us, that anyone can state a technical opinion about this product „wiper“, which had until recently a relatively low technological product image. Here is the reason for the many thoughtless testing methods and prejudices which in part originate from the test laboratories of even well-known international HiTech companies. To those belong the initial „testing methods“ developed in 1985 *„Stretching cleanroom wipers above the probe of an air particle counter“* (Fig. 10) and *„Tapping cleanroom wipers above the probe of an air*

Year	Description	Parameters	Evaluation by
1980	Scrape-blade Method	Abrasion strength of textile materials	Light microscope
1986	Labuda-Hermans-Probe	Particular surface cleanness	Air-particle-counter
1988	Ball-hammer Method	Particle residue on textile materials	Air-particle-counter
1992	Colander Method	Dry particle attrition of textile materials	Air-particle-counter and rotative-wiping-simulator with colander
1994	Bowl Method I	Dry or wet particle attrition while wiping over surfaces of a given roughness	Wiping-simulator I and liquid-particle-counter
1994	Method after Fissan &Opiolka	Cleaning efficiency of wipers for dry submicron-particles on smooth surfaces	Linear-wiping simulator/spec. picture analysis
1996	Part-Lift Method	Particulate cleanness of flat surfaces especially appropriate for mesoparticles (> 10 µm)	Picture analysis after Klumpp
1997	Bowl Method II	Particle release dependent on pressure and the friction coefficient surface/wiper	Wiping-simulator II and liquid-particle-counter Torque-scanner, liquid-particle-counter or microscope
1998	Ellipsometer Method	Cleaning efficiency of wiping materials for contamination layers < 1 nm thickness	Dr.-Riss-Special-Ellipsometer, software and linear-wiping-simulator

Fig. 1 Table of testing methods and apparatus for the simulation of particle release and cleaning efficiency in the wiping process, developed by the author

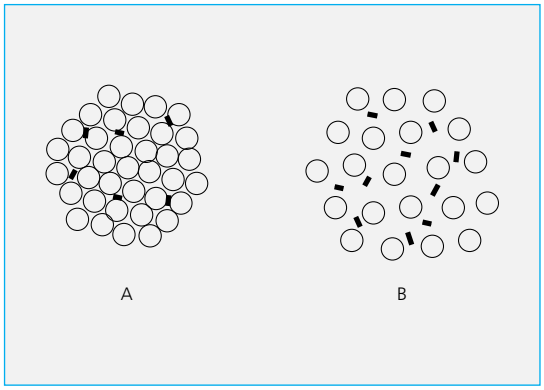

Fig. 2 Cross-section of a thread of high twist count or rather low texturing (A) and low twist count or high texturing (B). Here it becomes evident that the movability of the particles in a liquid (immersion method) is considerably larger with B than with A.

particle counter" (Fig. 11). From these „testing methods", of course, no meaningful results could be expected, not even if comparative testing were carried out by the same person. The tapping and moving energies were just too remote from the real stresses to lead to reproducible results.

In the beginning years of clean technology it was necessary first of all to develop testing methods for several clearly defined parameters of HiTech wipers that at least could give limited evidence about their quality. This was especially true for the features *particle release, ionic contamination* and *nonvolatile residue.*

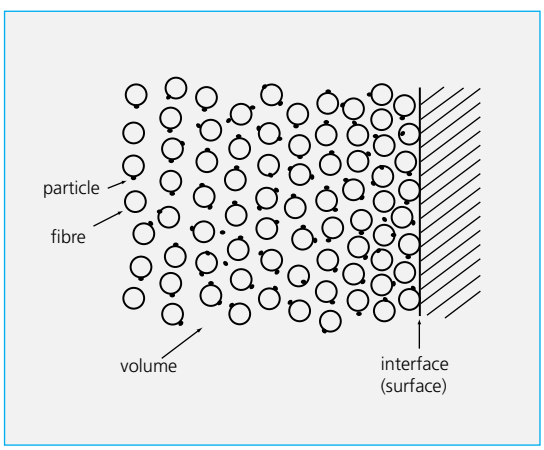

Fig. 3 Interface

The American Misconception and the lost Leadership

The testing methods by which a product can be comparatively tested and judged sometimes have a central significance for the optimal utilization of a products possibilities of application, for its technical lifespan, for the direction of its further technical development, and for its operational value as a product. This is especially so with the product *cleanroom wipers.* The U.S. cleanroom-wiper-manufacturers, which by the beginning of the 70s were worldwide the first to be on the market, historically follow another technical approach and have devised different methods for testing the key parameter *particle- and/or fibre-release* from cleanroom wipers than for example the European manufacturer Clear & Clean.

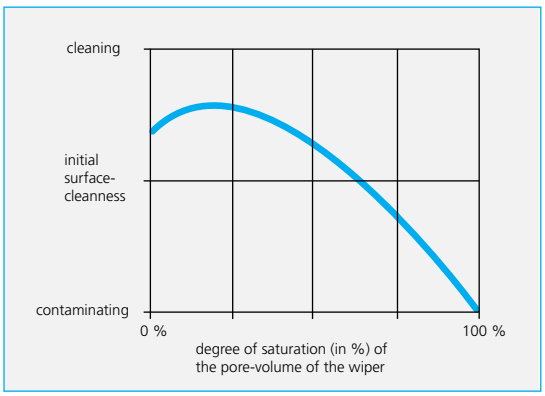

Fig. 4 Cleaning-efficiency of cleaning by wiping dependent on the degree of the wiper-saturation with a solvent

The American manufacturers are obviously of the opinion, that the best way to test particle release of a wiper is bringing the wiper into contact with deionized water in an immersion or pouring procedure. Thereafter, the particles released into the deionized water are counted and classified.

However, the author is of the opinion and that is part of the experience after many years of research:

- All testing methods by which the quality of a cleanroom-wiper is determined by recording the particles, fibres, ions or organica present in the structures of the wiper are false.

- No correlation exists between the wiper-content of such components and the residue of the same left on a surface which has been cleaned by wiping.

This applies in particular to the *„Minimal stress method“*, to the „biaxial shake“ method, and to the methods *IES-RP-CC-004-87T* and *RP-CC-004.2.* All of these methods were devised in the U.S.A. and published in part by a private institute (Institute of Environmental Science, U.S.A.). These methods are cited by popular U.S. manufacturers of cleanroom wipers in their catalogues as basis for the parameter *particle release* of their cleanroom wipers. At the same time they suggest in this way that the „better“ wiper is the one that releases fewer particles into the liquid while it is being immersed for testing.

The users of HiTech wipers, however, do not want to know how clean a wiper is. They want to know to which extent the cleanness of a surface improves after cleaning it with the wiper (cleaning efficiency).

If the parameter *cleaning efficiency* was e.g. not directly measurable, the user would may be satisfied with readings recording the *particle transfer* in a simulated cleaning process from the wiper onto a clean test surface like

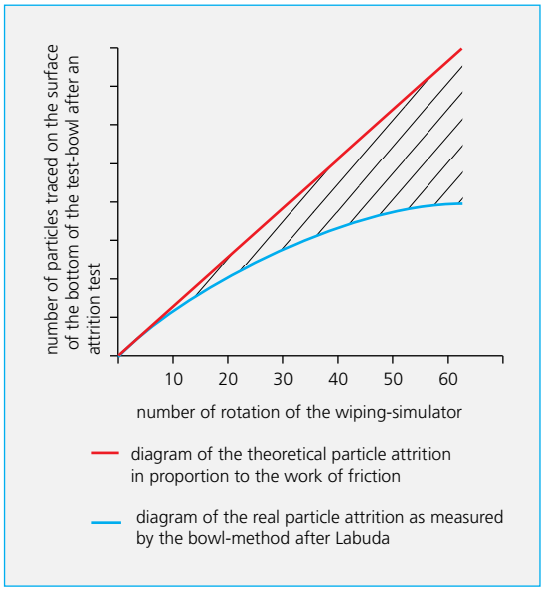

Fig. 5 Diagram of the recapturing effect of wipers

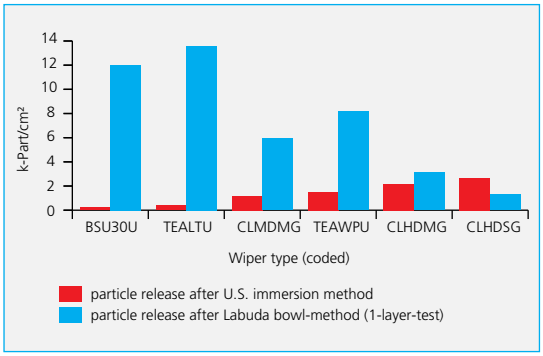

Fig. 6 Testing of the particle release of the knit-type cleanroom wipers after two different testing methods

is available by the *bowl-method* of the author (see page 12). Thereby, the surface roughness, the degree of moisture of the wiper and the resulting material-attrition, as well as the *recapturing behaviour* of the wiper enter considerably into the measurement reading. However, today in 1998 also the *cleaning efficiency* of a wiper can be directly measured e.g. by the ellipsometric method (see page15).

