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最近の主な研究成果

中村新男

これまでの研究成果のうち、豊田理化学研究所で行っている研究に関連するカーボンナノチューブと金属ナノ粒子の最近の成果を紹介する。

１．カーボンナノチューブの光物性の研究

カーボンナノチューブはグラファイトのシート（グラフェン）を円筒状に丸めた物質であり、炭素原子のみからなる直径 1~5nm、長さ約 1mm の微小なチューブである。丸め方（カイラリティ）によって半導体または金属の物性を示す。単層カーボンナノチューブの電子状態は擬一次元的であるため、バンド端近傍で状態密度の発散（van Hove 特異点）と強い励起子効果を示し、特徴的な光学応答が現れることが知られている。励起子とは、光励起によって伝導帯と価電子帯に生成された電子と正孔がクーロン引力によって束縛された水素原子に類似の複合粒子である。

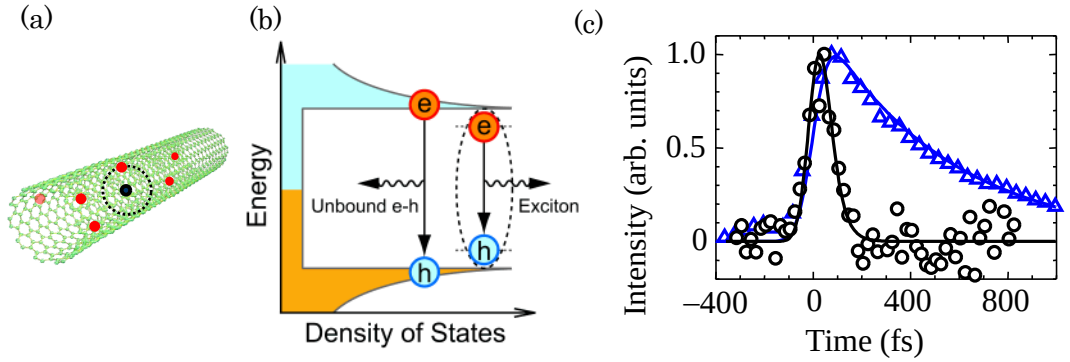
１）金属ナノチューブの励起子による発光

金属には多数の自由電子が存在するために電子－正孔間のクーロン引力が遮蔽されるので、励起子が安定には存在しないと言われている。しかし、擬一次元系では遮蔽効果が抑制されるので、金属ナノチューブにおいて励起子が存在することが理論と吸収・レイリー散乱スペクトルの解析から示されている。本研究では、多くの半導体結晶の励起子研究に用いられたフォトルミネセンス（発光）の観測から、励起子の存在とその安定性を明らかにした。

図 1(b)に示されるように、光励起された電子と正孔はそれぞれのバンドを自由に動き回りながら再結合して消滅する過程と励起子を形成して再結合する過程が存在する。図 1 (c)の△印はフェムト秒のポンプ・プローブ分光で測定された過渡吸収の減衰曲線、○印はアップコンバージョン法で測定された発光の減衰曲線を示す。過渡吸収（△印）は約 500fs の時定数で減衰する。このような減衰挙動は、レーザー光で励起された電子・正孔によって金属中の自由電子がホットな状態になり、その温度が冷却する過程を反映している。一方、発光の減衰時定数（○印）はこれに比べて 1 桁以上短く、その値は約 40fs である。この結果から、励起子は 40fs という超短時間で消滅するが、自由電子の海の中に励起子状態が安定に存在することが明らかになった[1]。

[1] T. Koyama et al., Phys. Rev. B85, 045428 (2012).

