

(19) **DANMARK**

(10) **DK/EP 3234054 T3**



Patent- og
Varemærkestyrelsen

(12) Oversættelse af
europæisk patentskrift

-
- (51) Int.Cl.: **C 09 J 109/06 (2006.01)** **C 09 J 7/38 (2018.01)** **C 09 J 125/10 (2006.01)**
C 09 J 153/02 (2006.01)
- (45) Oversættelsen bekendtgjort den: **2023-01-09**
- (80) Dato for Den Europæiske Patentmyndigheds bekendtgørelse om meddelelse af patentet: **2022-11-02**
- (86) Europæisk ansøgning nr.: **15871139.0**
- (86) Europæisk indleveringsdag: **2015-12-18**
- (87) Den europæiske ansøgnings publiceringsdag: **2017-10-25**
- (86) International ansøgning nr.: **US2015066561**
- (87) Internationalt publikationsnr.: **WO2016100763**
- (30) Prioritet: **2014-12-19 US 201462094463 P**
- (84) Designerede stater: **AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR**
- (73) Patenthaver: **Tredegard Surface Protection, LLC, 1100 Boulders Parkway, North Chesterfield, Virginia 23225, USA**
- (72) Opfinder: **SANTOSO, Ricky, 6225 Ginda Terrace, Glen Allen, VA 23059, USA**
PATEL, Shailesh, Chunilal, 5643 Charter Oak Drive, Chesterfield, VA 23832, USA
RAY, Carl, Douglas, 9015 Spyglass Hill Mews, Chesterfield, VA 23832, USA
DESAI, Bankim, Bhupendra, 9021 Spyglass Hill Mews, Chesterfield, VA 23832, USA
- (74) Fuldmægtig i Danmark: **PROXIP, 3 rue de l'Arrivee, 75749 Paris cedex 15, Frankrig**
- (54) Benævnelse: **SURFACE PROTECTION FILM**
- (56) Fremdragne publikationer:
EP-A1- 0 479 457
WO-A1-2013/021788
WO-A1-2014/168069
CN-A- 104 220 548
JP-A- H09 143 439
US-A1- 2013 295 312
US-A1- 2014 335 334
US-A1- 2014 349 053
US-B2- 8 043 682
US-B2- 8 182 926

DESCRIPTION

BACKGROUND OF THE INVENTION

[0001] The present application relates generally to surface protection films and, more particularly, to surface protection films with improved release of the film from substrates to which the film has been applied and reduced unwinding tension when the surface protection film is removed from a roll during use.

[0002] Surface protection films, also known as masking films, are typically used to provide a physical barrier to prevent damage, contamination, scratching, scuffing, or other marring of a substrate to which they are adhered. Surface protection films may be used to provide such protection during manufacture, shipping, or storing prior to use of the substrate. Substrates used as optical components for televisions, computer monitors and other displays require surface protection films that are adhered to the surface of the substrate and then subsequently removed from the surface of the substrate without damaging the substrate or leaving residue, staining or other defects on the surface of the substrate.

[0003] The application of surface protection films to the substrates to be protection usually takes place by unwinding from a roll of surface protection film, a single layer of surface protection film. As the film is pulled off the roll, the spiral orientation of the surface protection film on the roll may have the adhesion layer disengaging from the release layer as the film is removed from the roll of film. The disengagement may result in a higher unwind tension or peel force while the film is unwound. The higher unwind tension or peel force may provide problems for an end user of the film including breakage and/or deformation in the film during unwinding, lower efficiency when converting the film, more difficult laminating the film to a substrate of an end user (e.g., including damage to substrate of the end user during lamination of the film to the substrate), and/or the like. As such, there still exists a need to provide a surface protection film having improved unwind tension or peel force for an end user such that there may be improvement in film breakage, efficiency during converting, ease of laminating a film to a substrate without causing damage, and/or the like.

[0004] Patent publication WO 2013/021788 A1 describes a pressure-sensitive adhesive sheet comprising a pressure-sensitive adhesive layer that contains at least one selected from a polyolefin-based resin and a styrene-based resin.

Patent publication JP H09143439 A describes a surface-protective film composed of a base material film made of a polyolefin-based resin and an adhesive layer on one surface. The adhesive layer is composed of a resin composition comprising 50-75wt.% of a block copolymer and 25-50wt.% a high-density polyethylene resin, and an ethylene- α -olefin copolymer in an amount of 0-25wt.% based on 100wt.% of the resin composition.

Patent publication US 2014/335334 A1 describes a protective film that includes a non-adhesion layer and adhesion layer. A surface layer of the non-adhesion layer is formed from a blend of

homo-polypropylene (H-PP) and low density polyethylene (LDPE), a middle layer of the non-adhesion layer is formed from a blend of high density polyethylene (HDPE) and LDPE among polyethylene series. The adhesion layer is formed from thermoplastic elastomer added with low hardness plastic material.

Patent publication WO 2014/168069 A describes a self-adhesive surface-protecting film produced by laminating an adhesive layer on one surface of a base layer comprising a polypropylene resin and also laminating a release layer on the opposite surface of the base layer by co-extrusion. The adhesive layer comprises a styrene elastomer (A) that is a hydrogenated product of a block copolymer of a styrene polymer block and a butadiene polymer block and has a styrene content of 5 to 40 wt% and a styrene elastomer (B) that is a hydrogenated product of a random copolymer of styrene and butadiene and has a styrene content of 40 to 70 wt%. The ratio of the weight of the styrene elastomer (A) to the total weight of the two styrene elastomers is 5 to 80 wt% inclusive, an adhesiveness-imparting resin is contained in an amount of 5 to 25 parts by weight relative to the sum total, i.e., 100 parts by weight, of the two styrene elastomers.

SUMMARY OF THE INVENTION

[0005] An aspect of the invention provides a film as defined in claim 1, which is appended to the description. Embodiments may comprise additional features some of which are defined in the dependent claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[0006]

FIG. 1 illustrates an example process and process configuration for unwinding a roll of film as described herein.

FIG. 2 illustrates an example of the roll that may be unwound using the process and process configuration of FIG. 1 being marked at different portions or spots such that a measurement may be taken at such portions or spots as described herein.

FIG. 3 is a graphical representation of surface protection film unwind tension test results.

FIG. 4 shows the image results of a residue test of a sample including 80% styrene block copolymer and 20% LDPE.

FIG. 5 shows the enhanced image results of a residue test of a sample including 80% styrene block copolymer and 20% LDPE.

FIG. 6 shows the image results of a residue test of a sample including 100% styrene block copolymer.

FIG. 7 shows the enhanced image results of a residue test of a sample including 100% styrene block copolymer.

