

WIPER

The present invention relates to an improved low linting non-woven wiper, in particular to a wiper having low metallic ion content.

Improvements in the manufacturing of high
5 technology items such as micro-electronic devices have necessitated the maintenance of essentially a clean room atmosphere. These devices are, in essence, micro processors, the equivalent of the central processing unit of a small computer. They are manufactured using
10 "wafers", thin slices of silicon on which circuits have been fabricated. The integrated circuits accomplish the separation and interconnection of transistors and other circuit elements electrically. The circuit elements are interconnected by a conducting film of
15 evaporated metal that is photoengraved to leave the appropriate pattern of connections. An insulating layer is required to separate the underlying semiconductor from the metal film except where contact is desired. This insulating layer is formed on the surface of the
20 wafer after the wafer has been processed and before the conducting metal is evaporated on it. Contamination, even by bits of lint or dust can bridge these circuits and cause such devices to be defective and is a major source of rejection. Therefore, there is a need to

maintain all surfaces and workpieces as free from such contamination as possible. This is usually accomplished in part by wiping these surfaces, and a number of specialized wipers have been developed for this purpose. 5 However, it is critical that the wiper, itself, in addition to being able to wipe cleanly, not contribute to the problem of dusting or linting. Conventional cloth wipes and tissue wipes, accordingly, are not entirely satisfactory. Various nonwoven wipes are also 10 available, but while some are low linting, these require treatment for wettability in order to provide the absorbency and clean wiping characteristics desired for these applications. Such treatments have been most effective utilizing an anionic wetting agent such as 15 Aerosol OT which is high in sodium ion content. These metallic ions present special problems since, if present in high enough concentrations, they may change the electrical properties of metal oxide semiconductors making the devices defective. Accordingly, such wipers 20 have also not been entirely satisfactory. A need, therefore, has been demonstrated for a low linting, clean wiping wiper low in metallic ion content for these specialty applications and for other applications as well.

As stated above, nonwoven disposable and limited use wipers are well-known. Any of a number of nonwoven processes can be used to form base materials for wipers. For example, nonwovens formed by meltblowing, spinning, carding, and fibrillating techniques have been utilized. When formed from synthetic thermoplastic filaments, such materials are normally hydrophobic and non-wettable. For most applications, therefore, it is necessary to treat the nonwoven to make it wettable. A wide variety of anionic and nonionic wetting agents has been developed for this purpose and are in use. Among these, sodium dioctyl sulfosuccinate, such as Aerosol OT, has become a highly preferred agent as providing rapid wettability. For less demanding applications, other wetting agents such as those identified below have been used.

For clean room applications such nonwoven wipers have not proven entirely satisfactory because the treatments for wettability have required a compromise between wetting characteristics produced by Aerosol OT and the need for low metallic ion content. Other specialty wipers have been developed for these applications. For example, woven textile wipers have been used but are expensive and linty. Long fibered, wetlaid cellulose wipers tend also to be linty and of low absorbency

and bulk.

Representative descriptions of the prior art products and materials include U.S. Patent 3,811,957 dated 21 May 1974 to Buntin which describes meltblown materials and wettability treatments, U.S. Patent 2,999,265 dated 12 September 1961 to Duane et al directed to a cleansing and deodorizing pad having a surface active agent treatment, U.S. Patent 3,520,016 dated 9 October 1968 to Meitner describing a cellulosic wipe, U.S. Patent 3,254,642 and 3,956,155 dated 4 May and 11 May 1976, respectively, to Schwuger describing textile wipes, and U.S. Patent 3,978,185 dated 31 August 1976 to Buntin et al describing a meltblown material useful as a wiper.

The present invention is directed to a highly effective, low linting nonwoven wiper having a low metal, particularly sodium, ion content for disposable or limited use application. In order to achieve desired wiping properties for nonwoven wipers made from hydrophobic thermoplastic synthetic fibers, such materials must be treated with a surfactant to obtain wettability with aqueous materials. While it is known to treat such nonwoven materials with surfactants, including sodium dioctyl sulfosuccinate such as Aerosol OT, such treatments increase the metallic ion content of the

5 wiper. For many applications this is acceptable. However, for clean room applications as necessitated in the manufacture of micro-electronic devices mentioned above, and the like, high metallic ion content wipers cannot be tolerated. In such cases treatment with other surfactants has been necessary which, although reducing the level of metallic ion content within acceptable limits, significantly adversely affects wiping properties.