The basis of the criticism concerning all immersion and pouring methods is the following realization: A textile for the cleaning of surfaces by wiping is in its analogy a 3-dimensional storage system which during its application provides a 2-dimensional interface to the surface to be cleaned (see fig. 3).

The desired condition after a cleaning procedure is the absence of contamination on the cleaned surface. Looking at it in an inversed view it is the residue of contamination after a cleaning procedure which marks the success of it.

During every procedure of cleaning by wiping, in particular with the aid of a solvent, residue is built on the surface. The residue consists of

- a chemical part (grease from a mixture of oil, tenside, nonvolatile parts of the solvent and dissolved parts of the polymer-structures of the fibres and yarns)

- a particle and fragmented fibre part consisting of attrition from the surfaces of the wiper, loose particles and fragmented fibres attached to the wiper surface

The release of particles during the wiping procedure depends on a number of surface and material conditions, in particular, however, on:

- the degree of solvent-saturation of the wiper during the cleaning procedure
- the recapturing-efficiency of the wiper for particles and fragmented fibres (an effect of cleaning by wiping which was described by the author in 1989)
- the thread-density and fibre-diameter of the yarn used for producing a knit-type-wiper

In the system as described above there is only a single condition in which the number of particles contained in the volume of the wiper has an effect to the surface-cleanness after the cleaning procedure:

this is the condition of the *over-saturation* of the wiper with the solvent. In case the pore-volume of the wiper is more than 65 % filled with the solvent the fig. no. 4 shows the beginning of a marked reduction of the attainable surface-cleanness by a wiping procedure.

The *recapturing effect* of a wiper which the author could establish after the introduction of the bowl-method is the wipers propensity to capture particulate and other contaminants, which had lost their anchorage in the fibre-system, but are recaptured again by the wiper in the course of the same wiping procedure (see fig. no. 5).

The quantity of particles transferring from the surface of a knitted wiper into the surrounding liquid (e.g. deionized water) like is used as an analytical key factor by the U.S. test-methods is dependent on the *thread density (porosity)* of the yarn used to knit the wiper. This, on the other hand, is in direct proportion to the kind of *texturing (crimping)*, the *degree of texturing*, and the *mean diameter of the fibrils*.

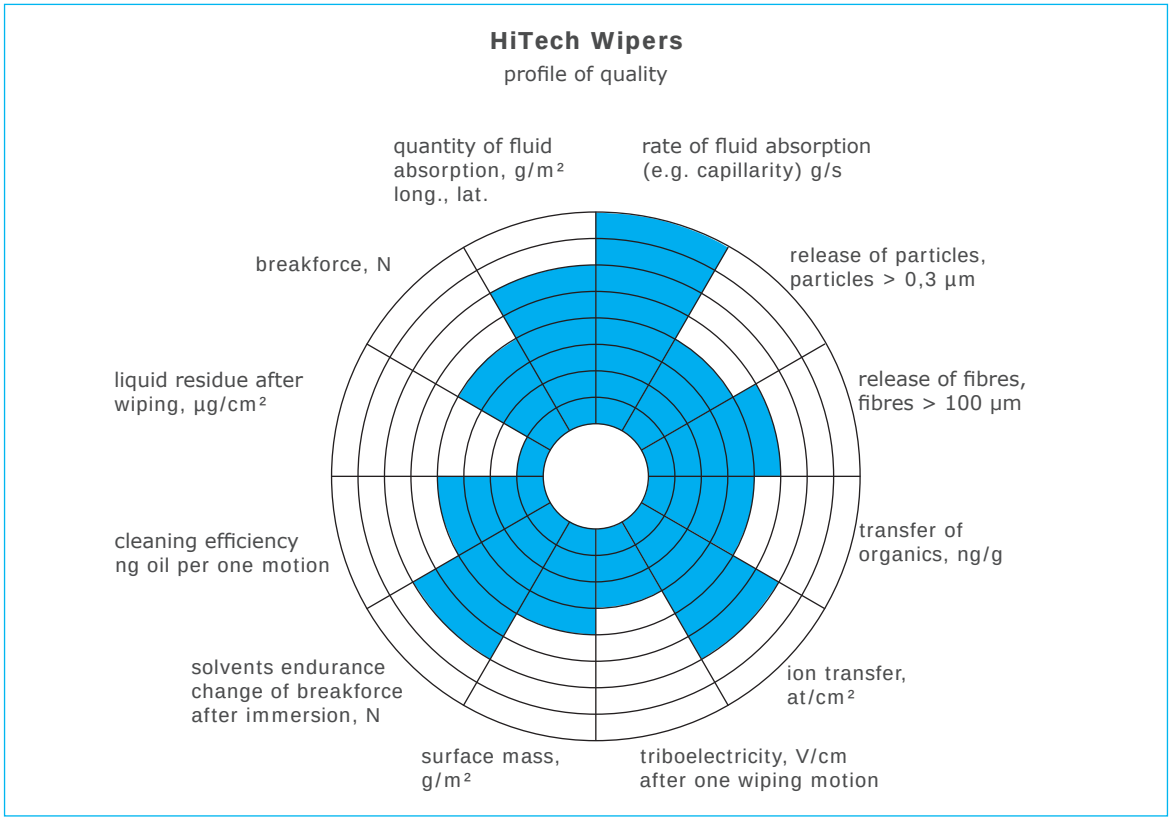

Fig. 7 The circle profile allows a quick comparative survey of the qualities of various HiTech wipers (here the example of a viscose-standard-wiper)

On knitted wipers which are made of threads with a high twist count, a small amount of texturing, and small thread diameters, the particle release into the liquid will be markedly less than on little-twisted and highly textured threads. The material attrition caused by a surface of mean roughness of e.g. 17 µm (Rz) will, however, be about the same for both thread types.

Thus, with the U.S. immersion methods there is a pretence that cleaning efficiency increases the higher the density of the thread and the lesser the texturing. *The attrition, however, is not at all simulated by the U.S. method, although a major cause for the release of particles during a wiping procedure. So it does not come as a surprise that immersion method and bowl method show in fact contradictory results (Fig. 6).*

The *bowl method* as described below promises to simulate the wiping operation substantially better (see page 12). But even this method is not ideal in the extent of simulating the wiping procedure, because it starts out from a clean surface and records the increasing contamination of it through the wiping process. Ideally one has to start with an already definedly soiled surface and record the surface cleanness before and after the wiping motion. This is now the case with the *ellipsometric cleaning efficiency test*, which the author introduced in 1998 (see page 15).

How does the difference in the chosen analysis method affect the long term product development in this case ?

If, for example, a HiTech wiper manufacturer assumes that the increase of the extent of the washing out of a wiper creates „better“ wipers, then he will concentrate his development on processes that make his wipers more and more clean. The goal is thus the wiper which releases zero particles into the DI-water-bath when being immersed. This product, however, seen for itself (statically) as ultraclean, in practice becomes more and more unclean upon wiping it (dynamically) over a surface, because naturally with each wiping-motion the previously described micro-attrition lessens its degree of cleanness.

Nearly twenty years have passed since the formulation of the first testing methods for the release of particles in 1980 to the ellipsometric profilometry for testing cleaning efficiency down to a few atomic layers in 1998. With each further testing method the Clear & Clean research-lab had introduced, however, our knowledge of the physics of cleaning by wiping had increased and at the same time we had taken a further small piece of the American lead in the area of HiTech wiping materials.

Sometime around the introduction of the bowl method we thought we are now a step ahead. The American leadership had become a quantitative aspect rather than a technological one. Of course we are very happy to claim leadership for Europe in a technology originally based in the United States.

The development of several testing methods by the author and the Clear & Clean Research Laboratory is described below in a historical context.