DETAILED DESCRIPTION OF THE INVENTION

[0007] The present application relates to an improved surface protection film provided improved release of the film from substrates to reduce residue and to provide unwinding sound abatement when the surface protection film is removed from a roll during use. The adhesion layer of the surface protection film comprises an improved styrene block copolymer with an increased melt flow rate (MFR). The adhesion layer is combined with a release layer and an optional core layer comprising polyolefin polymers. The surface protection film may be applied to a substrate surface by unwinding the surface protection film from a roll of surface protection film. The surface protection film may be subsequently removed from the substrate surface leaving minimal residue on the substrate surface. The surface protection film may also be tailored to provide desired degrees of adhesion to different surfaces.

[0008] The surface protection film may comprise an adhesion layer, an optional core layer and a release layer. The adhesion layer is the layer placed into contact with a substrate surface. The composition of the adhesion layer affects, not only the adhesion of the film to substrates to be protected, but also the adherence of the film to itself when wound in a roll and, thus, the unwinding tension (or peel force during unwinding) of a single layer of surface protection film from the roll.

Adhesion Layer

[0009] Adhesion means adherence to a surface of the substrate to be protected through intimate contact via the natural blocking adhesion which exists between a very smooth surface and another smooth surface via polar bonding, ionic bonding and, in some instances, hydrogen bonding, and/or Van der Waals secondary bonding. The adhesive-less adhesion intended herein is a releasable adhesion where the adhesion is reversible such that neither the film nor the substrate to which it is applied is modified or damaged. Adhesion does not include materials known as pressure sensitive adhesives, heat bonding or cross-linking functionality of adhesives as the adhesion force between the substrate surface and a film with pressure sensitive adhesives, heat bonding or cross-linking functionality of adhesives is elevated to a point where the peel strength needed to remove such a film will exceeds the tensile strength of such a film itself thus causing such a film to tear or break before it will peel away from the substrate.

[0010] The thickness of the adhesion layer of the present surface protection film is between about 1 and about 20 microns, such as between about 3 microns and about 15 microns, such

as 6, 7, 8, 9, 10, 11, 12, 13, 14, or 15 microns.

[0011] Hydrogenated Styrene block copolymer - The adhesion layer comprises essentially of a hydrogenated styrene block copolymer to achieve the desired unwinding sound abatement when the surface protection film is removed from a roll during use and the reduction of residue on a substrate after the surface protection film has been applied and then is subsequently removed from a substrate.

[0012] Hydrogenated block copolymer having a polystyrene block - polydiene block polymer structure prior to hydrogenation. The Hydrogenated block copolymer, before hydrogenation, can be linear or radial. Suitable polydienes include polybutadiene (1,3 -butadiene), polyisoprene and mixtures thereof. Hydrogenation of the polystyrene block - polydiene block structure results in a styreneethylene-(butylene/isoprene)-styrene polymeric structure, otherwise referred to as "SEBS". See US7439301 (see, e.g., Col. 4, line 8 - Col. 8, line 4), US7348376 (see, e.g., Col. 3, line 9 - Col. 6, line 19), US20130225020 (see, e.g., paragraphs [0030] - [0051]), WO 2014/087814, and WO 30 2014/087815 for further descriptions.

[0013] Styrene content - the styrene content of the hydrogenated styrene block copolymer is less than 40 wt% by weight of the copolymer, such as between about 30 and about 40 wt%, such as 34 wt%. Following hydrogenation of the polystyrene block - polydiene block polymer, from 0 to 10 percent of the styrene double bonds in the polystyrene block have been hydrogenated.

[0014] Melt flow rate (MFR) - the MFR of the hydrogenated styrene block copolymer is inversely correlated to viscosity of the hydrogenated styrene block copolymer. A high melt flow rate means that the styrene block copolymer has a low viscosity and vice versa. As used herein including the claims, unless otherwise stated, "melt flow" shall mean the melt flow value determined according to ASTM D-1238, at 230° C, under a 2.16 kg mass. In one embodiment, the hydrogenated styrene block copolymer have a melt index of greater than or equal to 12. In another embodiment, the hydrogenated styrene block copolymers have a melt index of greater than or equal to 20. In still another embodiment, the hydrogenated styrene block copolymers have a melt index of greater than or equal to 40. Another embodiment a hydrogenated styrene block copolymers having a melt index of from about 20 to about 100. See US7439301, US7348376, US20130225020, WO 2014/087814, and WO 2014/087815 for further descriptions.

[0015] Suitable hydrogenated styrene block copolymer includes Kraton MD 6951 manufactured by Kraton Performance Polymers Inc., having a styrene content of about 34 wt% and a MFR of 48.

[0016] The adhesion layer may include one or more other polymers or copolymers. Various embodiments of the adhesion layer have been contemplated and tested, which allowed arriving at the claimed invention. For example, the adhesion layer may include a low density polyethylene (LPDE). The one or more other polymers or copolymers may be blended with the

hydrogenated styrene block copolymer. For example, the adhesion layer may include the hydrogenated styrene block copolymer and the other polymers or copolymers such as LPDE. According to examples, the adhesion layer may comprise 10% to 100%, by weight, hydrogenated styrene block copolymer and 0% to 50%, by weight, LPDE. In particular examples, the adhesion layer may comprise about 60%, by weight, hydrogenated styrene block copolymer and about 40%, by weight, LDPE, or about 80%, by weight, hydrogenated styrene block copolymer and 20%, by weight, LPDE. As such, the adhesion layer may include a mixture of hydrogenated styrene block copolymer and LDPE in a ratio range of about 100:0 to 51 :49 by weight, including for example, ratios of about 80:20 and about 60:40.

[0017] In various embodiments, the adhesion layer may comprise 10% to 90%, by weight, hydrogenated styrene block copolymer. In particular embodiments, the adhesion layer may comprise 70% to 90%, by weight, hydrogenated styrene block copolymer. In such embodiments, the adhesion layer may also comprise 10% to 30%, by weight, LDPE. In a particular embodiment, the adhesion layer can consist essentially of the hydrogenated styrene block copolymer alone.

[0018] In alternative embodiments, the adhesion layer may comprise 15% to 90%, by weight, styrene block copolymer and 10% to 50%, by weight, LDPE, and, optionally, 10% to 35%, by weight, HDPE.