10 According to this invention, there is provided a wiper comprising a nonwoven substrate having a basis weight in the range of from 0.25 oz/yd² to 6 oz/yd² (8.5 - 203.4 g/m²) which substrate comprises hydrophobic, thermoplastic fibers treated with
15 0.2 to 0.7% by weight relative to the wiper weight of a surfactant mixture comprising 25 to 75% by weight of an alkali metal dialkyl sulfosuccinate and 75 to 25% by weight of a nonionic surfactant, the said wiper having an alkali metal ion content
20 of less than 100, preferably less than 77, parts per million relative to the wiper weight and a sink time wettability (as hereinafter defined) of less than 5 seconds.

The alkali metal both of the sulfosuccinate
25 salt surfactant and to which the alkali metal ion content refers will generally be sodium. While the particularly preferred alkali metal dialkyl sulfosuccinate is sodium dioctyl sulfosuccinate (Aerosol OT), other alkali metal di-(C₆₋₁₂ alkyl)
30 sulfosuccinates may also be used to advantage. The preferred nonionic surfactants include alkyl phenoxy ethanols such as that available under the trade name Triton ^(Registered Trade Mark) X-100.

In accordance with the present invention, it has been found that treatment of such nonwoven materials with a specific mixture of surfactants including an alkali metal dialkyl sulfosuccinate such as sodium dioctyl sulfosuccinate and a nonionic surfactant such as Triton X-100 (alkyl phenoxy ethanol) reduces the metallic ion content of the wiper to within acceptable limits and yet maintains or even improves the highly effective wiping characteristics obtained through the use of a sulfosuccinate surfactant alone. Thus, the wiper of the invention provides, especially for micro-electronics manufacturing clean room applications, a product possessing the low lint, effective wiping, and low metallic ion content properties essential to these operations.

Certain test procedures have been employed to determine wettability and linting characteristics.

Wettability determinations were made by filling a pan with distilled water to a depth of a least
5 one inch (2.54cm). A four inch by four inch square (10.2cm by 10.2cm) of the material to be tested was placed carefully on the surface of the water and the time was measured for about 90% of the upper surface of the material to be wetted by strike through;
10 this time is referred to herein as the "sink time wettability". Visual observation was employed.

Lint tests were carried out using a Climet particle counter. This test also employed a mechanical particle generator which applies bending, twisting,
15 and crushing forces to sample specimens. Samples measuring six inches by six inches (15.2cm by 15.2 cm) are placed in position in a one cubic foot (0.28 m^3) enclosure. A bottom holder raises four inches (10.2cm) and simultaneously turns through 180° then returns to
20 the start position with the cycle completed in slightly

less than one second. The enclosure is connected by tubing to the particle counter which draws the particles at 20 cubic feet per hour ($0.57 \text{ m}^3/\text{hour}$). Each count takes 37 seconds and represents the
5 number of particles 10 microns or larger in 0.01 cubic feet (283cm^3) of air.

Sodium ion concentrations were determined by extraction of all hot water soluble materials from a sample of wiper material. These were then subjected
10 to atomic absorption spectroscopy using a Model 305 Perkin Elmer Atomic Absorption Spectrophotometer.

Many nonwoven synthetic webs of thermoplastic, hydrophobic fibers may be employed as the substrate material for the wiper of the present invention.
15 For cost purposes, nonwovens made by spinning or meltblowing thermoplastic polyolefin materials such as polypropylene and polyethylene are preferred. Most preferred for clean wiping and absorbency properties are meltblown polypropylene materials described, for
20 example, in U.S. Patent 3,959,421 dated 25 May 1976 to Weber et al. Such materials are composed of microfibers having an average diameter of generally 10 microns or less and, when treated for wettability, are effective in absorbing both aqueous and oily materials. In general,
25 the substrate material will have a basis weight in the range of from 0.25 oz/yd^2 (8.5 g/m^2) to 6 oz/yd^2 (203.4 g/m^2), preferably 0.4 oz/yd^2 (13.6 g/m^2) to 3 oz/yd^2 (102 g/m^2) and will be bonded to provide

