I. INTRODUCTION

Nanoparticles (NPs), which comprise atoms numbering from several tens to thousands, are of great interest in scientific research and technological applications due to a strong correlation between their size and their electronic behavior. Stable, controllable, and monodispersed NPs offer an opportunity to explore the dimensionally dependent characteristics of materials, such as optical performance, surface effect, and structural dynamics. The various properties of NPs have therefore attracted continuously much intriguing research.

Photoemission spectroscopy is a powerful tool to investigate the electronic structure of nanomaterials. The electron energy measured in a photoemission experiment is, in principle, influenced by effects in both the initial and final states. The initial-state effect is related to the intrinsic electronic properties of materials, while the final-state effect is ascribed to the photohole in the final state and the possible interaction between the outgoing electron and the positively charged state left behind. This charging effect in the final state is related to a quantum phenomenon in single-electron tunneling.

Photoemission spectra of low-dimensional metal clusters dependent on their size have long attracted much attention. A simple dynamic model that takes into account the finite lifetime of the photohole provides a quantitative interpretation for the photoemission spectra of metallic clusters near the Fermi level; furthermore, the magnitude of the lifetime has been found to depend on the interaction between cluster and substrate. In contrast, a detailed investigation of the energy shift, dependent on size, of core-level and valence-band photoemission spectra for semiconductor NPs is lacking. In their pioneer work Colvin et al. discussed the relative shift in the valence band among NPs. Other authors subsequently concentrated on the quantum shift, relative to the bulk, of the initial state in the valence-band edge by subtracting the core-level shift. In order to decrease the problem of charging, NPs of thickness less than one monolayer on substrates were used in that work.

In our preceding report of photoemission at high resolution produced with synchrotron radiation, we showed that the core-level shift of CdSe NPs passivated with trietylphosphine oxide (TOPO)/hexadecylamine (HDA) is satisfactorily described according to a static effect in the final state involving the remaining photohole interacting with the induced polarization, and the valence-band edge shift revealed an additional shift in the initial state due to quantum confinement. Here we report additional data of CdSe NPs treated with pyridine and make a comparison with TOPO/HDA-passivated CdSe NPs. We found that the energy shifts of...
CdSe NPs treated with pyridine, which are smaller than with TOPO/HDA but still considerable, can be related to a dynamic final-state effect, in that the photohole becomes neutralized before the outgoing photoelectron escapes far due to the stronger coupling between NP and the metallic substrate and between particles. We adopt a realistic calculation that considers the inelastic mean free path (IMFP) of photoelectrons and make a comparison with our preceding analysis that involved an assumption of an effective photohole position. Our analysis provides a quantitative understanding of the measured photoemission shift for organically passivated semiconductor NPs. We demonstrate also that the charging effect in the final state in thin multilayer films can be studied in detail without being affected by the macroscopic charging observed in thick insulator samples.

II. EXPERIMENTS

CdSe NPs were synthesized according to a literature method.\textsuperscript{16,17} Briefly, CdO was dissolved in a mixture of TOPO and HDA, and heated to about 270 °C under gaseous argon. Adequate octadecylphosphonic acid (OPA) was injected into the mixture to form the Cd precursor with a color varying from dark red to transparent. The Se solution was prepared on mixing Se pellets with trioctylphosphine (TOP) and maintained at 150 °C; TOPSe was then rapidly injected into the Cd precursor. By controlling the duration of reaction and the growth conditions, CdSe NPs passivated with TOPO/HDA of varied size were obtained. These crude products were dissolved in toluene and methanol to remove the unbound ligands and to improve the size distribution. This purification was repeated several times to achieve CdSe NPs of high quality. CdSe NPs capped with pyridine were obtained on refluxing the TOPO/HDA-passivated CdSe NPs in pyridine for 10–12 h at \( \sim 60 \) °C. After that treatment, the NPs were readily dispersed in pyridine and showed a greatly decreased photoluminescent yield.