1978 - The first Experiments

At the beginning of my clean technical research in 1978 I had only a vague idea of what I wanted to do. However, I bought a huge microscope from the Zeiss Company. It was so heavy that two men had to carry it and it was wickedly expensive for my circumstances at that time. The microscope is called *Ultraphot 2*, and one can do almost anything with it, even interference-contrast-depictions after Nomarski with particle diameters of 0,2 µm. I had the intense and irrevocable desire - and that is true even until today - to know more about wiping processes than any other person in the world. At first I tested the macroscopic phenomena of all wiping products then available industrially. Soon I found out who the manufacturers of raw materials were and which ones utilized nonwoven or knitting techniques. Then I began weighting down wiper samples with a metal weight and dragging this experimental arrangement over flatly stretched-out sandpaper of the grain size 400. I counted the fiber fragments and particles microscopically and discovered in this way how many particles and fibres of the different kinds of wiping materials were left on the rough surface upon wiping. Then I tried the whole experiment once again with moist wipers and with some that

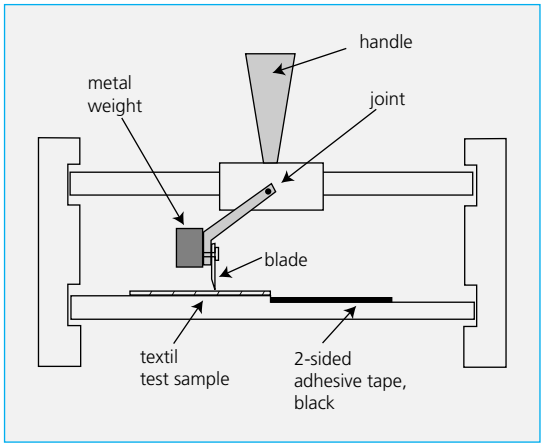

Fig. 8 Scrape Testing Device of 1980

had been immersed in alcohol for a time. The particle size that I was looking for then lay in the range of 20 - 100 µm. For me, these tests were the most instructive experiments that I have made until today with textile surfaces. At that time it was clear to me that the wiper residue which remains on a cleaned surface is not dependent on any parameter as much as *surface roughness*. But these experiments were extremely time-consuming, particularly because with textile materials one has to make a great number of evaluations in order to reach a useable mean.

1980 - Scraping Abrasion

At this time textile sleeves for cleaning the read/write heads of computer disk drives were responsible for the largest turnover of the HiTech wiper industry. The sleeves were made out of cotton and were pulled over a fork-shaped sleeve-holder. In order to clean the surface of the disk drives, the sleeve was saturated with alcohol and pressed lightly onto the rotating disk, which then had a diameter of about 14 inches. The read/write heads were also cleaned by wiping the alcohol-saturated sleeve over their surfaces. These heads had very sharp edges. Therefore a clearly increased attrition of fibres and particles occurred upon wiping with many of the products with inadequate fabrics. The attrition damaged the faultless functioning of the disk memory.

A (somewhat imperfect) scrape testing device (Fig. 8), which could be moved horizontally, soon developed out of an old tile-cutting machine. Below the handle to turn it on, a free-hanging steel blade was attached and weighted down with a weight fitting the purpose. It then scraped across the cotton sample. Some of the fibre fragments and particles which were scraped off fell onto a black-dyed adhesive film, which was fastened behind the test sample. The number and length of the scraped-off fibers were evaluated under the light microscope and provided a certain measure for the abrasion strength of the textile material.

1986 - The Labuda-Hermans-Probe

At the time of the construction of the first large cleanroom at Siemens AG in Regensburg the Clear & Clean Laboratory consisted of a cleanbench, our famous Zeiss microscope and an electronic air-particle counter. The task then was to test the surface cleanness of disposable gloves made out of polyethelene, which were used in the cleanroom in large quantities. Lodevicus Hermans and I developed the concept of a surface probe for this purpose. We assumed that particles could be removed from the surface with the assistance of an air stream flowing over it. The first model of such a probe consisted of a pipe which had a suction plate on the underside. In the middle of this suction plate there was a drill-hole (Fig. 12). Four tunnel-like canals were cut into the board surface which ran from the outer edge to the inner drill-hole. At the other end of the pipe a hose extension was attached. The hose led to an electronic particle counting device which sucked in 0,028 m3 of air per minute. The particles which were inside this volume of air were counted automatically and classified according to their Feret-diameter. When ever we put this probe e.g. onto the surface of the glove or a cleanroom paper we could, in fact, establish the existence of particles. In the first probe that we constructed the four canals were cut only a few millimeters long. Then Lodevicus Hermans had the idea to considerably lengthen the air stream above the surface to be tested in order to increase accordingly the number of the particles removed from the surface. We built the second „Labuda-Hermans Probe“ with a spiral-shaped canal cut into the head of the probe so that the path of the air stream over the experimental surface would be substantially longer and the area affected by the air stream correspondingly larger.

At first we were enthusiastic about the results of our testing. No matter on which flat surface we put the probe, the counter always showed us the existence of particles. But soon we noticed that the counter analysis of the same surfaces analysed with particle counters *for liquid media* always gave many more and especially many more small particles as a result than with our probe. In addition, we observed that the test results which we gained with our probe were dependent on the kind of materials on whose surface we carried out the tests, on the microroughness of it, and also on the relative humidity of the test environment. Obviously we had not taken enough into consideration the laws of particle adhesion on surfaces in flowing gases. We gave up this project not without disappointment, because we wanted to build a *measuring instrument* in the physical sense and not a *detector.*

Several years later - we had forgotten the probe long ago - an instrument appeared on the American market which took up the principle of our probe, but in addition it blows clean air from several jets inside the probe directed at the surface to be analyzed - with the goal of removing even more particles from it and making them accessible for counting. This device is, in principle, even if the utilization purpose has turned out to be rather large, a very useful surface particle detector. The problem with it is that the maker equipped it with a digital outlet, which by means of a printer and printing programme of high quality neatly prints out the particle data even classified by size. These, of course, do not correspond even approximately to the quantity of particles on the analyzed surface. It is an interesting case here of „cheerful engineering“. At any rate, after the appearance of this device, young engineers, but also even experienced analysts began interpreting data received on such a basis. A young technician in Munich even implemented a comprehensive series of tests on the surfaces of cleanroom wipers made by different manufacturers (against the authors advice and unhindered by his superior). The questionable goal of these tests was to collect comparative data on particle release during the wiping process and thus find the „best“ wiper of all.

In 1996 at an ICCCS convention in the Hague where the American representative of the instrument-maker was present, two colleagues from Applied Materials, Dr. K.J. Hansen and Dr. H.D. Pham, gave a lecture held in a positive tenor about the utilization possibilities of this device. However, unfortunately they did not go into the fundamental physical aspects of the particle release from the analyzed surface through a blowing stream and the particle collection from gases in a turbulent state. Thus the analytical limitations of the device were not mentioned.
After it became known that the counter was used in several factories, a German company rebuilt the device, again with a digital printer and particle data classified by size.

1987 - The Ball-Hammer Testing Method

In 1985 the VDI - Association of German Engineers invited me to work on forming national guidelines for cleanroom technology, and asked me in a letter to develop a testing method with the assistance of which the particle generation on cleanroom wipers could be tested comparatively, which up to that time was not possible.

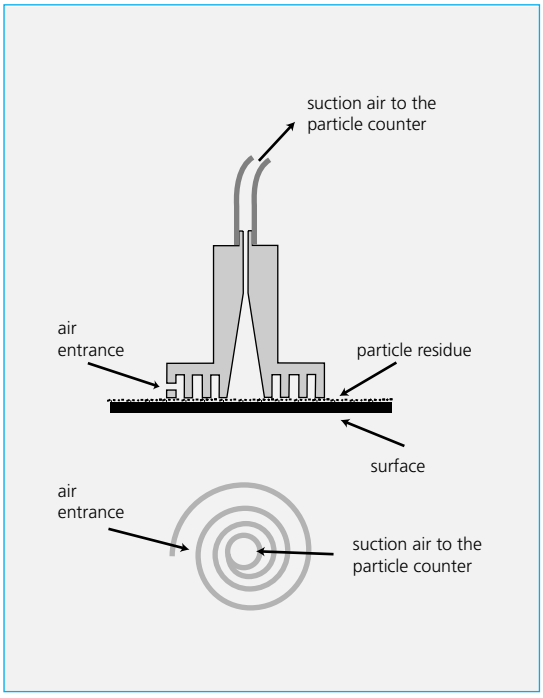

Abb. 9 Labuda-Hermans-Probe

Fig. 10 circa 1985: Moving over a probe

Fig. 11 circa 1985: Tapping over a probe

Fig. 12 *Labuda-Hermans-Probe, first version*

At this time two testing methods to record particle generation were known in the U.S.A., which Europeans took over without criticism, as we often do. It concerned the so-called *„Gelboflex" method* and an *immersion method* which was developed in the environs of an IBM laboratory. In the flex method a wiper in a closed box of acryllic glass was exposed to a cyclical pull/twist strain while clean air was lead past the test sample to a particle counter and analyzed. The immersion method was referred to in detail on pages 4 and 5.