strength properties of at least about 4 lbs/oz (64 g/g) MD grab tensile and sufficient for the intended wiping purpose. Tensile results were obtained essentially in accordance with ASTM D-1117-74. Samples 4 inches by 6 inches (10.2 cm by 15.2 cm) are prepared with 5 each having its length in the "machine" and "cross" directions. An Instron ^(Registered Trade Mark) machine is used having one jaw face 1 inch square (2.54 cm square) and the other 1 inch by 2 inches (2.54 cm by 5.1 cm) or larger with the longer dimension perpendicular to the direction of load. At a crosshead speed of 12 inches per minute (30.5 cm per minute), the full scale load was recorded and multiplied by a factor as follows: Readings in lbs (kg in brackets): 2 (0.91), 5 (2.27), 10 (4.54), 20 (9.07), 50 (22.68); factors (respectively): 0.0048, 0.012, 0.024, 0.048, 0.120. The results were reported in units of weight (e.g. lbs. weight) required to break the sheet. Bonding may be achieved by any of the conventional means such as patterned application of heat and pressure, needling, adhesives, or utilizing the thermoplastic properties of the filaments themselves.

25 The method of incorporating the surfactant mixture in accordance with the invention is not critical. Thus, the surfactant mixture can be added by conventional techniques such as spraying, dipping, coating, impregnating, and printing.

30 In general, the amount of the mixture added will be dictated by the ratio of the components, the permissible sodium ion content level, and the degree of wettability desired. For most

applications this will result in addition of the surfactant mixture in the range of from about 0.2% to 0.7% by weight based on the weight of the nonwoven substrate, and preferably within the range of from 0.3% to 0.5%. For cost and performance reasons, the surfactant mixture is preferably added by means of the quench spray described in the above-mentioned Weber et al U.S. Patent 3,959,421 dated 25 May 1976.

Some embodiments of the invention will now be described by way of example and with reference to the accompanying drawings, in which:

Figures 1 to 4 are graphs illustrating sink time wettability determinations for wipers containing various ratios of the surfactants Triton X-100 and Aerosol OT-75 (75% solids) ranging from 100% Triton X-100 to 75% Aerosol OT-75;

Figures 5 to 8 are graphs illustrating in similar fashion sink time wettability determinations for wipers containing various ratios of the surfactants Igepal CO630 (nonyl-phenoxypoly (ethyleneoxy) ethanol - a nonionic surfactant) and Aerosol OT-75 ranging from 100% Igepal CO630 to 75% Aerosol OT 75;

Figures 9 to 12 are graphs illustrating in similar fashion sink time wettability determinations for wipers containing various ratios of the surfactants Sandozin D-100 (alkylphenol polyglycolether - a nonionic surfactant) and Aerosol OT-75 ranging from 100% Sandozin D-100 to 75% Aerosol OT-75; and

Figures 13 is a graph illustrating the increase in sodium ion content in parts per million resulting from the addition of Aerosol OT to the wiper substrate.

The ratio of the surfactant mixture components

5 must be within certain limits to achieve the benefits of the present invention. For example if sodium dioctyl sulfosuccinate is used the maximum amount will be determined by the permissible level of sodium ion content in the wiper. In general, for clean room applications
10 such as the manufacture of micro-electronic devices, alkali metal (sodium) ion content should not exceed 70 parts per million and, preferably, is less than 60 parts per million. In accordance with FIG. 13, this dictates
15 a percentage add-on by weight of Aerosol OT-75 in the wiper of up to 0.20 and preferably 0.16 or less.

To obtain most effective wiping properties, a wettability as determined by sink time of less than 5 seconds and, preferably, less than 3 seconds, is needed.