In this work we used CdSe NPs of mean diameters \( D_m \) in a range 18–42 Å, \( D_m \) is defined by \( (D_{\text{major}} \times D_{\text{minor}}^2)^{1/3} \), in which \( D_{\text{major}} \) and \( D_{\text{minor}} \) are the major and minor diameters of the prolate particles, respectively. An aspect ratio \( D_{\text{major}}/D_{\text{minor}} \) about 1.2 indicates a nearly spherical shape of these NPs. The diameters of synthesized CdSe NPs, which have a distribution with a standard deviation of about 5\%, were determined with x-ray powder diffraction (XPD) and a transmission electron microscope (TEM). TEM images, shown in Fig. 1, reveal that NPs passivated with TOPO/HDA were well separated, while NPs treated with pyridine could touch each other. We verified also the optical-absorption spectra of samples in a solution formed on dispersing NPs less than 1 mg passivated with TOPO/HDA and pyridine in toluene or pyridine 3 mL, respectively. We found the relation between the optical-absorption features and the average size to be consistent with the literature.\textsuperscript{18}

Photoemission experiments using synchrotron radiation were performed at beamlines BL-08A1 and BL-24A1 in the National Synchrotron Radiation Research Center (NSRRC), Taiwan. BL-08A1 is a low-energy beamline with a spherical-grating monochromator (LSGM) that provides radiation with energy in a range 20–160 eV; we used it to measure valence-band and Se 3\( d \) core-level spectra. BL-24A1 is a wide-range beamline with a spherical-grating monochromator (WR-SGM) offering photons up to 1500 eV, which we used to measure Cd 3\( d \) core-level spectra.

All NP samples for the photoemission experiments were prepared on casting a dilute solution with dispersed NPs onto a tantalum foil; the solvent (toluene or pyridine) was allowed to evaporate under gaseous nitrogen. The samples were then transferred to the photoemission chamber within less than 10 min to minimize the possibility of oxidation. The photoemission spectra of samples thus prepared showed no oxidized components from contamination. Pure CdSe powder
spectra were fitted with a single Voigt function; hence either
the surface component is much smaller than the interior com-
ponent or the two components of the Cd 3d_{5/2} spectra cannot
be differentiated in energy.\textsuperscript{19} The spectrum of bulk solids
shows a binding energy 405.6 eV, in agreement with a value
reported in the literature.\textsuperscript{20} For NP samples the Cd 3d_{5/2}
spectra are shifted toward higher binding energy relative to
the bulk crystallite. The energy shift of NPs passivated with
TOPO/HDA is slightly larger than that of NPs treated with
pyridine. These spectra notably exhibit line broadening result-
ing from a finite distribution of size and an increased
structural disorder.\textsuperscript{19,21}

Figure 2(b) presents Se 3d core-level spectra taken at a
photon energy 120 eV; to probe the same depth of the
samples, this energy was chosen to obtain a kinetic energy
similar to that in the Cd 3d_{5/2} core-level spectra. A satisfac-
tory fit was achieved with one single spin-orbit split doublet
of Voigt function for the bulk sample. The obtained Se 3d_{5/2}
feature has a binding energy 54.4 eV, consistent with the
literature value.\textsuperscript{22} In contrast, two spin-orbit split doublets of
Voigt functions were necessary to obtain fits of reliable qual-
ity for all Se 3d core-level spectra of the NP samples. The
intensity of the component with higher binding energy in-
creases relative to that of the other component as the particle
size decreases.\textsuperscript{15} The component at higher binding energy is
thus assigned to the surface Se atoms of CdSe NPs because
the ratio of surface-to-bulk atoms increases with decreasing
size, while the other component arises from interior Se at-
oms. The surface component of the Se 3d core-level spectra
of CdSe NPs that exhibits a chemical shift due to unsaturated
bonds or bonding to the organic ligands is not discussed
here. In contrast, the interior Se 3d component has no chemi-
cal shift but shows a clear shift to higher binding energy rela-
tive to bulk CdSe, with the magnitude of the energy shift
comparable to that of the Cd 3d_{5/2} core-level spectra.