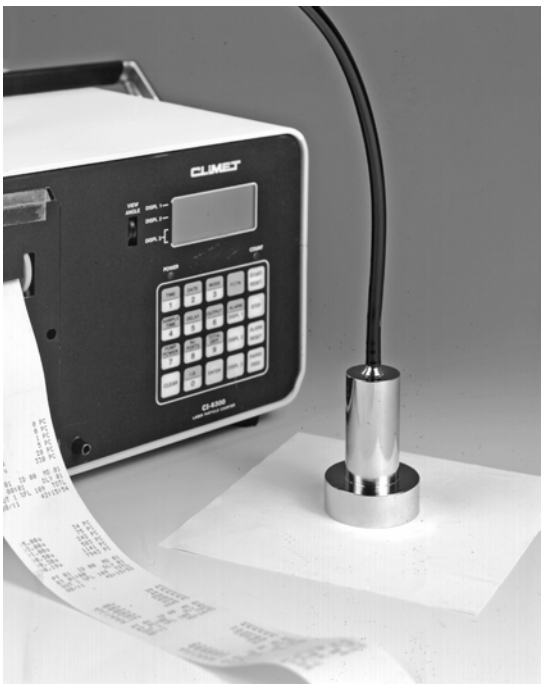

Fig. 13 *Labuda-Hermans-Probe, second version*

With the assistance of the ball-hammer testing device then introduced by the author (Fig. 15), it was possible to record particle residue on the surfaces of objects such as wovens, nonwovens, gloves, foils, and papers. Higher particle values were achieved with it than with other dry measuring methods, e.g. the Gelboflex method mostly used in the U.S.A. In constructing the device we started from several fundamental considerations:

In the above testing methods the agitation energy to simulate the handling stress of a wiper was applied to the *whole surface* of the test sample, (e.g. wiper) of about 200 x 200 mm, while only a fraction of the flow of released particles from the test sample could find its way to the particle counter (flex method). The effective circle-shaped area, inside which a particle stream is measurable by means of a 30 mm Ø isokinetic probe of an air particle counter, has a diameter of about 40 to 45 mm. If one wants to achieve optimal readings, we thought, the mechanical agitation of a test sample would have to be limited to a circle of about 45 mm diameter. It should furthermore be placed closely above the probe. In order to avoid measuring errors through a lateral shaving-off of fibres the test sample would have to be tightly locked into a special holding device before and during the testing

to prevent side-shifting. In addition, it would have to be able to be put in and taken out of the holding device without a great amount of effort. During the testing no flow of air should occur through the wiper. This is to eliminate errors which have been known to arise from air suction and to apply the testing to object surfaces (foil gloves, paper, synthetic foils), through which no flow of air is possible. The testing should be carried out without consulting specially trained personnel.

In essence, the experimental apparatus consists of two horizontally arranged platforms of which one is vertically movable. The test sample can be firmly clamped between the platforms. In the middle of both platforms there are circular openings, so that the test sample can be stretched like a membrane but hangs freely. In order to ensure an approximately constant material tension, one of the platforms is equipped with a conical ring. In the stretching process this ring presses the test sample against an elastic ring on the opposite platform and exercises therewith a peripheral area tractive power. A hammer mill is placed above the platforms. It lets a pile-driver with a ball-head drop down on the test sample 30 or 60 times a minute in a free fall. Through the impact of the ball-head on the test sample particles are released from the test sample. They are sucked into the air stream of the probe and can be lead to the particle counting process. In order to avoid jumping impact on the sample, the hammer mill is mechanically caught immediately after the first impact until the next one.

An isokinetic probe is centered at a small distance below the platforms. It is connected by a hose with a counter for air-borne particles down to a size of up to 0,19 µm and a starting quantity of 1 cubic-ft/min (e.g. Climet 6300). A ventilation-cylinder is placed around the isokinetic probe, so that no air suction could occur through the sample. The ventilation cylinder, which is open down below, also has the function of shielding against the particles flying down from above which could interfere with the test results.

This apparatus was utilized by several companies, among others Siemens in Villach, for a long time in order to record the washing condition of cleanroom textiles. In addtion, it was used in a comprehensive project of the Textile Institute in Denkendorf to measure the particle disposition of reusable wipers. [Ref. 18]

1990 - The Colander Method

As explained before, a great part of the particles, fibres and fibre fragments generated by wiping processes are caused by the *scouring friction* between the wiper and the surface to be cleaned. In this area is also the basis for working out a further testing method which is founded on physical forces that have an essential effect here and that can be reproduced. This effect remains unconsidered both with the wet testing methods DIN-50452, ASTM-F312 and all of the other liquid testing methods which describe the particle generation by immersing the wiper into a liquid medium with subsequent particle counting by the liquid particle counter, and also with the ball-hammer testing method after Labuda. A testing method needed to be developed with the assistance of which the scouring friction could be represented and numerically recorded. The Labuda Colander Method, presented in 1990 at the ICCCS Conference in Zürich, promised to fulfill these requirements.

Fig. 14 Ball-Hammer Testing Device after Labuda

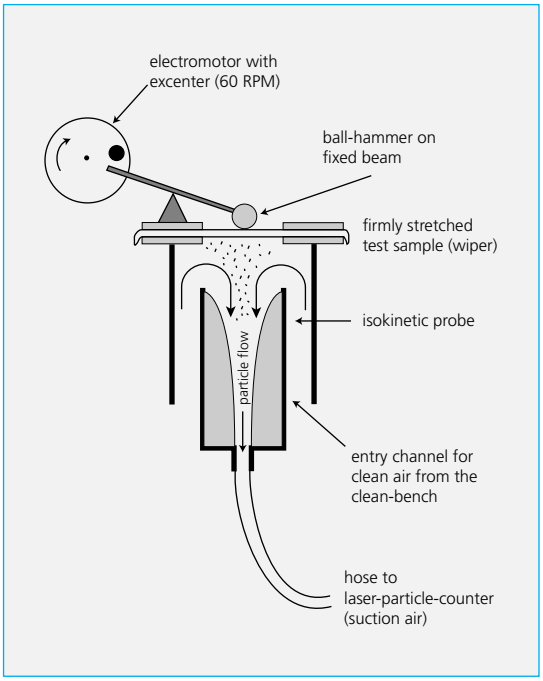

Fig. 15 Ball-Hammer Testing Device after Labuda to record particle residue on surfaces

In October 1997 Mr. Steve Paley et al. (Texwipe U.S.A.) surprisingly presented the Labuda-Colander-Method from 1990 as a new invention (as usual without bibliographical reference) in the American periodical MIKRO. The only technical difference: linear instead of rotational movement of the test sample.

The Labuda-Colander-Method functions as follows (Fig. 16):
A circular cutting of a wiping material is fastened to the underside of a cylindrical metal body of a defined weight. This combination is put onto a metal colander, which has been chosen for its technical features, is taut, and has been previously cleaned, and there it is rotated one minute long at 50 rpm. Beneath the colander the isokinetic probe of a particle counter for air-borne particles is fastened. In this way the following particles will be recorded:

1- The particles which have been generated by the scouring friction of the object surface, which have gone through the openings of the colander, and which reach the probe in this way.

2- Such particles which lie in the size-recording margin of the particle counter. With a laser particle counter, for example, which is capable of counting the sizes 0,19 to ca 10µm, all of the particles which are smaller than 0,19µm are not counted, and all of the particles over 10µm are not selectively recorded. That is one of the greatest problems with particle counters. There are particles generated in the submicro-spectrum and at the same time fibre fragments in the millimeter spectrum. The essential disadvantage of this method is that a certain quantity of rubbed-off particles stick to the colander and therewith are not available for measurement. This is especially true for small particles.

1992 - The Bowl Methods I and II

This modern testing method aids the registration of particle and fibre generation of textile materials both in a dry and wet state through controlled abrasion over surfaces of a defined roughness. The example for application is the recording of particle generation upon utilization of cleanroom wipers. The method is mentioned as the „Bowl Method after Labuda“ in DIN/VDI 2083 Page 4 - Paragraph 7.6.5.

It was developed in the framework of the authors collaboration in the VDI guideline committee for cleanroom technology and presented publicly at the Conference of the German Association of Engineers in Stuttgart in 1993 in the framework of the lecture „A Strain Diagram for Cleanroom Wipers“. This lecture was published in the VDI-report Nr.1095 in 1994.

The method works as follows (Fig. 19):
A circular sample having the diameter of 60 mm is cut out of a wiper. Shortly before the beginning of the experiment the sample is soaked with deionized water to 75 % of its full water absorption capability and mechanically fastened in a *rotative-wiping-simulator* with electric drive and electronic control. The fastening ensues by means of a magnetic ring clamp under a cylindrical rotor of known mass (600p). This arrangement is rotated 250 times in a bowl made out of V2A-steel with the assistance of a flexibly coupled electromotoric drive. Thus

particle and fibre abrasion occur, just as they do while working with wipers. Then the bowl is filled with deionized water, which then contains the rubbed off particles and fibre fragments. The deionized water which has thus accumulated the particles and fibres can be analyzed by means of the following methods.

A - with the assistance of an automatic particle counter for liquids according to the number and the size of the particles. This method simulates the wiping-cleaning operation quite practically: A wiper is also in practice utilized by moving it with a certain pressure across a surface, which has a surface roughness of Rz > 0.

B - by filtering the particle-loaded deionized water and the microscopic evaluation of the filter.