From FIGS. 1 to 4, it can be seen that
20 the use of 25% Aerosol OT-75 in the surfactant mixture requires a per cent add-on of nearly one per cent to achieve a sink time wettability of 3 seconds. In contrast, a 50/50 ratio requires less than 1/2 per cent add-on as does a ratio of 25% Triton X-100 and 75% Aerosol OT-75. However,
25 at the 75% Aerosol OT-75 level, the sodium ion addition would be in excess of the preferred level. Accordingly, the amount of Aerosol OT-75 in the mixture in accordance with the present invention is within the range of

25 to 75% and preferably, within the range of 40 to 60%; ideally, the components are included in about equal proportions.

The following Examples are provided to illustrate the scope of the present invention:

Examples 1 - 4

A series of wiper materials was prepared at varying levels of add-on using the following surfactant compositions: 100% Triton X-100, 75% Triton X-100 and 25% Aerosol OT-75 (75% solids), 50% Triton X-100 and 50% Aerosol OT-75, and 25% Triton X-100 and 75% Aerosol OT-75. The substrate material was a meltblown nonwoven as generally described in U.S. Patent 3,959,421 dated 25 May 1976 to Weber et al and having a basis weight of 85 g/m². These treated materials were tested for wettability by the sink time method, and the results are illustrated in FIGURES 1 through 4.

Examples 5 - 8

The meltblown nonwoven substrate materials as used in Examples 1 - 4 were similarly treated with the following surfactant compositions: 100% Sandozin D-100, 75% Sandozin D-100 and 25% Aerosol OT-75, 50% Sandozin D-100 and 50% Aerosol OT-75,

and 25% Sandozin D-100 and 75% Aerosol OT-75. These materials were also tested for wettability by the sink time method, and the results are shown in FIGS 5 - 8.

Examples 9 - 12

5 The meltblown nonwoven substrate materials as used in Examples 1 - 4 were similarly treated with the following surfactant compositions: 100% Igepal CO630, 75% Igepal CO630 and 25% Aerosol OT-75, 50% Igepal
10 CO630 and 50% Aerosol OT-75, and 25% Igepal CO630 and 75% Aerosol OT-75. The treated materials were tested for wettability by the sink time method and the results are shown in FIGS 9 - 12.

To further illustrate the present invention,
15 the material of Examples 1 - 4 was treated with various add-on levels of Aerosol OT-75, alone and Triton X-100 alone. Wettability tests by the sink time method were performed, and the results are shown in Table I. Also included for comparison are the results
20 obtained with the 50/50 mixture of the present invention. As can be seen, the wettability results obtained with the mixture are nearly equal to those obtained with Aerosol OT-75, alone, and much better than those obtained with Triton X-100, alone, at the higher
25 add-on percentage levels.

TABLE I

Total Add-On / g	Time(s)				Experimental 50/50 Mixture
	If All Aerosol OT-75	If All Triton X-100	If 1/2 Aerosol OT-75		
3.0	0.3	0.75	0.3		.4
2.0	0.3	0.75	0.3		.4
1.0	0.3	1.7	0.9		.45
0.75	0.6	2.4	1.3		.6
0.5	0.9	4.1	4.0		1.2
0.4	1.2	5.6	5.0		4.2
0.3	3.3	>120	>120		10.5
0.25	4.0	>120	>120		>120

For comparative purposes, Table II below lists the results of lint tests performed on the preferred wiper of the present invention including the mixture of 50/50 Triton X-100 and Aerosol OT-75 (referred to in Table II

5 as "Wiper of The Present Invention") along with

various commercially available wiping materials including those used for clean room applications.

Also included are measurements of sodium ion content.

Wiper A is a conventional meltblown, treated wiper.

10 Wiper B is representative of a clean room long fiber cellulose wipe. Wiper C is a dry creped tissue wipe.

Wiper D is a woven cloth wiper intended for clean room use. Wiper E is a thermally bonded polypropylene

15 carded web. As can be seen, with the exception of Wiper E, the wiper of the present invention is unique among those tested in providing both low metallic ion content and low lint results.

The exception to this is Wiper E which has low wettability and poor wiping properties and, therefore,
20 not readily adaptable for clean room applications.