To examine the passivants, we measured also the P 2p and
N 1s core-level photoemission spectra. The P 2p signal of
NPs treated with pyridine was much smaller than that of
TOPO/HDA-passivated NPs, indicating that pyridine effect-
ively displaced TOPO/HDA on the NP surface. Because we
found no N 1s signal in the spectrum of NPs treated with
pyridine, most pyridine molecules left the NP surface in
vacuum, consistent with a report in the literature.\textsuperscript{23}

Figure 3 shows valence-band photoemission spectra of
the same samples as shown in Fig. 2, measured at a photon
energy 50 eV so as to have the same probing depth. We
verified that the organic compounds contribute to the spectral
region below about 5 eV. The first feature below the
valence-band edge due purely to NP sharps with the de-
creasing size of particles, reflecting an increasing effect of
quantum confinement.\textsuperscript{15} On extrapolating the linear part just
below the edge to its intersection with a linear background,
we determined the valence-band edge. For the NP samples
the valence-band edges exhibit shifts towards binding energy
greater than for the spectrum of a bulk sample. NPs treated
with pyridine possess a spectrum in the valence-band range
similar to that of NPs passivated with TOPO/HDA, but have
a smaller shift of the valence-band edge.
IV. DISCUSSION

A. Core-level shift

According to a three-step model of photoemission, an electron is excited to an unoccupied state leaving a hole; this photoelectron travels to the surface and crosses into vacuum. An electrostatic interaction arises between the outgoing photoelectron and the system left behind, and between the hole and its surrounding medium. The kinetic energy of the outgoing photoelectron is a sum of the photon energy and the system energy of the initial neutral ground state minus the energy of the final state. The integration is performed over the IMFP depending on the kinetic energy during the photoemission. The associated energy shift of the core levels with respect to bulk becomes

\[ \Delta E_{CL}(R, r_h) = E_f(R, r_h) - E_f(\infty, r_h). \]

The energy of the final state defined here arises because the organic ligands (TOPO/HDA) with long carbon chains, which passivate the NP surfaces, prevent the photohole from becoming neutralized by an electron tunneling from the metallic substrate before the photoelectron escapes far. The bulk CdSe powder sample was, however, not isolated and the photohole was readily neutralized from an electron at the Fermi energy during the photoemission. The associated energy of the final state is thus expected to be minimal, which in the following analysis allows us to take that energy as zero in the bulk limit. The practice justifies this assumption as we subsequently discuss. In the metallic limit as \( e \to \infty \), the energy of the final state approaches \( e^2/(2R) \), the classical charging energy.

In photoemission spectroscopy the primary electron signal becomes attenuated through inelastic scattering as it travels through the interior and then escapes from the surface. The measured energy shift is related to the position of the photohole weighted with an exponential factor describing that attenuation,

\[ \Delta E_{CL}(R, r_h) = \int_0^{R-\delta} r^2 dr \int d\Omega E_f(R, \tilde{r}_h) e^{-\ell(r_h)/\lambda(E_{kin})}. \]

This form ensures that the potential vanishes at infinity. If the photoelectron travels to infinity, there is no interaction between the photoelectron and the remaining system with a photohole. The energy of the final state is the energy between the photohole and its image. According to Eq. (1) and after rearrangement to facilitate numerical computation, we express the final-state energy as

\[ E_f(R, r_h) = \frac{e^2}{2R} \left( \frac{e}{e-1} \right) + \frac{e^2}{2Re} \left( \frac{e}{e+1} \right) \frac{r_h^2}{R^2-r_h^2} \]

\[ + \frac{e^2}{2Re} \left( \frac{e}{e+1} \right) \sum_{k=0}^{\infty} \frac{1}{k(e+1)+1} \left( \frac{r_h^2}{R} \right)^{2k}, \]

in which \( r_h \) is the position of the photohole residing in the NP; the factor one half denotes the self energy or the integration of \( q \) from 0 to \( e \). Brus formulated an identical expression as the loss of dielectric solvation energy. In the limit of a large particle or the bulk (\( R \to \infty \)), the first term vanishes. The second term approaches \( e^2/(4h)(e-1)/(e(e+1)) \) for \( r_h=R-h \), which is the correct form of image potential at a planar surface. The third term also vanishes as the series approaches \( \ln(R) \). As there is no electrostatic energy in the initial neutral ground state, the measured binding-energy shift of the core levels with respect to bulk becomes

\[ E_{CL}(R, r_h) = E_f(R, r_h) - E_f(\infty, r_h). \]
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FIG. 4. Energy shifts of the core levels for CdSe NPs (a) passivated with TOPO/HDA and (b) treated with pyridine vs mean diameter, compared with model calculations. The thick solid curve is the shift calculated with Eq. (5) with the position of the photohole weighted by an exponential function describing attenuation; the dotted curve is overplotted according to a simplified model with an effective photohole position for comparison. The dashed curve in (b) is the calculation based on a model for a dynamic effect in the final state that takes into account the lifetime of the photohole. The thin solid and dotted-dashed curves include the contribution from the nearest-neighboring particles with and without considering the dynamic effect in the final state, respectively.