Measurements of the average pressure used by people for cleaning surfaces resulted in values around 600 Pond. The experiment can be implemented with either a dry or a moist test sample. In the test between moist or dry wipers made out of viscose there were substantial differences in particle generation. If in the experiment sample bowls are used which are subsequently graduated in their roughness, then one can make a material-specific strain diagram of the particle generation of wipers depending on the roughness of the wiped surface. In addition, interesting conclusions can be drawn about the abrasion-behaviour of wipers on surfaces with breaks - namely whenever the testing bowls are equipped with grooves or depressions or with reliefs. The floors of the testing bowls were equipped with the following surface-roughness: (DIN-Rz) = 5, 17, 33, and 39.

In 1998 we added an essential parameter to this method. We set the bowl onto a very sensitive torque transmitter and can now gain valuable insight into which fibre and thread constructions, degrees of moisture, equipment and surface roughness bring about the highest and the lowest torque friction and how this corresponds to the quantity of generated particles. Also the bowl can be furnished with a defined particle-layer by a particle generator and the cleaning efficiency of different wipers for the particle contamination can be determined.

1996 - The Part-Lift Method

While the problem of recording the number of particles in liquids or gases has been solved satisfactorily, there is a considerable deficit of methods which serve to identify and count particle and fibre fragments on even surfaces. With the aid of the particle collector, which was presented by the author at the ICCCS Conference in the Hague in 1996, it is possible to gain a quick survey of the number of particles and fibre fragments which are brought by contact transfer from any smooth surface to an adhesive collector plate (Lift Method). In addition, by utilizing electronic image analysis it is possible to carry out an automatic count and classification of the particles, from relatively small sizes up to several millimeters, depending on which magnification scale was used. Within a few minutes it is possible to gather almost complete information about the spectrum of particle residue on an even surface.

The two-part collector consists of a closeable metal capsule. In the centre, on the base of the capsule, a cylindrical spring is attached.

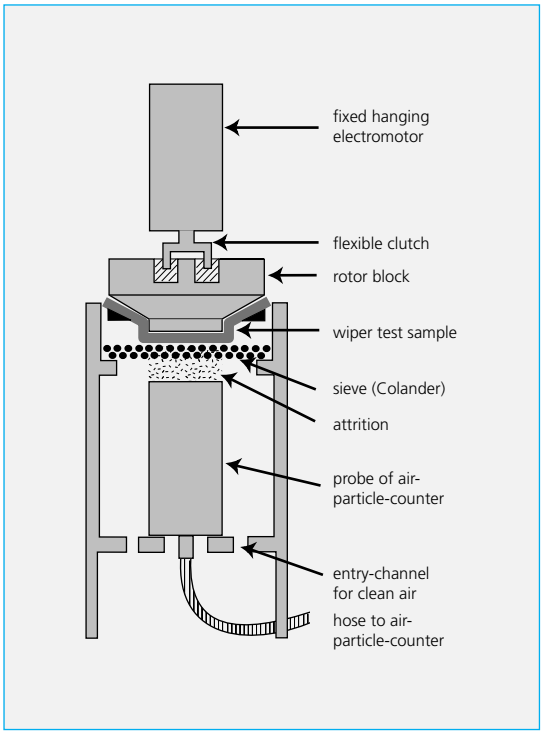

Fig. 16 Colander

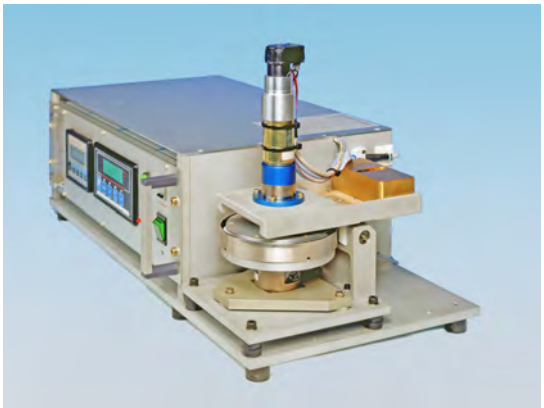

Fig. 17 Rotative-wiping-simulator, closed

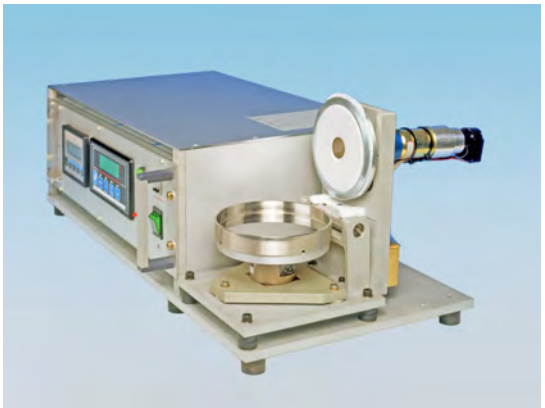

Fig. 18 Rotative wiping-simulator, open

Whenever the bottom of the capsule with the collector plate is pressed against a dry, comparatively flat test surface for at least 5 seconds, the particles, fibres, fibre fragments and microbes lying loosely on the surface adhere to the collector plate. The upper part of the collector plate rises several millimeters above the thread or rather the contact plane, just enough to ensure that the effective pressure force on the test surface is 5...6 Newton. After the completion of the collector process, the upper part of the capsule can be screwed on, and the particle sample thus secured is ready for analysis in a laboratory. After the collector lifts off the test surface, a great part of the particles existing on the surface should be on the collector plate. How great the percentage may be depends on the quality of the analyzed surfaces. The Institute for Process and Aerosol-Measuring Technology of the University of Duisburg has tested the efficiency of a collector in removing a polished silicon chip (wafer) and given the result as > 90 %.

In the first field experiments with the particle collector it turned out that the surface roughness of the collector plate is a little too high, so that for reasons of depth of focus particles < 5 µm will not be recorded with one hundred per cent certainty.

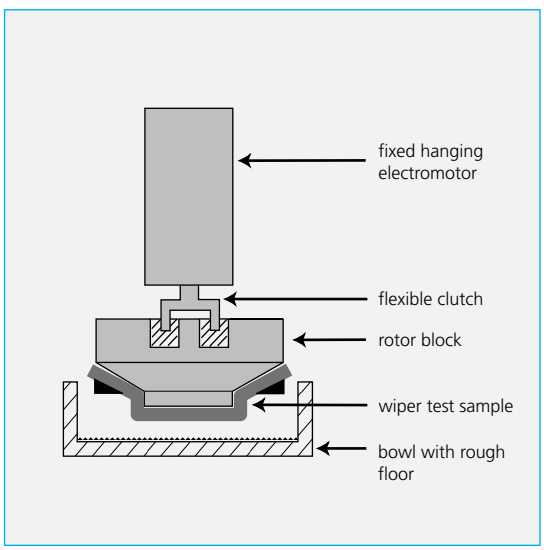

Fig. 19 Bowl Method I

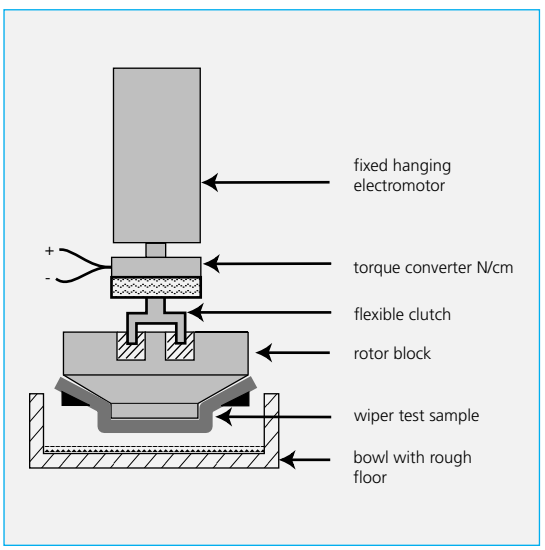

Fig. 20 Bowl Method II

In addition, there is the problem of the small optical surface of the image when recording the smallest particles microscopically. If one assumes that the optical registering of objects up to a size of 5% of the picture diagonal is possible, then with an assumed particle diameter of 1 µm a (square) picture field of only 14,2 x 14,2 µm (=201 µm²) results. Applied to the whole collector surface of 1 cm² there are thus about 5000 observation fields for a 100 % evaluation of the collector plate. By particle sizes of 10 µm the number of observation fields is reduced to tenable 50, whereby e.g. 20 would have to be evaluated in order to ensure a justifiable reliability of the report. With the assistance of modern image analysis this is possible without any problems. Through the light scattering which results from the halogen light directed from the side, the particles are depicted considerably larger than they are in reality (Halo), so that one can assume that the method is proper for an evaluation of particles >5µm. The collectors manufactured up to now have a metal casing and are designed to be reusable. It has turned out, however, that the possibility of cleaning the collector plate cannot be fully ensured. Therefore the collector will be manufactured as a disposable part in the future.