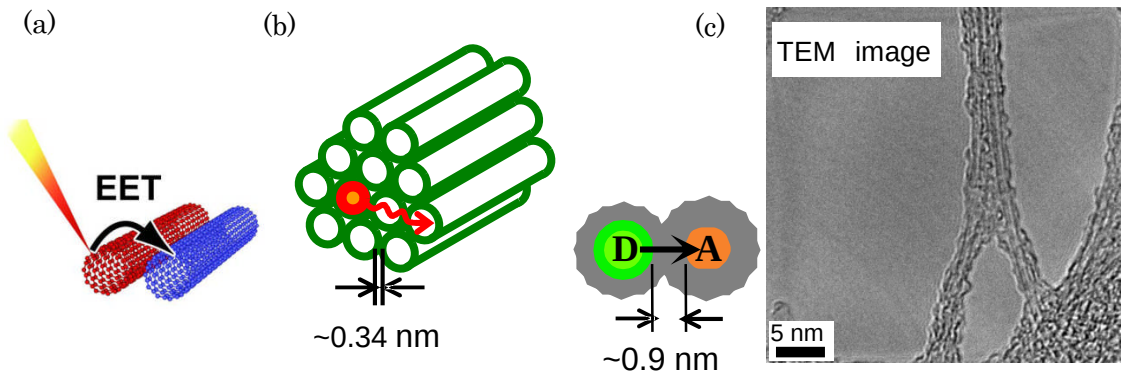
図 1　金属ナノチューブの励起子



２）カーボンナノチューブ間の励起子エネルギー移動

半導体カーボンナノチューブのバンドギャップや励起子のエネルギーは直径の逆数にほぼ比例しているので、図 2(a)に示されるように隣接したナノチューブ間で励起子が移動する励起エネルギー移動が起こる。直径の異なるナノチューブが集まるとファンデアワールス相互作用によってバンドルが形成され、界面活性剤などでラップされていないナノチューブの場合には、二次元三角格子の規則性をもったバンドルになることが知られている（図 2(b)）。このとき、ナノチューブの壁間の距離は~0.34nm である。直径が 0.6nm から 1.7nm の範囲に分布しているナノチューブからなるバンドルの場合、発光の減衰時間は細いナノチューブでは~35fs、太いナノチューブでは~380fs になる。直径分布を考慮した減衰時間の解析から、ナノチューブ間の励起子エネルギー移動レートは $1.9\times10^{12}s^{-1}$であることがわかった[2]。

図２　ナノチューブ間の励起子エネルギー移動



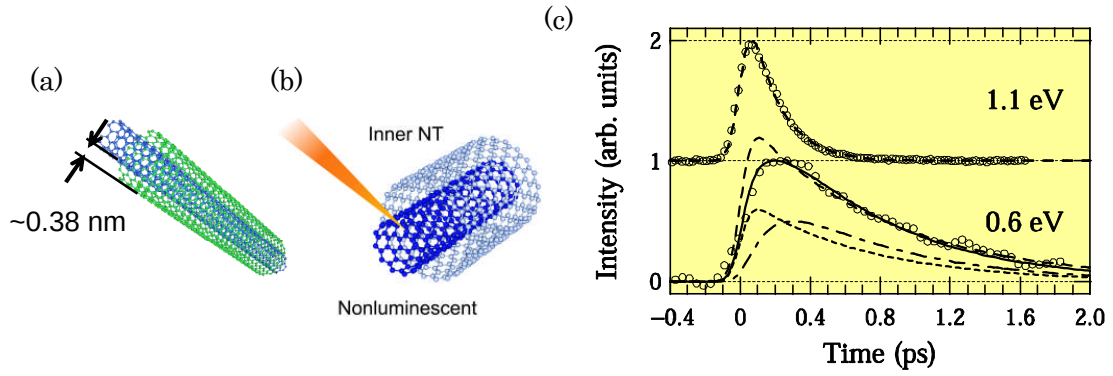
カーボンナノチューブを界面活性剤や高分子でラップすることにより、バンドル化を抑えてナノチューブを孤立させることができる。しかし、このような試料においても数本のナノチューブからなる小さなバンドルが形成される場合がある。PFO (poly(9,9-dioctylfluorenyl-2,7-diyl) でラップされたナノチューブをペーパー状にした試料では、図 2(c)の透過電子顕微鏡像に示されるように、２本または数本のナノチューブが隣接してバンドルを形成する。この場合のナノチューブ壁間の距離は~0.9nm である。発光の減衰時間は直径に依存して 1~4ps となり、二次元三角格子を組むナノチューブの場合に比べて約 1 桁長くなる。励起子エネルギー移動レートは $2.7\times10^{11}s^{-1}$となり、この値はむき出しのナノチューブがバンドルを組んだ場合に比べて約 1/7 の値である[3]。

[2] T. Koyama et al., J. Phys. Chem. Lett. 2, 127 (2011).
[3] T. Koyama et al., J. Phys. Chem. Lett. 1, 3243 (2010).

直径が異なる２本のナノチューブが同軸シリンダー構造になったナノチューブは、二層カーボンナノチューブと呼ばれ、内層チューブと外層チューブ間の距離は~0.38nm である。半導体ナノチューブからなる二層ナノチューブの場合、内層ナノチューブのバンドギャップが大きいので、内層ナノチューブから外層ナノチューブへの励起子エネルギー移動が期待される。一方、外側のチューブで覆われた内層チューブは分子の吸着や欠陥の導入が抑えられるので、非常に強い発光が生じるという実験結果も報告されている。図 3(c)は内層ナノチューブからの発光（1.1eV）と外層ナノチューブからの発光（0.6eV）の減衰曲線である。内層ナノチューブの励起子発光は 170fs