determined according to the “TPP-2M” formula proposed by Tanuma, Powell, and Penn. 27 The physical parameters involved in this formula are the density of the solid, the number of valence electrons per atom or molecule, the atomic or molecular mass, and the energy of the band gap. We used 4.66 Å for an IMFP of CdSe obtained with a photoelectron kinetic energy about 60 eV.

Figure 4(a) plots the measured energy shifts, dependent on size, relative to the bulk for Cd 3d 5/2 and the interior component of Se 3d core-level spectra of CdSe NPs passivated with TOPO/HDA compared with a calculation (thick solid curve) according to Eq. (5), in which the values of the high-frequency dielectric constant ε of CdSe NP dependent on size are obtained from the literature. 28 The horizontal error bars specify the full width at half maximum of the size distribution of CdSe NPs, while the vertical error bars indicate the measurement uncertainty. The calculated shifts of the final state without adjustable parameters show satisfactory agreement with the measured energy shifts of the core levels for TOPO/HDA-passivated CdSe NPs with a negligible effect of the final state in the bulk. The magnitude of the Cd 3d 5/2 core-level shift for each size is in general slightly larger than that of the Se 3d levels. We attribute this phenomenon to the unresolved Cd 3d spectra with the surface Cd contribution that is weighted more to the shift of the final state. To avoid divergence in our classical model, a finite surface thickness δ must be included in Eq. (5).

A simplified model calculation undertaken in our preceding work, 15 in which we considered an effective photohole position \( r_h = R - \delta \) as the fitting parameter for the experimental data, is plotted as the dotted curve in Fig. 4(a) for comparison. A slightly poorer fit than through use of Eq. (5) is obtained with \( h = 3.6 \) Å. This value is reasonable, since it is close to the IMFP of 4.66 Å. The simpler model for an effective photohole position provides a satisfactory approximation to the more complicated spherical IMFP model.

Using the same parameters, we calculated the bulk term \( E_{bulk}^{\infty} \) considering the IMFP to be 0.088 eV, which would yield a constant downward shift of the calculated curve in Fig. 4(a), apparently deviating from the measured energy shift. If one retains the bulk term while treating the IMFP and the surface thickness as fitting parameters, the resulting fit is much poorer and has an unrealistically large IMFP and an unphysically small surface thickness. Including the bulk term in the effective photohole position model results in a poor fit with an unrealistic photohole position \( r_h = R - 0.8 \) Å. These results support our assumption to neglect the bulk effect in the final state.

Figure 4(b) shows the energy shifts of CdSe NPs treated with pyridine with respect to bulk CdSe, which are always smaller than those of NPs passivated with TOPO/HDA. To explain the energy shifts of NPs treated with pyridine, we introduce a dynamic effect in the final state: i.e., the photohole becomes neutralized when the photoelectron travels to \( R + \hbar \tau \), the potential energy transfers to the dashed curve as only the electron-image energy remains.