1998 - The Ellipsometer-Method

This method is based on the fact that the user wants to know how the surface cleanness has increased as a result of the cleaning-procedure by wiping in comparison to the initial surface condition.

A testing method needed to be developed which measured the cleaning efficiency of a wiper for different kinds of standardized contaminants of extremely small mass. These could be e.g. pasty contaminants like grease, oils or pastes. They could also be microbes, resin residue, or particles. In order to measure the efficiency of a cleaning process it must be simulated very exactly. The linear-wiping-simulator (Fig. 25) was developed for this purpose.

The testing method works as follows:
On a horizontally arranged sled-apparatus (linear-wiping simulator) four glass-plates measuring 25 x 75 mm are mounted successively (see Fig. 29). The second plate is covered with a thin layer of a standardized contaminant, e.g. a middle-viscous oil. The mass of the applied film is weighed by a µg-scale. The homogeneity of the layer thickness is registered profilometrically with the aid of an ellipsometer. The ellipsometer can ideally measure substance film thicknesses down to one atomic layer.

On the first plate of the linear wiping simulator there is a metal weight with the mass of 500 g. Under its supporting surface a wiper cutting of 20 x 70 mm is being attached. The sled is moved in the direction of the fourth plate with a given speed. While gliding over the second plate the wiper cutting takes up part of the contaminating mass (oil film). While gliding over the third plate the wiper cutting may transfer some of the contaminating mass present in it to the surface. Both mass differences can be measured gravitometrically. The metal weight with the wiper cutting comes to a stop on the fourth plate. In this way the testing method gives insight into the capability of a wiper to remove grease layers from contaminated surfaces to a precision which was not attainable up to now, indeed even to several nanometers of film thickness. The same could be done with particles etc.

1. The figure 26 shows the initial contamination of a glass surface with a low-viscous oil film of 83,3 nm mean thickness. In

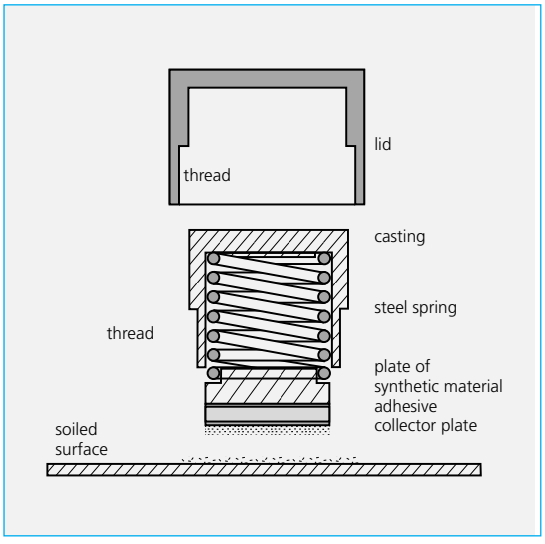

Abb. 21 Part-Lift method after Labuda

total, 256 periodical individual places were measured ellipsometrically on a surface of X = 10 and Y = 6 mm and the result was min. 42,8 nm and max. 119,9 nm. For the following diagrams a Riss-Ellipsometer was used. Through this technique it is now possible for the first time to reliably measure cleaning efficiencies of wipers in units as small as nanometers.

2. After a single wiping operation with the microfibre-knit MICROWEB™ the mean thickness of the original contamination was reduced from 83,3 to 31,8 nm (min. 10,3 nm - max. 102,8 nm) (Fig. 27). That corresponds to a cleaning efficiency of 61,8 % with the first wiping.

3. After five further wiping movements with the MICROWEB™, the remaining surface contamination was reduced to 3,7 nm (min, 2,51 - max. 8,7 nm). That corresponds to only about 74 atomic layers and is already considered „ultra-clean“ in the meaning of cleanness standards in technical work processes (Fig. 28).

In the course of the years even other parameters of cleaning by wiping were researched besides the parameters particle generation and cleaning efficiency. They are in particular:

- triboelectricity in the wiping process
- the liquid-absorption of cleanroom wipers
- liquid residue after moist wiping
- the transfer of ionic contaminants

Fig. 22 Riss Ellipsometer

The Measurement of Triboelectricity

In order to research this parameter for cleanroom wipers in the proper way we applied a testing system which *Dr. Peter Ehrler* impressed upon us at that time. It was the *drop slide after Ehrler* (Fig. 30). This apparatus functions as follows:

The drop slide after Ehrler

Description

The drop slide after Ehrler consists of - because of the small electrical chargeability of the material wood - a vertically constructed wooden frame, in which there is a vertically run drop slide (4), also made of wood. Tightly attached to the drop slide are two polystyrol rods (3) A and B with a diameter of 12 mm. In its initial position the drop slide is locked in the top part of the wooden frame. Upon operation it can be electrically released and falls down then on the collision cushion (6). The wiper or paper to be analyzed (2) is put into a grounded clamp which is on the top of the wooden frame. Afterwards, the wiper is carefully placed around the polystyrol rods, without causing friction which could produce undesirable electric charges. On the free end of the wiper a weight (7) is clamped which assures close contact between the wiper and the two polystyrol rods, only with the aid of gravitational forces. After the test sample has been placed into the drop slide, and the field meter and the subsequent instruments have been switched on, the actual experiment begins.

Implementing the Tests

From every wiper and paper usually five test samples 50 x 300 mm were cut off and put in a temperature and humidity chamber at 40% relH and + 22° C. After that, still in this test climate, the samples were put one after the other in the drop slide, charged, and measured. The drop slide was also in the temperature and humidity chamber. The spontaneous charges and the subsequent decay times were registered on the oscillograph. The oscillograms were evaluated, and the data thus received were recorded in a table. In this way a survey was written over the possible electrostatic chargeability of both cleanroom wipers and cleanroom paper products of the various manufacturers under the exact usual humidity conditions in cleanrooms.

Fig. 23 Microscale

Fig. 24 Ellipsometer, Precision-standing-motors in the nm-spectrum

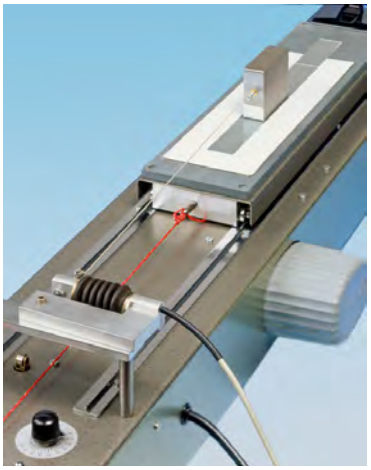

Fig. 25 Linear-wiping-simulator

The charge diagrams thus received (Figures 31 and 32) show both the charge level in kV and the decay time. In 1997 the author published an essay which dealt extensively with the topic of triboelectricity in the framework of a VDI-conference in Fulda (Ref. 12).

The Absorption of Liquids by Cleanroom Wipers

Wipers are usually wetted with a cleaning fluid before the cleaning operation. The process of saturation usually occurs in practice in such a way, that the wiper is held in the left hand of the operator, and with the right hand he pushes the handle pump of the spray bottle filled with cleaning fluid, so that the wiper is wetted in one or two places. According to the general cleanroom-regulations, the wiper is folded twice (thus four layers over another) and is normally in the left hand. Upon wetting it, the liquid is distributed into the layers lying over each other and towards the edges as well. In order to attain a sufficient and above all a *sufficiently rapid* saturation of the wiper, one must constructively ensure that

- the wiper can absorb a sufficient amount of liquid
- the amount of liquid can be distributed so quickly that a small handling time is ensured

The same is true for the absorption of splashes and the spilled remains of liquids (Spill-Control). The liquid is taken up by the wiper surface (lateral) and spreads after that in the longitudinal direction. There is a testing method for that which works on the basis of the *falling water drop*. It records the distribution of the drop-volume into the wiper capillaries opto-electronically. A problem with this testing

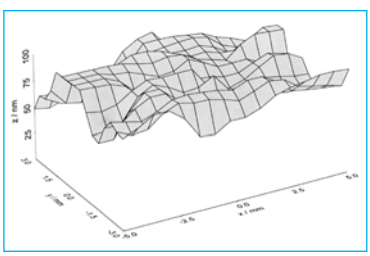

Fig. 26 Initial contamination

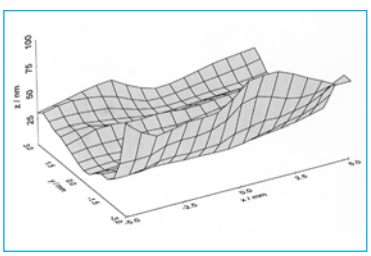

Fig. 27 Contamination after wiping

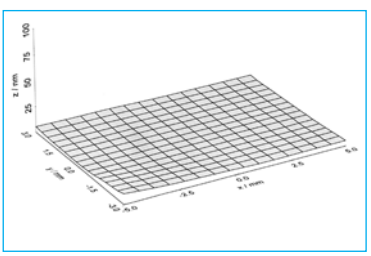

Fig. 28 *Contamination after wiping 5 x*

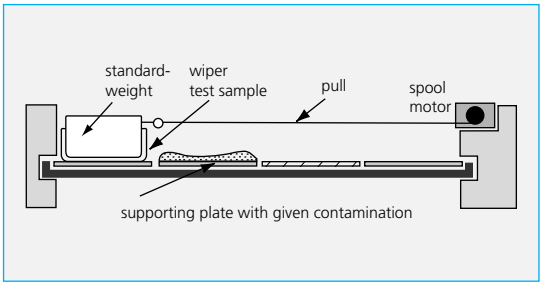

Fig. 29 Linear-wiping-simulator

method, however, is generally the volume-inaccuracy by the production of drops. We at Clear & Clean have therefore abandoned the absorption of liquids in the wiper surface and measure as a substitute up till now the longitudinal *capillarity* of the textile material.