で減衰するのに対し、外層ナノチューブの減衰曲線は 160fs で立ち上がり、960fs で減衰する。外層ナノチューブの立ち上がり時間が内層ナノチューブの減衰時間と一致することから、チューブ間の励起子エネルギー移動が高速で起こることがわかった。その移動レートは $6.3\times10^{12}\,s^{-1}$であり、ナノチューブが 0.34nm の距離で隣接しているバンドルの場合に比べて、約 3 倍大きい値である[4]。このような超高速のエネルギー移動のために、内層ナノチューブの発光は強く quench されることになる。これまでの研究では、内層ナノチューブが強い発光を与えるという報告と光るという内層チューブは試料中に残存する単層ナノチューブであるという矛盾する 2 種類の報告があり、論争となっていた。しかし、この研究から試料作製のプロセスにおいて二層ナノチューブから内層ナノチューブが飛び出しそのナノチューブが発光を与えることが明らかになった[4]。

図 3　二層ナノチューブのエネルギー移動



一般に、励起エネルギー移動は Förster モデルまたは Dexter モデルで理解されている。点双極子近似を用いた Förster モデルでは、ドナーとアクセプターになる分子間の距離に比べて、励起状態の広がりが十分に小さいとして点双極子近似を用いてその公式が与えられている。カーボンナノチューブの場合、励起子広がりが約 2nm であるのに対して、バンドルや二層ナノチューブにおけるナノチューブ壁間の距離は 1nm 程度であるので、双極子－双極子相互作用によるエネルギー移動のモデルは適当ではないと言える。励起状態の広がりを考慮した distributed transition monopole 近似による Förster 型エネルギー移動モデルがカーボンナノチューブにおける励起子エネルギー移動の解析に妥当であることがこの研究から示唆された[5]。

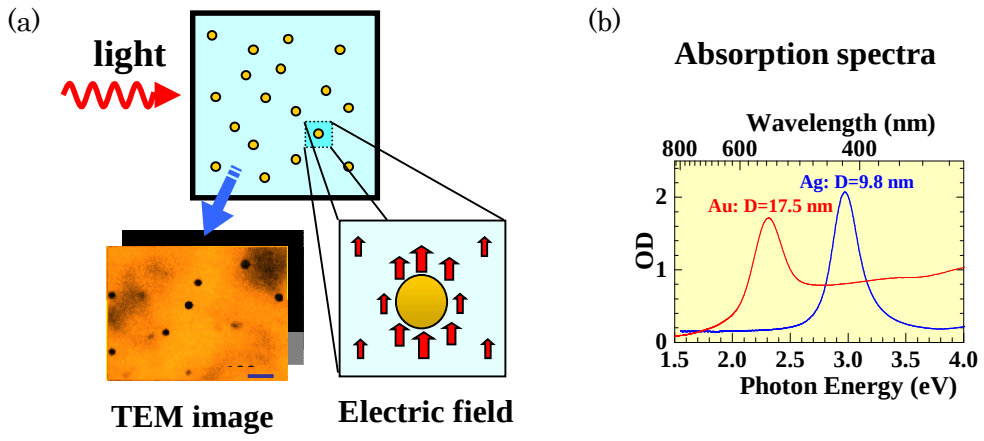
[4] T. Koyama et al., ACS Nano 5, 5881 (2011).
[5] T. Koyama et al., Phys. Chem. Chem. Phys. 14, 1070 (2012). Perspective Paper

２．金属ナノ粒子の非線形光学応答の研究

バルク状の金属が示す色は、金属中の自由電子のプラズマ振動数よりも低い振動数の光が全反射することによる。このような金属が数ナノメートルの粒子としてガラス中に分散されたガラスは、ステンドガラスとして知られているように美しい色を呈する。図 4 に示されるように、金属ナノ粒子の表面プラズモンによってナノ粒子周辺の光電場が増強され、特定に色の光に対して金属ナノ粒子分散ガラスの吸収が共鳴的に増大するために、ステンドガラスのような色が生ずる[6]。

[6]中村新男ら、Bull. Cluster Sci. Tech. 2, 135 (1998).

図４　金属ナノ粒子の表面プラズモンによる電場増強効果



１）金属ナノ粒子分散ガラスの非線形光学応答の研究

光電場の局所的な増強効果によって、蛍光やラマン散乱光の増強、非線形光学応答の増大などが期待される。屈折率や吸収係数の光電場依存性をもたらす３次非線形光学応答の感受率$\chi^{(3)}$が、金・銀・銅ナノ粒子のサイズ、形状、密度に依存して増大することを明らかにする研究を行った。粒子サイズが 2~50nm の範囲では、サイズの増加に対して非線形感受率$\chi^{(3)}$は増大する[7]。非線形性の応答時間は金属電子の冷却時間によって決まり、0.7~5ps の高速応答を示す[8]。また、ナノ粒子密度を高くすると、ナノ粒子間の相互作用による増大効果が現れてくる[9]。三角柱の形状をした銀ナノ粒子の場合、三角柱の頂点周辺に電場が集中すること、表面プラズモンの双極子モードに加えて四重極子モードによる非線形感受率が増大することが明らかになった[10]。

[7] 代表的な論文として、K. Uchida et al., J. Opt. Soc. Am. B11, 1236 (1994).