[0019] The adhesion layer includes an external surface (surface brought into contact with the substrate surface) having a measure of smoothness within an operational range of about 0 Ra to 60 Ra, such as about 0 Ra to about 4 Ra, such as about 4 Ra to about 30 Ra. Note that the range of roughness for the substrate to which the surface protection film is applied is usually between 0 Ra and 150 Ra. As discussed herein, smoothness and roughness will be defined as the arithmetic average height (Ra) of the micropeaks and microvalleys of a surface to the center line of such surface as measured by a profilometer. Smoothness and roughness defined in this manner is typically expressed with units of microinches (10~6 inches). All testing of surface textures (relative smoothness and roughness) were conducted in accordance with ANS17ASME Test Method B46. 1 -1985

[0020] Additional components may be present, but should not materially affect the basic and novel characteristics of the desired unwinding sound abatement when the surface protection film is removed from a roll during use and the reduction of residue on a substrate after the surface protection film has been applied and then is subsequently removed from a substrate.

Core Layer

[0021] The surface protection film may comprise an optional core layer. The core layer, when present, may be located contiguously between the adhesion layer and the release layer. The core layer, when present, comprises one or more polyolefins, such as low density polyethylene, linear low density polyethylene, high density polyethylene, medium density

polyethylene, polypropylene, random copolymer polypropylene, polypropylene impact copolymers, metallocene linear low density polyethylene, plastomers, poly (ethylene-co-vinyl acetate), poly (ethylene-co-acrylic acid), poly (ethylene-co-methyl acrylate), cyclic olefin polymers, polyamides, or poly (ethyleneco- n-butyl acrylate). Suitable polyolefins include low density polyethylene, high density polyethylene, polypropylene, polypropylene impact copolymers and mixtures thereof. One suitable polyolefin mixture is of low density polyethylene and high density polyethylene in a ratio in a range of 60:40 to 40:60 by weight.

[0022] The thickness of the core layer of the present surface protection film, when present, is between 10 and 50 microns, such as 12 to 40 microns, such as 20 to 40 microns, such as 30, 31, 32, 33, 34, 35, 36, 37, 38, 39 and 40 microns.

Release Layer

[0023] The surface protection film may comprise a release layer. The release layer may be located contiguous to the adhesion layer or, when present, the core layer. The release layer comprises one or more polyolefins, such as low density polyethylene, linear low density polyethylene, high density polyethylene, medium density polyethylene, polypropylene, random copolymer polypropylene, polypropylene impact copolymers, metallocene linear low density polyethylene, plastomers, poly (ethylene-co-vinyl acetate), poly (ethylene-co-acrylic acid), poly (ethylene-comethyl acrylate), cyclic olefin polymers, polyamides, or poly (ethylene-co-n-butyl acrylate). Suitable polyolefins include low density polyethylene, high density polyethylene, polypropylene, polypropylene impact copolymers and mixtures thereof. One suitable polyolefin mixture is of low density polyethylene and high density polyethylene in ratio of 60:40 to 40:60 by weight. The thickness of the release layer of the present surface protection film is between about 1 and about 20 microns, such as between about 1 microns and about 10 microns, such as 2, 3, 4, 5, 6, 7, 8, 9 or 10 microns.

[0024] The relatively rough surface of the release layer generally possesses a measure of roughness between 12 Ra and 600 Ra, and more preferably, between 30 Ra and 300 Ra.

[0025] As used herein, the term "film" means a thin sheet or web produced by a cast extrusion or blown process. The films of this disclosure are made of polymers. The polymer may be further processed between rollers and cooled to form the web. The multilayer films can be produced by extruding the different layers simultaneously in a coextrusion process or by forming each film layer separately and then bonding or laminating the individual films together. In a preferred embodiment, the multiple layers of the masking film are co-extruded using any co-extrusion process known in the art. The use of co-extrusion allows for the relatively simple and easy manufacture of a multi-layered masking film composed of distinct layers, each performing specific functions.

Substrates

[0026] While the films of the invention may be applied to any substrate, typical substrates include, by way of illustration only, polycarbonates, acrylics, polyvinyl chlorides, PET, PETG, glass, ceramics and metals. The typical surface smoothness for these substrates is in the range of 0 to 150 Ra.

Application to substrates

[0027] Any of a variety of conventional methods can be utilized for applying the multilayer masking film to the substrate and for pressing the applied masking film against the smooth surface of the substrate. Generally speaking, the multilayer film will be taken off from a roll and will be directly applied to the substrate by means of a nip roll or similar system through which the multilayer film and the substrate are passed. As the film is pulled off the roll, the spiral orientation of the surface protection film on the roll has the adhesion layer disengaging from the release layer as the film is removed from the roll of film. The disengagement results in an unwind tension or peel force that may cause film breakage, lower efficiency during further processing where the film is applied to a substrate, more difficulty during lamination to the substrate (e.g., damage to the substrate), and/or the like.

EXAMPLES

[0028] Table 1 lists the constituent amounts for exemplary protective films. The total film thickness of comparative examples 1 and 2 and examples 1 and 2 is about 50 microns (47 gsm). The thickness of the release layer is about 7.5 microns (15% of the total film thickness). The thickness of the adhesion layer is about 10 microns (20%, of the total film thickness). The thickness of the core layer is about 32.5 microns (65% of the total film thickness). The examples tested differ in terms of the relative percentage of styrene block copolymer in the adhesion layer. For each variation, the difference between the main example and the "comparative example" is that the styrene content of the hydrogenated styrene block copolymer used in the adhesion layer is different. For example, the styrene content of the hydrogenated styrene block copolymer G1657 in the adhesion layer of the comparative examples 1-2 is between 12.3%-14.3%, such as about 13% and the styrene content of the hydrogenated styrene block copolymer MD6951 in the adhesion layer of the examples 1-2 is higher at about 34%.

Table 1 - Films for Comparative Unwind Tests

	Comparative Ex. 1 Roll 1 Constituent Content (wt% of Layer)	Ex. 1 Roll2 Constituent Content (wt% of Layer)	Comparative Ex. 2 Roll3 Constituent Content (wt% of Layer)	Ex. 2 Roll4 Constituent Content (wt% of Layer)
Release Layer				
HDPE	60	60	60	60
LDPE	40	40	40	40
Core Layer				
HDPE	60	60	60	60
LDPE	40	40	40	40
Adhesion Layer				
Kraton G1657	100		80	
Kraton MD6951		100		80
LDPE			20	20

Testing - Unwind Tension or Peel Force

[0029] Unwind tension or peel force are tested for the films of the comparative examples 1-2 and examples 1-2 by winding the respective film onto a roll i.e. rolls 1-4. Each of the rolls 1-4 for the comparative examples 1-2 and examples 1-2 have a length of 1200 meters and a width of 9 inches. FIG. 1 illustrates an example process and process set up 100 or architecture for testing unwind tension or peel force. As shown, the wound roll 104 of surface protection film 102 is placed in contact with a rider roll 106 and fed to a load cell roll 108 in an unwind direction as shown. Prior to unwinding, each of the rolls 1-4 was marked with a marker at different locations or spots of the roll including an outer portion 112, a center portion 114, and a core portion 116 as shown in FIG. 2. The outer portion 112 is located 1/16 inch from the outer diameter of the roll 104 to be tested (after being strung into the unwinding apparatus 100), the center portion 114 is located at the one half of the radius of the roll to be tested (after being strung into the unwinding apparatus) and the core portion is located 1/4 inch from the paper or cardboard core 118 at the center of the roll of film. The rolls 1-4 are unwound from the unwind roll 104 at a rate of 60 ft/min. As the roll 104 is unwound, tension readings are recorded or taken with a tensiometer gauge (FMS tensiometer) at the load cell roll 108 where the tensiometer is attached at each of the markers at the different locations or spots 112, 114, 116 on the roll 104 as shown in FIG. 2.