A schematic diagram describing the effect of a dynamic final state is illustrated in Fig. 5, in which is drawn the electrostatic potential-energy curve as the photoelectron travels to infinity at which the zero energy is defined. In the presence of the photohole, the potential energy is the sum of the electron-hole energy \( U_{e-h} \) and the electron-image energy \( U_{e-im} \) following the solid curve. If the photohole becomes neutralized when the photoelectron travels to \( R + \hbar \tau \), the potential energy transfers to the dashed curve as only the electron-image energy remains.
the potential energy of the photoelectron transfers to the
dashed curve as only the image potential energy remains. Work
needed to be done to remove the photoelectron to in-
finity is thus decreased by the interaction energy of the elec-
tron and hole, deduced from Eq. (2),
\[
E_{e-h}(R, \vec{r}) = \frac{e^2}{|\vec{r}_e - \vec{r}_h|} \left( \frac{2}{\varepsilon + 1} \right) + \frac{e^2}{r_e} \left( \frac{\varepsilon - 1}{\varepsilon + 1} \right)
+ \frac{e^2}{r_e} \left( \frac{\varepsilon - 1}{\varepsilon + 1} \right) \sum_{k=1}^{\infty} \frac{1}{k(k+1)} \left( \frac{\omega}{r_e} \right)^k \cos (\cos \theta),
\]
wherein \(\vec{r}_e = \vec{R} + \Delta \vec{r}\) with \(\Delta \vec{r} = \vec{u}_r \tau, \vec{u}_r\) is the velocity of the photoelectron
away from the analyzer, and \(\theta\) is the angle between
\(\vec{r}_e\) and \(\vec{R}\). In this model, for simplicity we take \(\tau\) as an
average relaxation time instead of the characteristic time in
the tunneling probability used in Ref. 11. When the dielectric
constant approaches infinity, the total energy shift based on
the effect of the dynamic final state is given by
\[
\lim_{\varepsilon \to \infty} \Delta E_{e-h}(R, \vec{r}) = e^2 \left[ 1/(2R) - 1/r \right],
\]
closely resembling a model proposed for metallic clusters on a graphi-
the surface.\(^{11}\)

A fit of the formula \(\Delta E_{e-h}(R, \vec{r})\) to the core-level
energy shifts of CdSe NPs treated with pyridine is shown in
Fig. 4(b) as the dashed curve. The experimental results are
satisfactorily described with our model with a parameter \(\Delta r = 68\) Å. Hence the outgoing photoelectron leaves the
NP with a kinetic energy about 60 eV for the Se 3d level,
corresponding to a velocity \(\vec{u}_r = 4.6 \times 10^6\) m/sec. The
time-of-flight of the photoelectron left in the dielectric particle is thus
\(\tau = 1.48 \times 10^{-15}\) s, but the smallest and largest NP data show
a perceptible deviation from the model fitted curve (dashed
curve).

We further consider the influence of the nearest-neighbor
ingredients as a correction because the interparticle
distances were small for NPs treated with pyridine [see Fig.
1(b)]. An additional interaction between the remaining pho-
tohole and the induced image charges in the neighboring NPs
can diminish the apparent energy shift in the final state. The
TEM images showed an arrangement of nearly hexagonal
close-packed NPs. We estimate from, for example, Fig. 1(b)
that, on average, one NP in the surface layer of a film had
four neighboring NPs nearly in contact, but maintained a
distance \(\sim 15\) Å surface to surface from the other five nearest
neighbors, because of remaining TOPO/HDA surfactants on
the NP surface.\(^{23,29}\) For simplicity we assume that the re-
maining photohole was effectively located at the center of
the NP and interacted with the induced image charges of all
nearest-neighbor NPs. The final-state shift after this cor-
rection for the nearest neighbors is shown as the dotted-
dashed curve in Fig. 4(b). An additional decrease is clearly
required, which is again due to the dynamic effect in the final
state. The best fit including both the dynamic effect in the
final state and the correction for the nearest neighbors is
plotted as a thin solid curve in Fig. 4(b), showing satisfactory
agreement with data points of all sizes. In this fit, \(\Delta r = 128\) Å or \(\tau = 2.78 \times 10^{-15}\) s. This value is expected to cor-
respond to the upper limit of the lifetime, as most photoholes
are created near the NP surface leading to a smaller contri-
bution from neighboring particles than our assumption at the
center. The interaction between photoelectron and image be-
comes small for \(\Delta r > 30\) Å, because of its dipolar character, and is almost negligible in the above-mentioned dynamic
effect and the correction due to nearest neighbors. For NPs
passivated with TOPO/HDA, the mean distance surface to
surface between NPs was \(\sim 15\) Å. The energy shifts are thus
diminished by 0.02–0.04 eV considering the contribution from
the neighbors, which is insignificant to affect our con-
clusion.