The testing is carried out as follows: a receptacle filled with a defined quantity of water is put on the weighing scale and a test strip of the wiping material is dipped with one end into the liquid (Fig. 31). The mass of the liquid taken out of the receptacle is measured over time. Thus a diagram of high material specificity and meaningfulness is made.

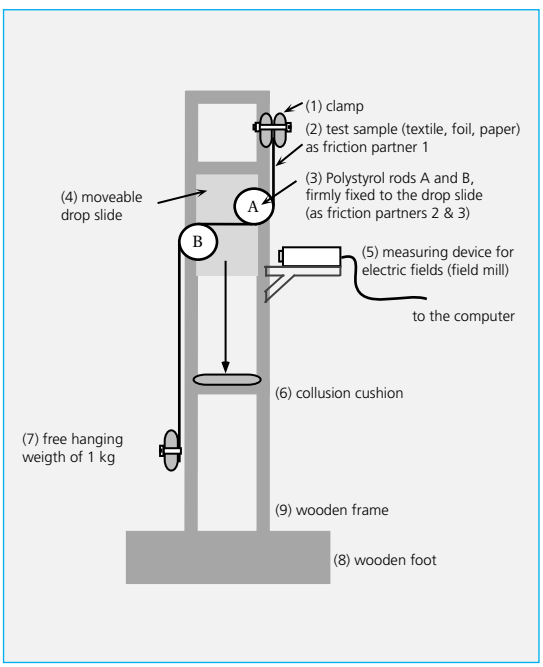

Fig. 30 Drop slide after Ehrler to measure the triboelectricity of porous surfaces (scheme)

Sven Siegmann from the Clear & Clean Laboratory introduced a new testing method recently with the aid of which it is possible to measure not only the *longitudinal* but also the *lateral* capillarity of a wiper folded into several layers. The sketch below (Fig. 34) explains the process. Through the introduction of this testing method it is assured that the testing simulates the practice.

Liquid Residue after moist wiping

This deals with the unavoidable liquid residue which remains when a surface is wiped with a moist wiper. Depending on the liquid content of the wiper, but also on the composition and structure of its basic materials and their surface-energies, the quantity of the liquid residue remaining on the surface fluctuates to a considerable extent. There are two aspects of it which have a special significance for the cleanroom wiper:

- In liquid residue there are comparatively large amounts of particles which remain on the dry surface after the evaporation of the liquid and which can get into the surrounding area.

- The liquid residue has a great influence on the time needed for a wiping procedure. Observations of wiping procedures show that a great portion of the persons tested only finish a wiping procedure when the surface is really dry. The dry-wiping capability of a wiper is thus of central commercial significance (time-costs) in a great number of wiping procedures which take place in cleanrooms.

The author does not know of any method with which this parameter could be measured rapidly and with a high degree of simulation. In the Clear & Clean Research-Laboratory we have tried out various measuring methods without achieving the desired results:

Method 1: Applying a given quantity of liquid onto a surface/ Laying the wiper over it/ Weighting this down with a metal weight/ Lifting of the metal weight and wiper/ Measuring the remaining quantity of liquid

The *disadvantage* of Method 1 is that the wiping movement which distributes the liquid

onto the surface is not included in the measurement.

Method 2: Moving the wiping simulator with the moist test sample over the surface of a representative material and the subsequent weighing of the difference of the material's weight.

The *disadvantage* of Method 2 is that with the small quantities of liquid remaining on the surface, the evaporation of the liquid causes a great inaccuracy. In addition, the size of the support depends on the receptive area inside the casing of a microscale.

The Transfer of ionic Components of the Wiper

Because the attrition of wipers on test surfaces of a given roughness often only lies in the microgram spectrum it is not easy to extract such small masses of the ions present in and on it. A method which seems suitable for it is the VPD (Vapor Phase Deposition) with subsequent TXRF (Total-X-ray-Reflection-Fluorescence) or AAS (Atomic-Absorption-Spectometry). With the Vapor-Phase-Deposition a silicon surface is vaporized with acid and all oxide layers are dissolved. This ultraclean surface is then wiped with a sample wiper five times. The wiping can take place with a dry and then alternately with a solvent-soaked wiper. The silicon surface is scanned off with a drop of deionized water. The ionic residue present on the surface is concentrated in this way in the water-drop. The drop is then analyzed by TXRF. With this method measuring sensitivities of up to 1010 atoms/cm^2 can be reached. The disadvantage of the TXRF is that very light elements such as e.g. Sodium cannot be measured. This can be achieved with the assistance of the AAS-Method after dividing the drop. In the first experiments with the VPD-TXRF-Method six cuttings 4x4 cm of various cleanroom wipers were laid between two ultrapure wafers and weighted down with a weight of 1000 g for 24 hours. The readings afterwards gave the following transfer data.

K	-	48,8x10^{10} At pro cm2
Ca	-	7,5
Ti	-	2,3
Fe	-	6,7
Ni	-	0,5
Zn	-	14,6

Thus the values received are extremely small. Whether the parameter ‚ionic contamination' can be completely forgotten in the future, however, must be shown by further VPD-TXRF testing on test surfaces of higher roughness.

The method has the disadvantage, that it requires very expensive equipment and can only be implemented by specially trained personnel. A further method with the aid of polarographic Voltammetry is presently being worked out in the Clear & Clean Research-Laboratory. There wafers contaminated during the wiping process are put into deionized water, and the ions which transfer into the water are determined either polarographically

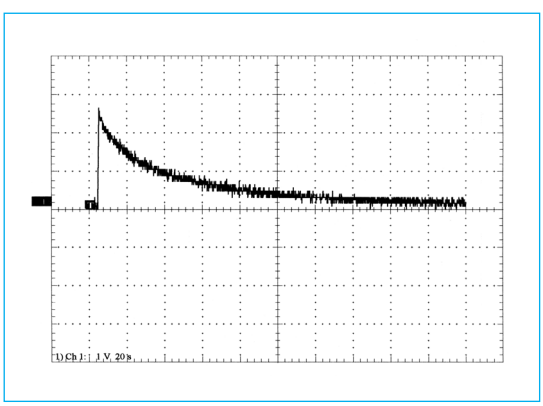

Fig. 31 Charge diagram of a cleanroom wiper in a dry state (Method: Drop Slide after Ehrler)

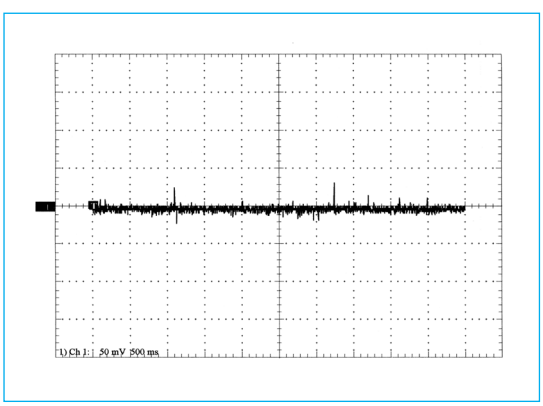

Fig. 32 Charge diagram of the same cleanroom wiper in a wet state (Method: Drop Slide after Ehrler)

or as a sum parameter with the aid of conductometry. The methods described above have the indispensable advantage of measuring not the ionic content of the wipers but the transfer of the ions which are transferred by the wiping procedure.

Conclusion

Research never ends. Although today we may have reached a leading position of insight in our narrow field of research, we must make a great effort everyday to hold this. That is at the same time our duty and our challenge. The specialized literature has grown so extensive that it cannot be taken in even by the most interested specialist in its existing breadth.

If you, dear reader, have advanced to this place and have actually read the previous chapters, then you are either a competitor or quite an extraordinarily interested and active contemporary. For the latter I (the author) congratulate you with all my heart.