[0030] The graph in Figure 3 illustrates the results of the unwind tension or peel force for the comparative examples 1-2 and examples 1-2.

[0031] As shown in Figure 3, at each of the markers, the examples 1-2 of rolls 2 and 4 that include an adhesion layer with MD-6951 have a lower unwind tension or peel force in lb-f (where 1 lb-f equals 4.448 Newtons) than the comparative examples 1-2 of rolls 1 and 3 at each of the markers that include an adhesion layer with G1657. Figure 3 illustrates that MD6951 and its blends when used as an adhesive layer may improve the ease by which the roll can be unwound during further processing such as applying the film to a substrate which ultimately translates to ease of converting the film by an end user (i.e. there may be less film breakage, higher efficiency during converting, easier lamination of the film to a substrate without damage, and/or the like).

[0032] Adhesion test results for are shown in Table 2. The adhesion is based on the films of the present application being laminated on commercial random textured optical substrates available as 3M™ QDEF (quantum dot enhancement film), ex 3M with a 10 pound silicone rubber roller with the adhesion side of the film being in contact with the optical substrate to form a laminate. The laminates are allowed to sit at room temperature for 1 hr and at 60c for 5 minutes respectively before being tested on Texture Analyzer TAXT+. A 1 inch wide strip of the laminate is then cut. The one inch strip of the laminate is used in a 180° peel or adhesion test conducted according to a modified version of ASTM Standard D3330-90. An Instron tensile testing machine is used to measure the force required to peel 4-6 inches of a one inch-wide strip of film from the optical substrate. The test speed or pull direction is selected as: 5 mm/sec.

Table 2 - Aged Adhesion Results

Sample ID	Adhesion at Room Temperature (g/in.)	Aged Adhesion at 60C (g/in.)
Roll 4 (Ex. 2)	1.84	2.908
Roll 3 (Comp. Ex. 2)	0.596	0.168

Additional Adhesion Testing on Polycarbonate Substrate

[0033] Additional substrate adhesion testing was accomplished by laminating a one inch strip of the film onto a polycarbonate substrate with a 10 lb silicone rubber roller. The laminate is conditioned at ambient condition ("RT"), 60 °C, and 90 °C for 5 minutes. The laminate is allowed to sit at room temperature for 2 hours before peel testing was completed. Peel testing is done at a rate of 12 inches/minute and in a 180 degree configuration in a Texture Analyzer machine model TAXT+ . Results are shown in Table 3.

Table 3 Adhesion Test Results

				Adhesion (g/in.)		
	Adhesion Layer Constituents (weight% of layer)			2-hrs at RT	5 min at 60C	5 min at 90C
Ex. No.	MD6951	HDPE 6018	LDPE NA217			
Ex. 3	15	35	50	1.3	6.7	20.9
Ex. 4	25	35	40	9.9	24.5	33.5
Ex. 5	33	35	32	23.3	47.2	55
Ex. 6	50	25	25	70	170.7	131.8
Ex. 7	60	30	10	134.6	324.4	beyond instrument level
Ex. 8	80	10	10	202.7	499.7	beyond instrument level
Ex. 9	90	0	10	383.4	503.8	beyond instrument level
	MD6951		LDPE NA217	2-hrs at RT	5 min at 60C	5 min at 90C
Ex. 10	80		20	126.6	161	211.9

Self-Adhesion Testing

[0034] The expected unwind behavior of a particular film can be assessed through self-adhesion testing. As used herein, self-adhesion refers to the adhesion between the free surface of the adhesion layer of a film to the free surface of the release layer of the same film. Self-adhesion of films according to certain embodiments of the invention was tested by pressing two sheets of film laminates in a Carver Press under 2000 psi for 1 hour. The film laminates were placed so that the adhesive layer of one laminate sheet is in contact with the release or skin layer of the other laminate sheet. Peel testing was then done at a rate of 12 inches/minute and in a 180 degree configuration in a Texture Analyzer machine model TAXT+.

[0035] Examples 7, 9, and 10 of Table 3 were placed into the Carver Press. The results are shown in Table 4.

Table 4 Self-Adhesion Test Results

	Adhesion Layer Constituents (weight% of layer)			Peel Force (g/in.)
Ex. No.	MD6951		LDPE NA217	
Ex. 10	80		20	6.0±1.1
	MD6951	HDPE 6018	LDPE NA219	

	Adhesion Layer Constituents (weight% of layer)			Peel Force (g/in.)
Ex. No.	MD6951		LDPE NA217	
Ex. 7	60	30	10	3.9± 1.4
Ex. 9	90	0	10	36.6 ± 3.1

Testing - Residue

[0036] Example 10 of Table 3 was tested for residue after being laminated to clean, poly(methyl methacrylate) (PMMA) substrates (which were warmed to 60 °C) using darkfield detection and image analysis. Nine samples of the Example 10 material and a comparison material having an adhesion layer formed from 100% MD6951 were tested at each of three adhesion conditions: (a) adhesion for 18 hours at room temperature, (b) adhesion for 18 hours at 60 °C, and (c) adhesion for 5 minutes at 90 °C. Figure 4 shows the darkfield images produced from residue tests for Example 10 for adhesion conditions (a), (b), and (c). Figure 5 shows the enhanced images derived from the images of Figure 4. Similar images were used to determine residue percentages for the material at each condition. The results are tabulated in Table 5. On average, the Example 10 samples tested for 18 hours at room temperature left only 1.8% residue, the samples tested for 18 hours at 60 °C had only 0.8% residue, and the samples tested for 5 minutes at 90 °C had only 1.8% residue.

