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Novel C_{60} Compounds: Hidden Surface Science

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Novel C_{60} Compounds: Hidden Surface Science*

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Among the lattice of C_{60} in Na_3HC_{60}, the three component molecular superconductor, the hydride ions, $H^{-\delta}$, are expected to form a conduction band. In $C_{60}X$(X=He, Ar, Kr), it could be inferred that some bonding state exists between C_{60} and rare gases intercalated C_{60} lattice. These works may suggests that a unique bonding field is generated in the molecular spaces. These studies of various phenomena in these field will be able to develop the new science. We would like to name the research filed as "Hidden Surface Science"

Keywords: Hidden Surface Science; Rare Gas Compounds; Molecular Semiconductors

INTRODUCTION

We have been studying the electrical conduction phenomena of organic solids since 1948. [1] Our research was concentrated in the years from 1948 to the end of 1970's on organic electrical conductors composed of a single organic component and on those of two components derived from charge transfer between the components. The study of the electrical conductivity in a graphite-alkali metal-hydrogen system by Sano and Inokuchi in 1979 helped to extend our work on organic conductors to three-component systems. [2]

A hydrogenated potassium-graphite intercalation compound ($C_8KH_{2/3}$), one of three-component graphite-intercalation compounds, contains three-layer structures made of K^+-H^--K^+ between graphite layers. The layer of H^- in the

* Dedicated to Prof. E. A. Silinsh for his Memorial Issue.

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K^+-H^--K^+ sandwich is deduced to be responsible for the electrical conduction of the compound from the results obtained by the electrical conductivity measurement and also NMR observation. [3] The conductivity of the hydride ion layer seems to originate from a unique energy state characteristic of the interlayer of graphite. These results suggest the availability of intermolecular spaces such as interlayers for a reaction field. Thus we have developed our research on the subjects of the three-component superconductors using intermolecular spaces of solid C_{60} and the reaction of rare gases with C_{60}.

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THREE COMPONENT MOLECULAR SUPERCONDUCTORS

Na_3C_{60} is a hopeful candidate of superconductor coming after K_3C_{60} and Rb_3C_{60}, but it is not superconducting (SC). The reason is understood by the phase separation into Na_2C_{60} and Na_6C_{60} from Na_3C_{60} at low temperature. In order to suppress the inherent phase-instability of Na_3C_{60} that small Na^+ ions occupy the large octahedral site, we tried to introduce "spacer" which fills the vacancy. We used thermal decomposition materials such as sodium azide (NaN_3) and sodium hydride (NaH), and found superconductivity in C_{60} based three component superconductors formed by Na doping and simultaneous intercalation of nitrogen or hydrogen, as shown in the following scheme. [4][5]

$$NaN_3 \text{or } NaH + C_{60} \xrightarrow[\text{vac}]{\Delta} \text{Na-X-}C_{60}(X = N, H).$$

Table I summarizes the synthetic condition and physical properties of Na-X-C_{60} (X=N, H) superconductors.

TABLE I Preparation and Physical Properties of Na-X-C_{60} (X=N, H) Superconductors

Compound	*Decomposition Material*	*Decomposition Temperature (°C)*	*Structure*		T_C *(K)*
$Na_xN_yC_{60}$	NaN_3	370–390	fcc	a=14.34 Å	13
$Na_xH_yC_{60}$	NaH	280	fcc	a=14.41 Å	15

The synthesis of $Na_xH_yC_{60}$ was done as follows: The mixture of stoichiometric amounts of NaH and C_{60} powders was loaded in a quartz tube in a dry box filled with Ar gas. Then the sample in the tube sealed under a pressure of $\sim 10^{-4}$ Pa was heated at 280 °C for 1 h in a muffle furnace.

We prepared the samples of $(NaH)_nC_{60}$ (n=1–6; n being an initial composition) and characterized them by ESR, LFS (low magnetic field microwave absorption signal) and SQUID. [6] The SC samples are obtained in n=3–4. Especially for

$(NaH)_4C_{60}$, we obtain the samples with a large SC volume fraction (V_{SC}). Among $(NaH)_4C_{60}$ samples prepared hitherto, the highest V_{SC} is 77 %. Figure 1 shows the temperature dependence of the zero-field-cooled magnetic susceptibility under H=2 G for the V_{SC}=77 % sample. [7] The onset T_C of superconductivity is found to be 15 K.