We attribute the dynamic effect in the final state of CdSe
NPs treated with pyridine to the distinct coupling strength
between NP and metallic substrate and between particles
from TOPO/HDA-passivated NPs. TOPO and HDA mol-
ecules with long carbon chains that are attached to the NP
surface via their lone pairs can prevent the particles from
being in contact. In contrast, pyridine is unstable to adsorp-
tion on the NP surface in vacuum, leaving the NP partially
uncovered with a smaller distance between the NP and the
metallic substrate and between the NPs themselves. We ex-
pect that the coupling in the latter system is stronger than
that in the former, resulting in a larger probability of electron
tunneling and a smaller lifetime of the remaining photohole
in the NP. Our estimate shows the photohole lifetime to be in a
range \(1.5 \sim 2.8 \times 10^{-15}\) s for NPs treated with pyridine. Ac-
cordingly the dynamic effect of the final state cannot be ig-
ored for a system with strong interactions between a dielec-
tric particle and a metallic substrate and between particles.
For comparison, the dynamic effect in the final state has been
observed in naked Ag NPs (Ref. 11) and in Ag and Au NPs
passivated with alkanethiolate supported on a graphite
surface\(^{12,13}\) with a photohole lifetime \(0.2 \sim 0.5 \times 10^{-15}\) s,
smaller than our case.

**B. Valence-band edge shift**

The measured shifts of the valence-band edge of CdSe
NPs passivated with TOPO/HDA are presented in Fig. 6(a).
The valence-band edge shift is invariably larger than the
core-level shift. The final-state shift of the valence-band edge
is expected to differ from that of the core levels because the
wave function of the valence-edge hole penetrates more
deeply into the interior of the NP.\(^{23}\) Following Ref. 24, we
model the hole 1S wave function with, for simplicity,
\(\sin(\pi r/R)\) as in an infinite well. This approximation was
used in Ref. 28 to obtain the dielectric constant and exciton
energy. The average shift of the final state with the same
IMFP is shown as the dotted curve. For a step well the hole
wave function is expected to have greater weight near the
surface. The pure effect of the final state evidently underes-
timates the valence-band edge shift. We expect an additional
shift due to quantum confinement as the effect of the initial
state. The simplest form is the kinetic-energy term in a quantum-confinement well as described by Brus,\(^{30}\) which has
been used with qualitative success to explain the shift in CdS
NPs.\(^{1}\) Wang and Zunger calculated the electronic structure of
Figure 6(b) shows the valence-band edge shifts of CdSe NPs treated with pyridine compared with the calculation based on the model for a dynamic effect in the final state with the same parameters as for the corresponding core-level shift. We considered also the contribution from the nearest-neighboring particles (dotted-dashed and thin solid curve). The calculation evidently overestimates the measurement. The deviation is similar to that in Fig. 6(a).

Several reasons underlie this deviation. One might suspect an approximation of a NP of prolate shape (aspect ratio 1.2–1.3) by a sphere to result in an error in the initial-state calculation, but the deviation of a prolate spheroid from the averaged sphere invariably produces an increase of quantum confinement and a larger shift in the initial state, contrary to the experimental observation. Another point is the presence of stacking faults in NP that have been experimentally observed, but a calculation of CdSe quantum dots in both wurtzite and zinc-blende structures shows no significant alteration of exciton energies. Stacking faults thus seem unable to explain such a discrepancy. A third possibility is that a lattice contraction up to 1% and structural disorder were observed experimentally while the calculation assumes a truncated bulk with a bulk lattice parameter. According to a theoretical investigation of CdS clusters, the band gap is decreased 0.11 eV assuming 1.4% lattice contraction. This effect might explain the discrepancy between experiment and theory.

V. CONCLUSION

We have investigated the energy shift dependent on the size of the core levels and valence-band edge for organically passivated CdSe NPs of various diameters. For NPs passivated with TOPO/HDA the core-level shifts are satisfactorily described with a model of a static final state based on an electrostatic interaction between the photohole and the dielectric background, including the inelastic mean-free-path effect of the photoelectron, with no adjustable parameter. For NPs treated with pyridine, the energy shifts are smaller. A dynamic model characterizing the finite lifetime of the photoelectron on a femtosecond scale is invoked to elucidate these energy shifts considering the interaction between photoelectron and photohole. For the valence-band edge an additional shift in the initial state due to quantum confinement is required to explain the data. A pseudopotential calculation of the valence-band edge shift in the literature appears to overestimate the effect in the initial state compared to our experimental observation. The discrepancy might reflect a lattice contraction in the NP that was neglected in the calculation.

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If the dilute solution to prepare thin multilayer films.

To avoid this unwanted phenomenon, we used a highly dilute solution to prepare thin multilayer films.