[8] 代表的な論文として、T. Tokizaki et al., Appl. Phys. Lett. 65, 41 (1994).

[9] Y. Hamanaka et al., Appl. Phys. Lett. 84, 4938 (2004).

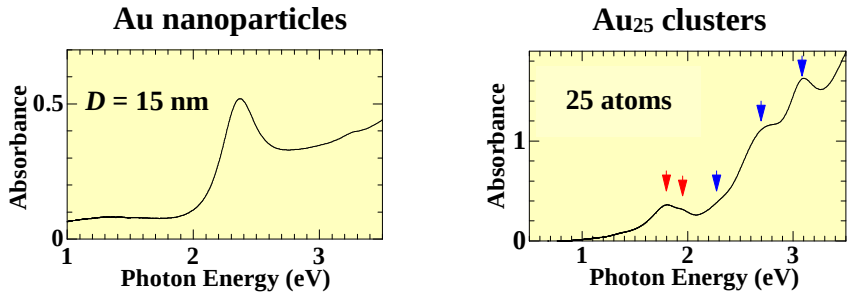
[10] N. Okada et al., J. Phys. Chem. B108, 8751 (2004).

２）非線形光学応答における表面プラズモン効果から量子サイズ効果へのクロスオーバー

金属ナノ粒子のサイズを 1nm 以下にすると、サイズが電子のフェルミ波長と同程度になるので量子サイズ効果が現れ、電子状態はバンドから離散的な状態に移り変わる。この電子状態の変化に対応して非線形光学応答の起源が変わり、非線形感受率は異なるサイズ依存性を示すことが明らかになった。チオール単分子膜で表面を修飾することによって原子数が 13 個、25 個などの金原子からなるクラスターを合成することができる。大きさの異なる数種類のチオールを用いてAu_{25}からAu_{835}までの金クラスター（対応する直径：0.9~3.0nm）を合成し、3.9~17.5nm のサイズをもつ金ナノ粒子をスパッタリング法によって作製した。

図 5 に示されるように、直径が 15nm の金ナノ粒子の吸収スペクトルには表面プラズモン共鳴による吸収帯が観測される。一方、Au_{25}クラスターでは、複数の吸収帯が観測され、表面プラズモンの吸収は現れない。Au_{25}クラスターの構造は、Au_{13}のコアと－SR－Au－SR－Au－SR－(R=thiolate)のリガンドから構成されることがアメリカのグループによる最近の研究から明らか

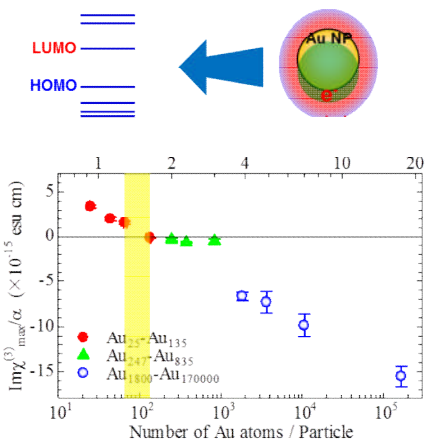
図５　金ナノ粒子とクラスターの吸収スペクトル



になっている。理論計算の結果から、クラスターで見られる吸収は HOMO－LUMO 遷移など分子軌道準位間の遷移に同定される。このような離散準位間の遷移吸収は Au_{66} クラスターまで見られるが、Au_{135} クラスターでは表面プラズモンによる吸収が現れ、Au_{247} クラスターでは表面プラズモン吸収が支配的になる[11]。

図 6 に示されるように、非線形感受率$\chi^{(3)}$は表面プラズモン共鳴の領域では負の符号であり、原子数、すなわちサイズの減少とともに値は減少する。直径が 2~3nm のサイズ領域では$\chi^{(3)}$値は非常に小さくなるが、それ以下の領域では符号が反転して正の値になりサイズの減少とともに値は増加する。およそ 135 個の原子数がクロスオーバーの値になる。分子的な電子状態になるクラスターの領域では、光励起が Au コアとリガンドとの電子的・幾何学的相互作用をわずかに変化させるために非線形な光学応答が生じると理解される[11]。

図６　非線形感受率のサイズ依存性



[11] Y. Hamanaka et al., J. Phys. Chem. C116, 10760 (2012).

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