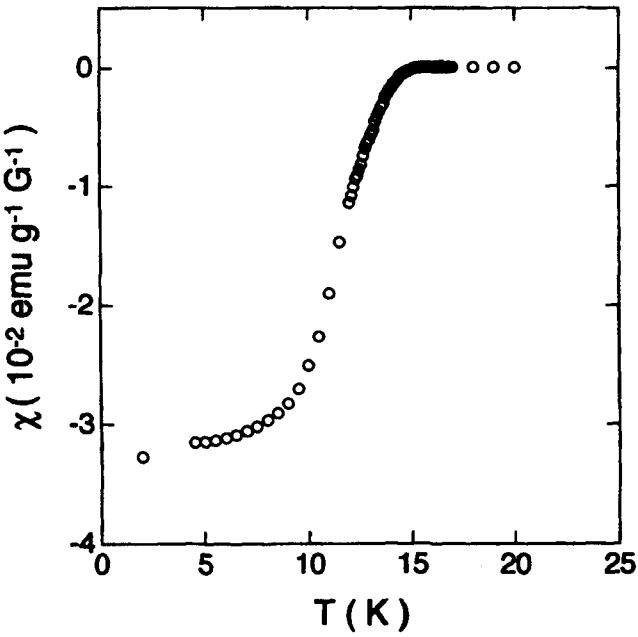

FIGURE 1 Temperature dependence of the magnetic susceptibility of $Na_xH_yC_{60}$

In order to obtain the structural information, we carried out powder X-ray diffraction (PXD) study for the sample with V_{SC}=65 %. The whole PXD profile corresponds to a single fcc (face-centered-cubic) phase with a lattice constant a=14.406(1) Å. Figure 2 shows the room-temperature crystal structure solved by Rietveld analysis. [8] The C_{60} molecules are orientationally disordered. The Na atoms in both tetrahedral (T) and octahedral (O) sites are displaced from the site center (off-centered). As shown in the three-dimensional view of Fig. 3, Na atoms in a T-site are positionally disordered at four corners of a tetrahedron with a side of 1.22 Å and those in an O-site at eight corners of a cube with a side of 1.58 Å. The refined occupancies of Na are 0.25 and 0.20 for the T- and O-sites, respectively, which gives one Na atom per T-site and 1.6 Na atoms per O-site, i.e., x=3.6 in $Na_xH_yC_{60}$.

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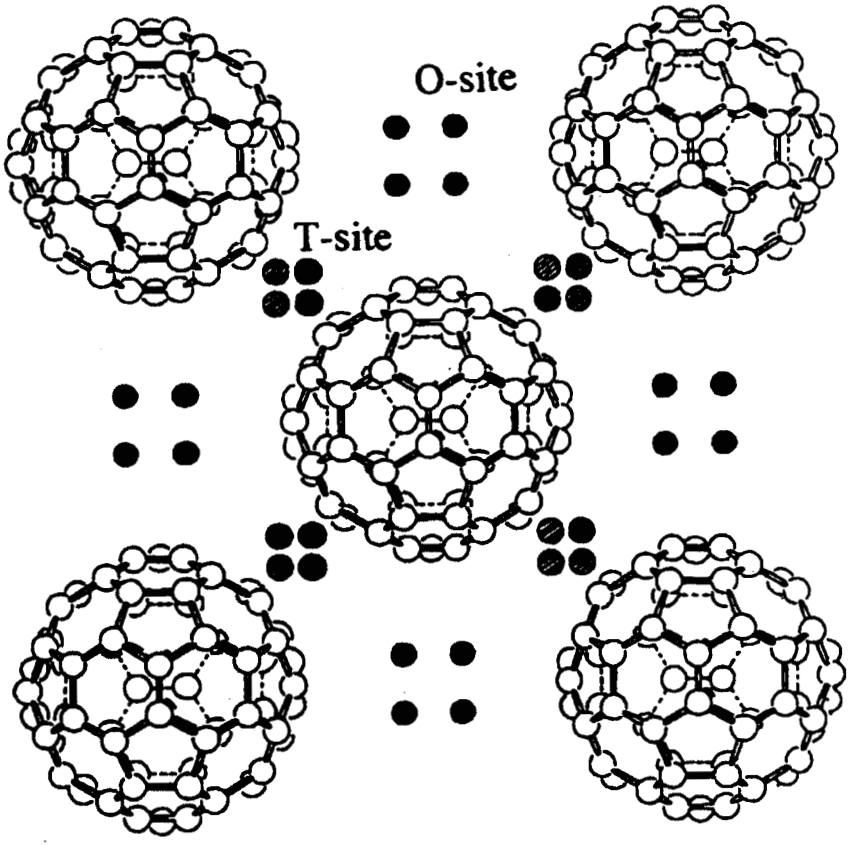