Table 5 Residue Test Results

Residue Percentage Surface Coverage Detected by Image Analysis			
{80/20} MD6951/LDPE {NA217} Blend	18 Hours @ Room Temp	18 Hours@ 60C	5 Minutes@ 90C
Sample 1	2.3	1.3	1.6
2	1.6	0.6	0.6
3	2.1	0.4	1.7
4	0.7	0.3	4.5
5	2.6	0.8	1.4
6	1.5	0.4	1.6
7	2.0	0.2	1.5
8	1.7	1.8	1.9
9	1.7	1.5	1.8
Average%	1.8	0.8	1.8

[0037] Figure 6 shows darkfield images produced from residue tests of the comparison material with an adhesion layer of 100% MD6951 at the same three conditions. Figure 7 shows the enhanced images derived from the images of Figure 6. Similar images were used to

determine residue percentages for the material at each condition. The results are tabulated in Table 6. On average, comparison samples tested for 18 hours at room temperature had 1.7% residue, the samples tested for 18 hours at 60 °C had 1.5% residue, and the samples tested for 5 minutes at 90 °C had 1.7% residue.

Table 6 Residue Test Results

Residue Percentage Surface Coverage Detected by Image Analysis			
100% Pure MD6951	18 Hours @ Room Temp	18 Hours@ 60C	5 Minutes@ 90C
Sample 1	2.7	1.1	1.9
2	0.9	1.4	2.9
3	1.0	1.3	1.6
4	2.5	1.5	1.6
5	1.3	1.3	1.6
6	1.9	1.8	1.7
7	1.3	1.8	1.0
8	1.3	1.8	1.4
9	2.5	1.1	1.7
Average%	1.7	1.5	1.7

[0038] The residue images in Figures 4 and 5 demonstrate that the residue average resulting from 18 hours at 60 °C for Example 10 is decreased as compared to the residue average resulting for 18 hours at 60 °C for a sample that includes 100% MD6951 (Figures 6 and 7).

[0039] The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range surrounding that value. For example, a dimension disclosed as "40 mm" is intended to mean "about 40 mm." Further, to the extent that any meaning or definition of a term in the present application conflicts with any meaning or definition of the same term in a cited document, the meaning or definition assigned to that term in the present application shall govern.

[0040] While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the invention as claimed. It is therefore intended to cover in the appended claims all such changes and modifications that are within their scope.

REFERENCES CITED IN THE DESCRIPTION

Cited references

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- [WO2013021788A1 \[0004\]](#)
- [JPH09143439A \[0004\]](#)
- [US2014335334A1 \[0004\]](#)
- [WO2014168069A \[0004\]](#)
- [US7439301B \[0012\] \[0014\]](#)
- [US7348376B \[0012\] \[0014\]](#)
- [US20130225020A \[0012\] \[0014\]](#)
- [WO2014087814A \[0012\] \[0014\]](#)
- [WO302014087815A \[0012\]](#)
- [WO2014087815A \[0014\]](#)

Krav:

1. En film bestående af:
et frigørelseslag, der definerer en første, ydre filmoverflade; og
et vedhæftningslag, der definerer en anden, ydre filmoverflade modsat den første, ydre filmoverflade, idet vedhæftningslaget består af en hydrogeneret
5 styrenblokcopolymer med et styrenindhold på mellem 30% til 40% efter vægt
hvor den hydrogenerede styrenblokcopolymer ligger mellem 15% til 80% vægt af vedhæftningslaget, og vedhæftningslaget omfatter yderligere 10% til 50% efter vægt polyethylen med lav tæthed og 10% til 35% efter vægt polyethylen med høj tæthed.
10
2. Filmen i krav 1, hvor den hydrogenerede styrenblokcopolymer ligger i et interval på 25% til 50% efter vægt af vedhæftningslaget, og vedhæftningslaget omfatter yderligere 25% til 40% efter vægt polyethylen med lav tæthed og 25% til 35% efter vægt polyethylen med høj tæthed.
15
3. Filmen i krav 2, hvor den hydrogenerede styrenblokcopolymer ligger på 33% efter vægt af vedhæftningslaget, og vedhæftningslaget omfatter yderligere 32% efter vægt polyethylen med lav tæthed og 35% efter vægt polyethylen med høj tæthed.
- 20 4. Filmen i krav 1, hvor frigørelseslaget omfatter mindst et sæt bestående af polyethylen med lav tæthed, lineær polyethylen med lav tæthed, polyethylen med høj tæthed, polyethylen med medium tæthed, polypropylen, tilfældig copolymerpolypropylen, heterofasiske copolymerer, metallocen, lineær polyethylen med lav tæthed.
25
5. Filmen i krav 4, hvor frigørelseslaget består af polyethylen med lav tæthed og polyethylen med høj tæthed.
6. Filmen i krav 5, hvor frigørelseslaget omfatter 40-60% efter vægt polyethylen
30 med lav tæthed og 40-60% efter vægt polyethylen med høj tæthed.

7. Filmen i krav 1 omfatter yderligere et kernelag mellem vedhæftningslaget og frigørelseslaget.
- 5 8. Filmen i krav 7, hvori kernelaget består af et sæt bestående af polyethylen med lav tæthed, polyethylen med høj tæthed og en blanding af polyethylen med lav tæthed og polyethylen med høj tæthed.