Essays on the topic of cleanroom consumable products, published by the author since 1985

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2. Labuda, Win - Ionic- und Particle-Contamination by Various Cleanroom-Commodities, VDI-publication 693, 1988 - München

3. Labuda, Win - Quality-Assurance at the Production of Cleanroom-Commodities, VDI-publication 783, 1989 - Stuttgart

4. Labuda, Win - Measurement of Particle Shedding from different Wiping Materials, ICCCS-World-Congress, Zürich 1990, Proceedings

5. Labuda, Win - A Product-specific Yield-Index - A new Dimension for the Valuation of Products, ICCCS-World-Congress, Zürich 1990, Proceedings

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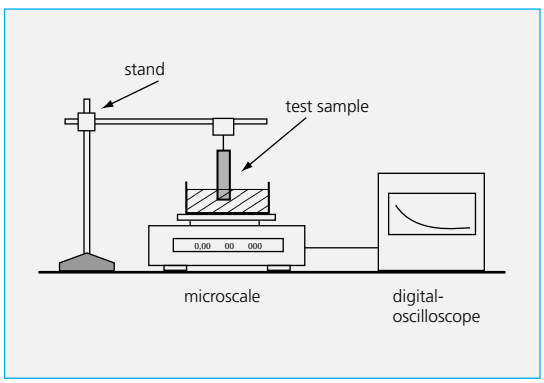

Fig. 33 Testing method for the lateral liquid absorption of cleanroom wipers

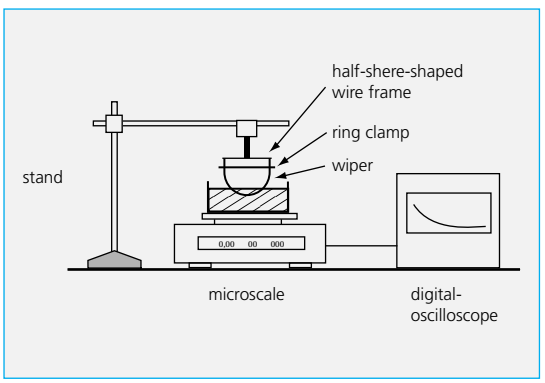

Fig. 34 Testing method after Siegmann for the lateral liquid absorption of multi-layered cleanroom wipers

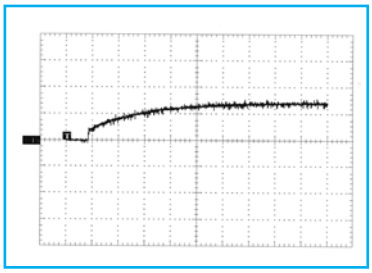

Fig. 35 Liquid absorption diagram, cellulose nonwoven 30 g/m2, 4-layered, lateral (1 field horizontal = 10 s; 1 field vertical = 10 g water absorption)

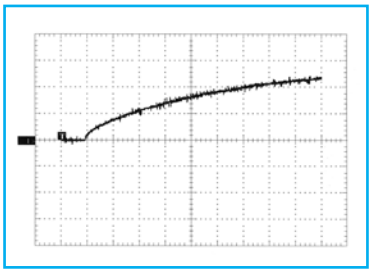

Fig. 36 Liquid absorption diagram, polyester-knit 185 g/m2, 4-layered,lateral (1 field horizontal = 10 s; 1 field vertical = 10 g water absorption)

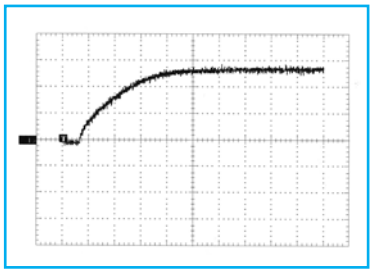

Fig. 37 Liquid absorption diagram, cellulose-nonwoven 70 g/m2, 4-layered, lateral (1 field horizontal = 10 s; 1 field vertical = 10 g water absorption)

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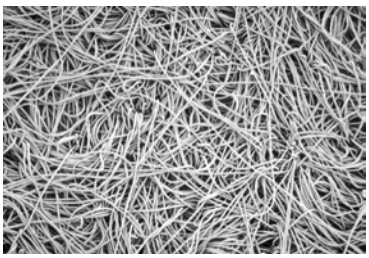
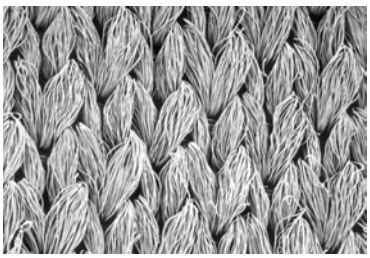
Translation: Carol Oberschmidt

Appendix 1: Electron-microscope photos of the surfaces of diverse HiTech wipers
(30 x enlarged)

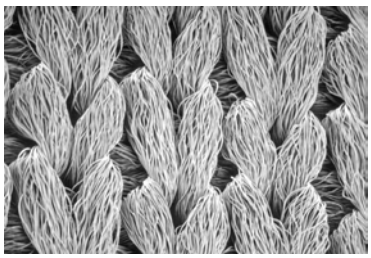
Cellulose-polyester-nonwoven, MANUFACTURER CODED CLABSG



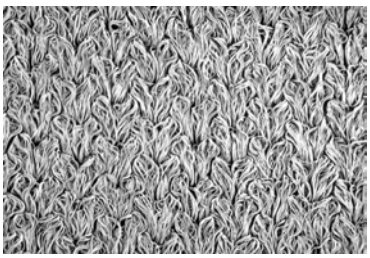
Viscose-nonwoven, MANUFACTURER CODED CLVICG



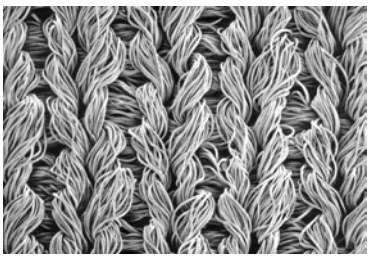
Polyester-knit, MANUFACTURER CODED CLHDMG



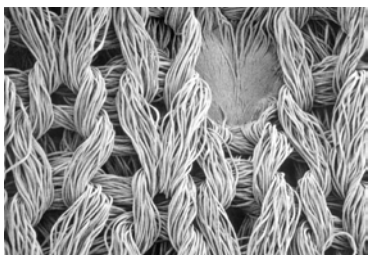
Robust polyester-knit, MANUFACTURER CODED CLHDSG



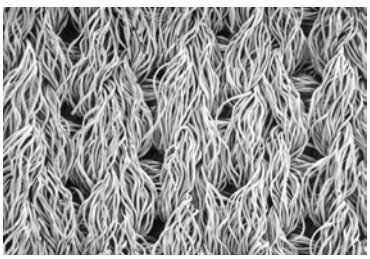
Mixed-fibre-microknit, MANUFACTURER CODED CLMWBG



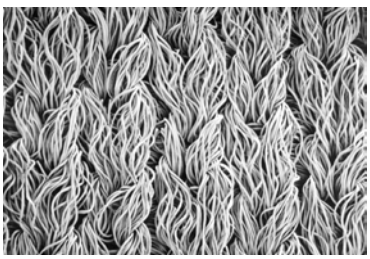
Polyester-knit, MANUFACTURER CODED TEA10U



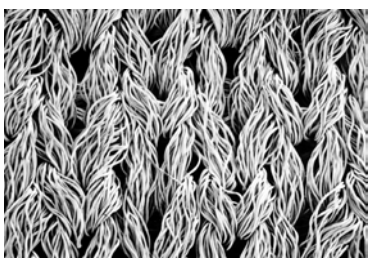
Multi-layered thermobonded polyester-knit (with visible bonding-point), MANUFACTURER CODED TEAS1U



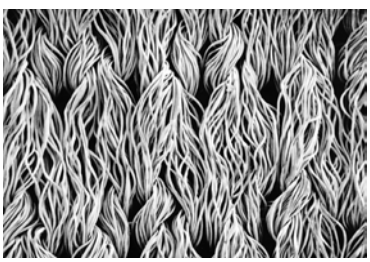
Polyester-knit, MANUFACTURER CODED TEALTU



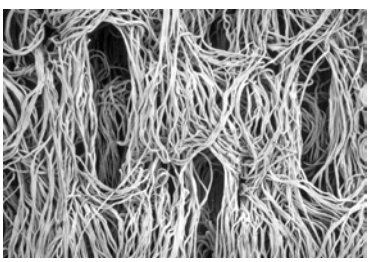
Polyester-knit, MANUFACTURER CODED TEAWPU



Polyester-knit, MANUFACTURER CODED MLANGU



Polyester-knit, MANUFACTURER CODED BSU30U



Cellulose-nonwoven, MANUFACTURER CODED AHBECJ