FIGURE 2 Crystal structure of $Na_xH_yC_{60}$. Open circles show C atoms and the orientationally disordered C_{60} molecules are drawn by solid and dashed lines. Full circles show Na atoms in the O-sites and slash-marked circles those in the T-sites

Although the position of H atom is unknown from X-ray diffraction, the presence of off-centered Na atoms suggests the intercalation of hydrogen in the interstitial sites. Hydrogen in the sample was experimentally detected by mass-analyzed thermal desorption. Figure 4 shows the thermal desorption spectra of H_2 molecules with m/e=2 at a constant heating rate of 5 K min^{-1} for the two samples with V_{SC}=35 % and 1 %. [5] The peak temperature T_p of the main desorption band is observed at 644 K and 673 K for the 35 % and 1 % samples, respectively. T_p shifts to a lower temperature with increasing the value of V_{SC} which is proportional to the amounts of intercalated hydrogen. This behavior indicates the kinetics of the second-order desorption process.[9] That is, hydro-

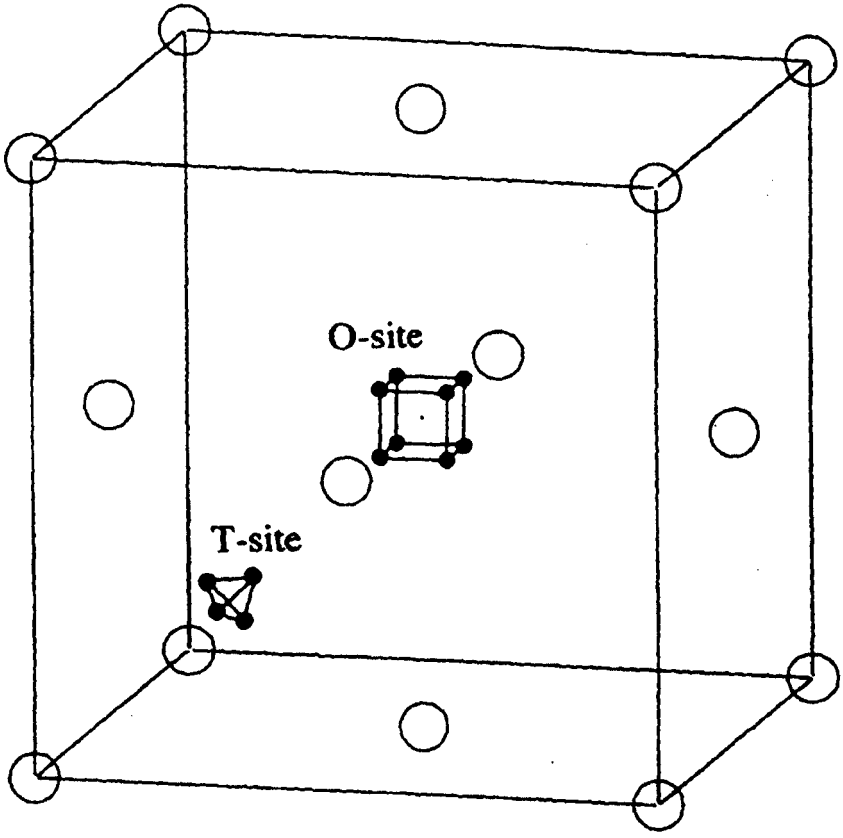

FIGURE 3 Schematic drawing of the crystal structure of $Na_xH_yC_{60}$. Open circles represent C_{60} molecules. The disordered Na atoms in one O-site and one T-site in a unit cell are depicted

gen in $Na_xH_yC_{60}$ exists as hydride ions (probably fractionally charged $H^{-\delta}$) and is desorbed upon heating from the surface after the formation of H_2 molecules through the association process between hydride ions.