DRAWINGS

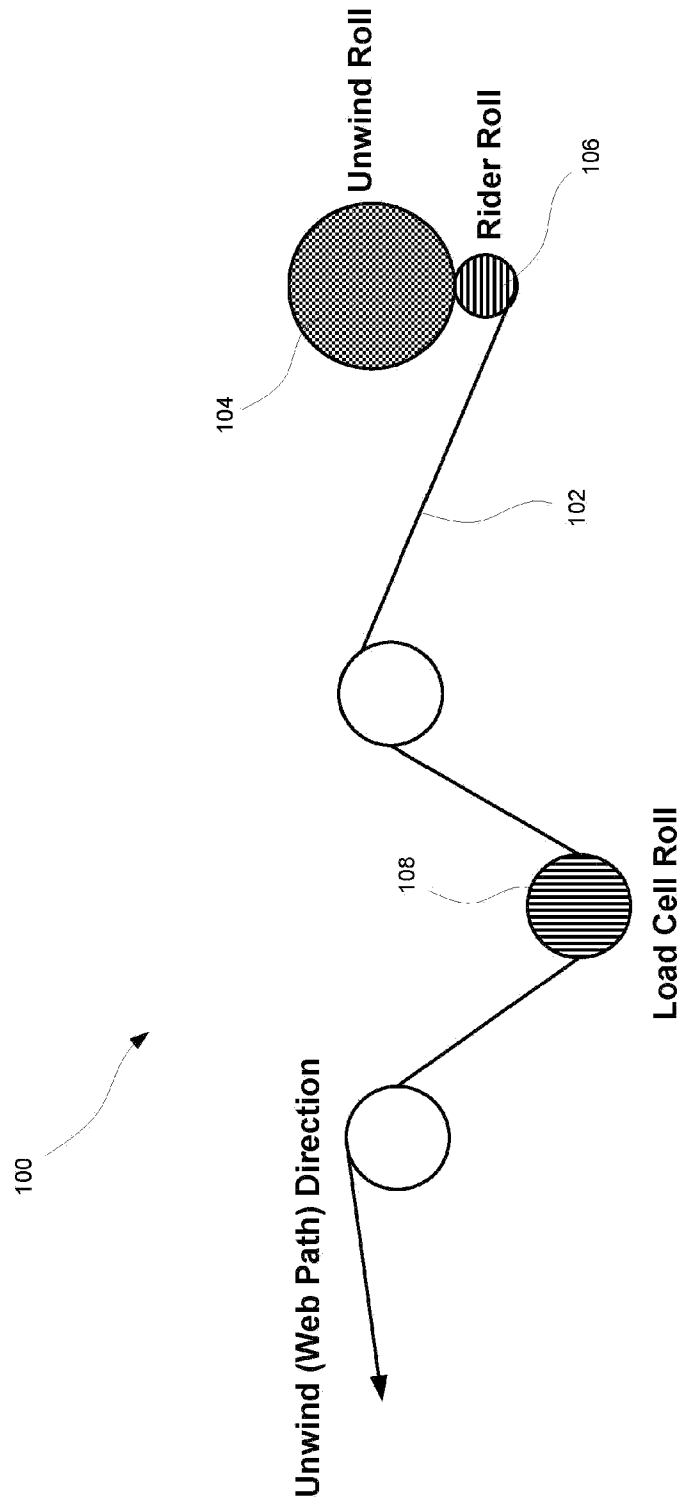


FIGURE 1

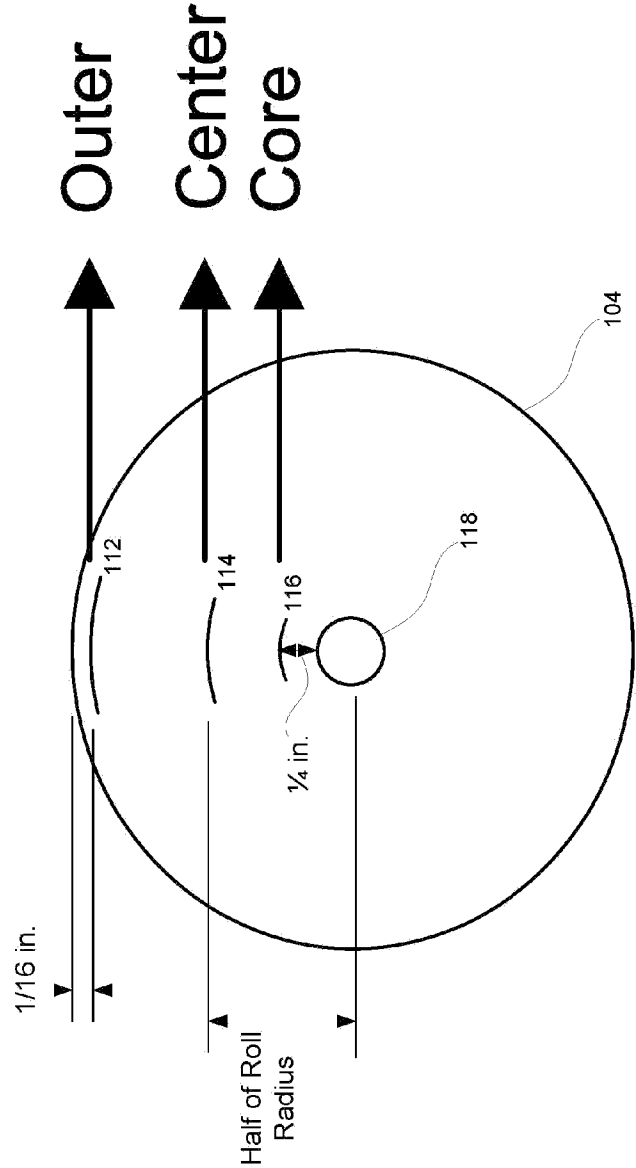


FIGURE 2

Unwind Tension (lb-f) at 3 Different Roll Locations