TABLE II

	PPM NA ⁺		Lint Particles				
	Hot Water	Cold Water	<10 Microns		<0.5 Microns		
			1 Cycle	2 Cycles	3 Cycles	4 Cycles	5 Cycles
Wiper A	161.7	104.9	.4	98	155	213	290
Wiper B	141.0	133.7	.4	978	1688	2115	2444
Wiper C	104.8	96.0	0.6	2185	3774	5134	6179
Wiper D	31.4	22.7	5.0	406	441	448	393
Wiper E	3.9	4.0	1.4	29	35	25	24
Wiper of The Present Invention	60.0	40.0	4.0	135	214	285	418

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Thus, it is apparent that there has been provided
in accordance with the invention, an improved, low
lint nonwoven wiper that contains reduced metallic
ion levels and fully satisfies the objects, aims,
5 and advantages set forth above.

CLAIMS

1. A wiper comprising a nonwoven substrate having a basis weight in the range of from 0.25 oz/yd² to 6 oz/yd² (8.5 - 203.4 g/m²) which substrate
5 comprises hydrophobic, thermoplastic fibers treated with 0.2 to 0.7% by weight relative to the wiper weight of a surfactant mixture comprising 25 to 75% by weight of an alkali metal dialkyl sulfosuccinate and 75 to 25% by weight of a nonionic surfactant,
10 the said wiper having an alkali metal ion content of less than 100 parts per million relative to the wiper weight and a sink time wettability (as hereinbefore defined) of less than 5 seconds.
2. A wiper as claimed in claim 1 wherein the
15 said alkali metal is sodium.
3. A wiper as claimed in either of claims 1 and 2 wherein the said alkali metal dialkyl sulfosuccinate is sodium dioctyl sulfosuccinate.
4. A wiper as claimed in any one of claims 2
20 to 3 wherein the said thermoplastic fibers are polypropylene fibers.
5. A wiper as claimed in any one of claims 1 to 4 wherein the said thermoplastic fibers are meltblown.
- 25 6. A wiper as claimed in any one of claims 1 to 5 wherein the said nonionic surfactant is an alkyl phenoxy ethanol.



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7. A wiper as claimed in any one of claims 1 to 6 wherein the said surfactant mixture contains 40 to 60% by weight of the said alkali metal dialkyl sulfosuccinate.

5 8. A wiper as claimed in any one of claims 1 to 6 wherein the said surfactant mixture contains substantially equal proportions by weight of the said alkali metal dialkyl sulfosuccinate and the said nonionic surfactant.

10 9. A wiper as claimed in any one of claims 1 to 8 wherein the said basis weight is in the range of from 0.4 oz/yd² to 3 oz/yd² (13.6 to 102 g/m²).

10. A wiper as claimed in any one of claims 1 to 9 wherein the said substrate contains 0.3 to 15 0.5% by weight of the said surfactant mixture.

11. A wiper as claimed in any one of claims 1 to 10 wherein the said sink time wettability is less than 3 seconds.

12. A wiper as claimed in any one of claims 1 20 to 11 wherein the said alkali metal ion content is less than 77 parts per million relative to the wiper weight.

13. A wiper as claimed in any one of claims 1 to 12 wherein the average fiber diameter in the 25 said substrate is no greater than 10 microns.

14. A wiper as claimed in claim 1 comprising a meltblown, polypropylene substrate having an average fiber diameter no greater than 10 microns



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and a basis weight of from 0.4 oz/yd² to 3 oz/yd²
(13.6 to 102 g/m²), the said substrate containing
0.3 to 0.5% by weight of a surfactant mixture containing
sodium dioctyl sulfosuccinate and an alkyl phenoxy
5 ethanol in which mixture the said sodium dioctyl
sulfosuccinate is present in an amount of 40 to
60% by weight, the said wiper having a sodium ion
content of less than 77 ppm relative to the wiper
weight and a sink time wettability (as hereinbefore
10 defined) of less than 3 seconds.

15 15. A wiper as claimed in claim 14 wherein in
the said surfactant mixture the said sodium dioctyl
sulfosuccinate and the said alkyl phenoxy ethanol
are present in substantially equal proportions
by weight.

16. A wiper substantially as hereinbefore described
comprising a nonwoven fibrous substrate containing
a mixture of an alkali metal dialkyl sulfosuccinate
and a nonionic surfactant.