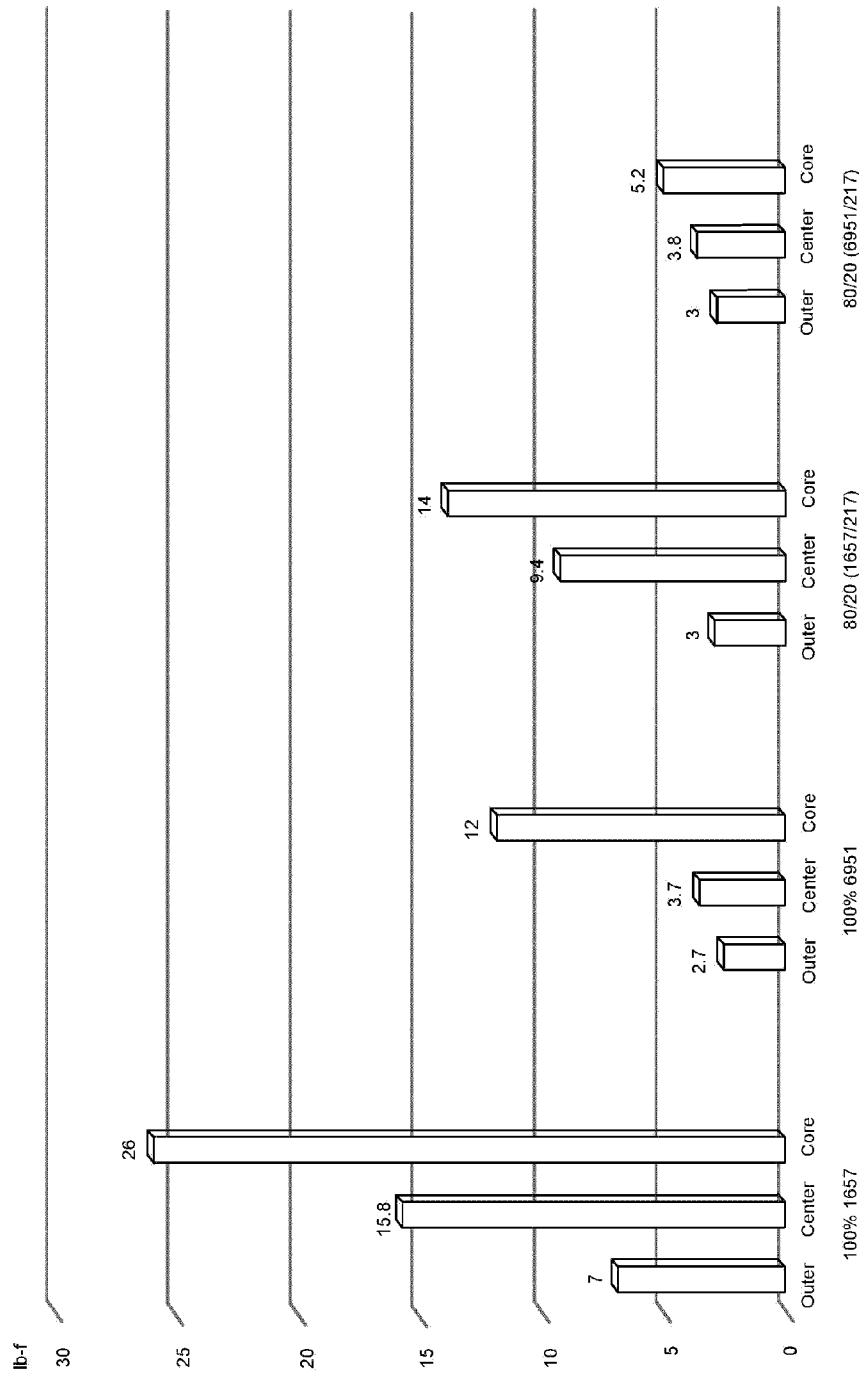


FIGURE 3

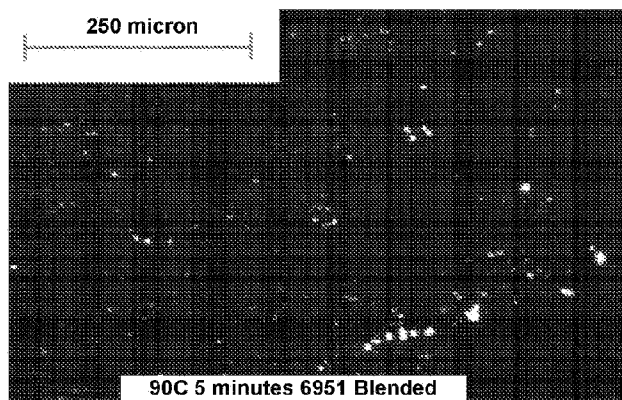
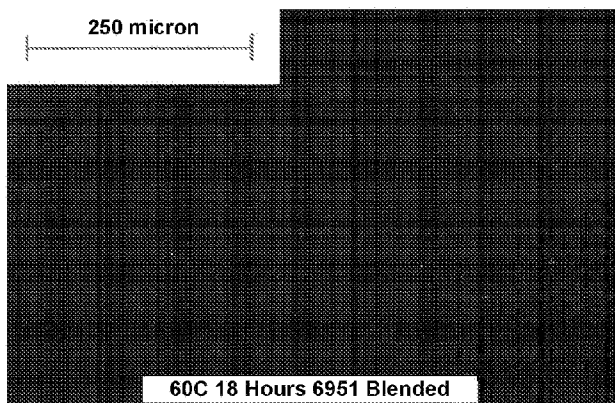
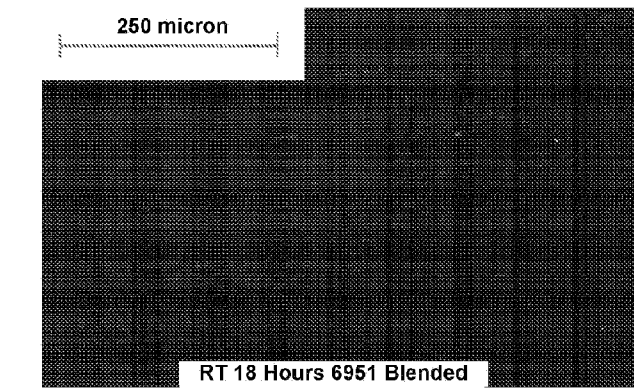


FIGURE 4

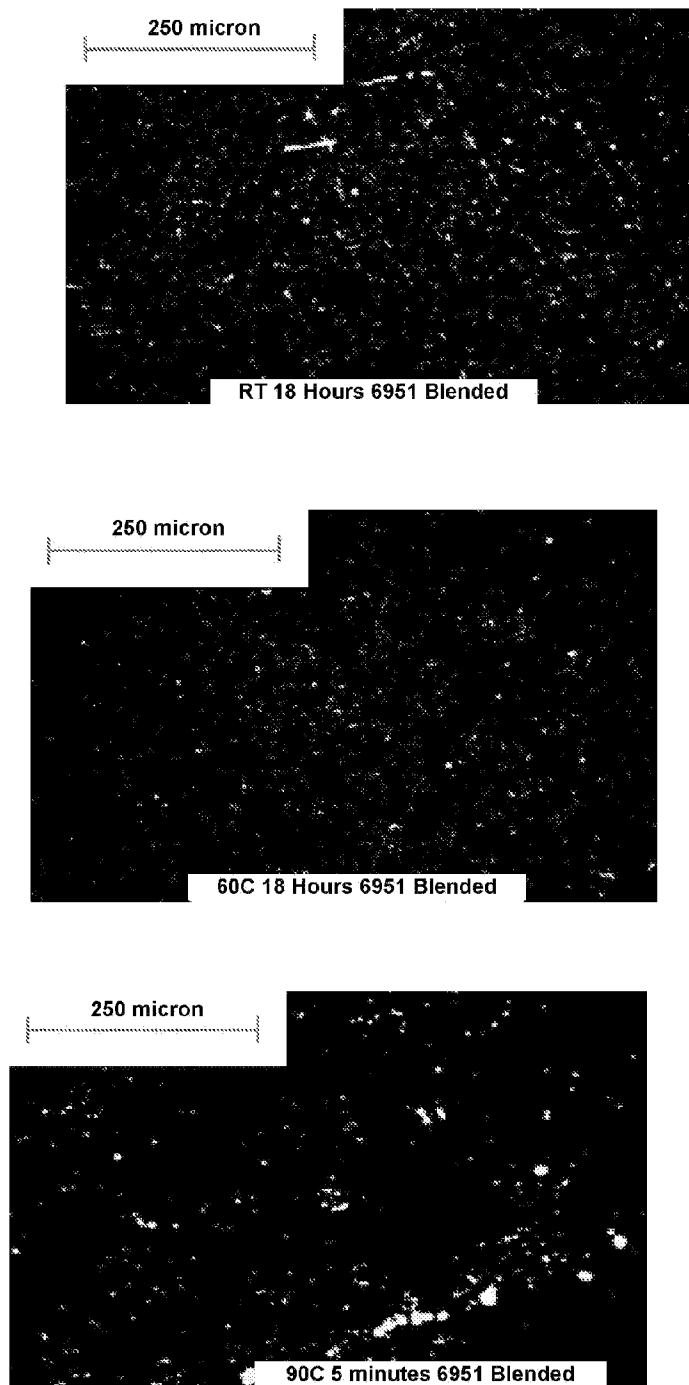


FIGURE 5

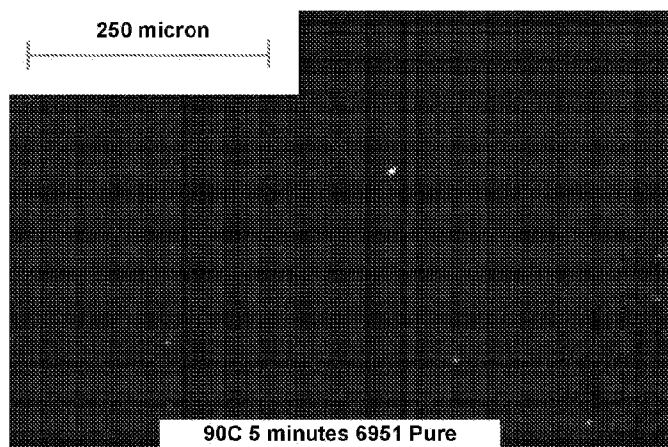
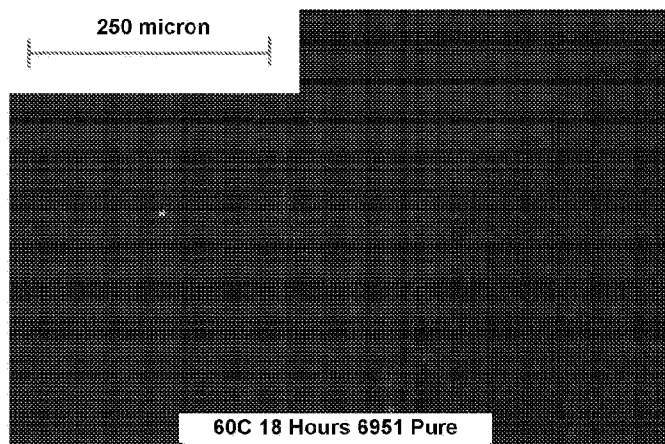
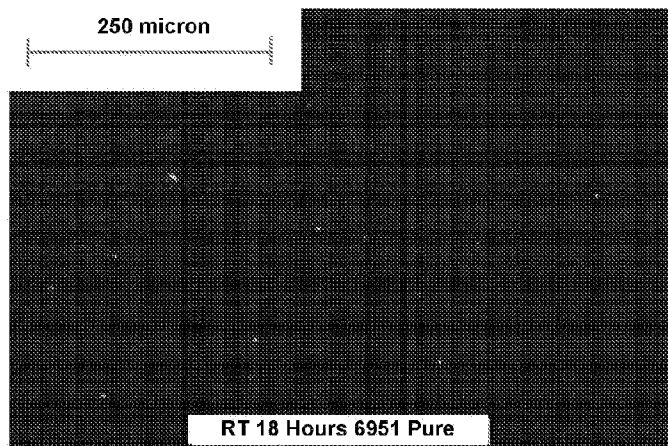


FIGURE 6

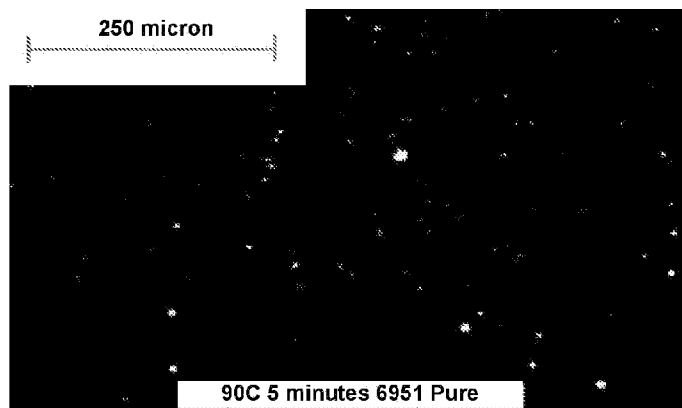
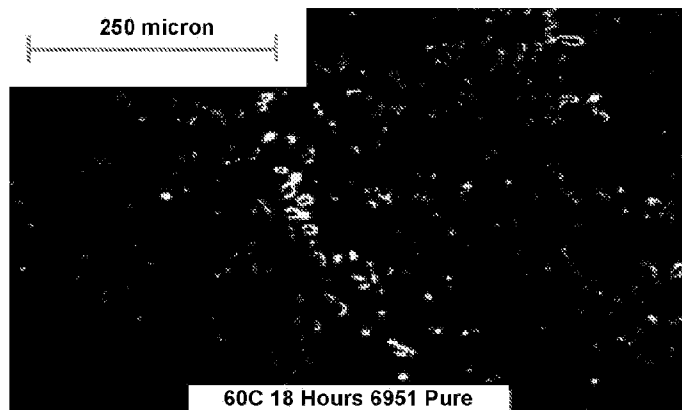
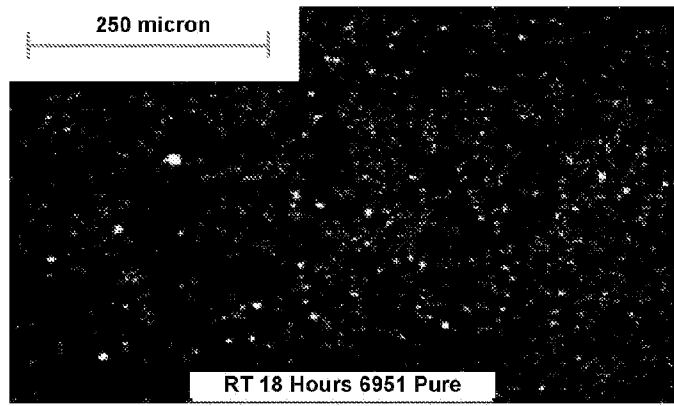


FIGURE 7