Furthermore, mass-analyzed thermal desorption gives the quantitative amount of hydrogen by integrating the desorption spectrum. The H content per C_{60} is estimated to be H/C_{60}=1.0 for the V_{SC}=35 % sample. In connection to the result of Na content from Rietveld analysis, the final composition will be $Na_{3.6}HC_{60}$.

Next we calculated the electronic structure of $Na_xH_yC_{60}$. Two model structures are constructed. One is a model of Na_3HC_{60} which contains one Na atom per T-site, and one Na atom and one H atom per O-site. The other is a model of

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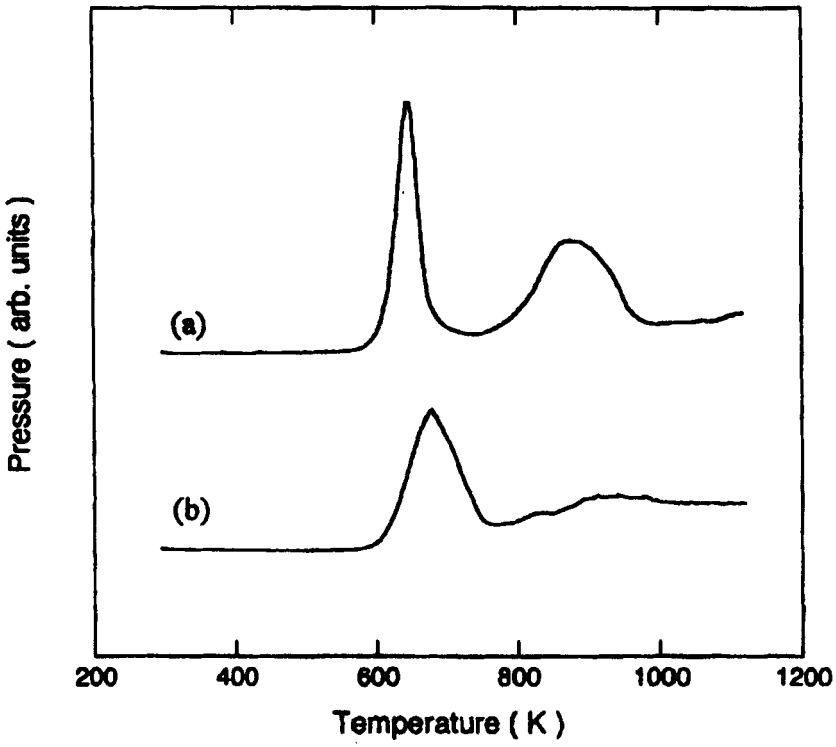

FIGURE 4 Thermal desorption spectra of H_2 for $Na_xH_yC_{60}$ with different V_{SC} (35 % (a) and 1 % (b))

Na_4HC_{60} which contains one Na atom per T-site, and two Na atoms and one H atom per O-site. We perform the first-principles calculations based on the density-functional theory within the local-density approximation. [10] Figures 5 and 6 show the electronic structures of Na_3HC_{60} and Na_4HC_{60}, respectively. In the band structures of Figs. 5(a) and 6(a), the upper three bands are derived from the lowest unoccupied molecular orbital (LUMO) of C_{60} molecules. This band picture is common in alkali-metal doped C_{60}superconductors. It is noteworthy that new bands derived from the 1s orbitals of H atoms appear below the LUMO-derived bands. The H-derived band in Na_3HC_{60} lies at ~0.1 eV deep from the Fermi level, while that in Na_4HC_{60} at ~0.4 eV. As shown in Figs. 5(c)-5(e) for Na_3HC_{60}, there is a large mixing among 2p orbital of C, 1s orbital of H and 3s orbital of Na at the Fermi level. The charge of H atoms is calculated to be δ =0.2 in $H^{-\delta}$. On the other hand, for Na_4HC_{60}, there is negligible mixing among the orbitals of C, H and Na. Also in Na_4HC_{60}, H atoms have a fractional charge of $H^{-0.3}$.

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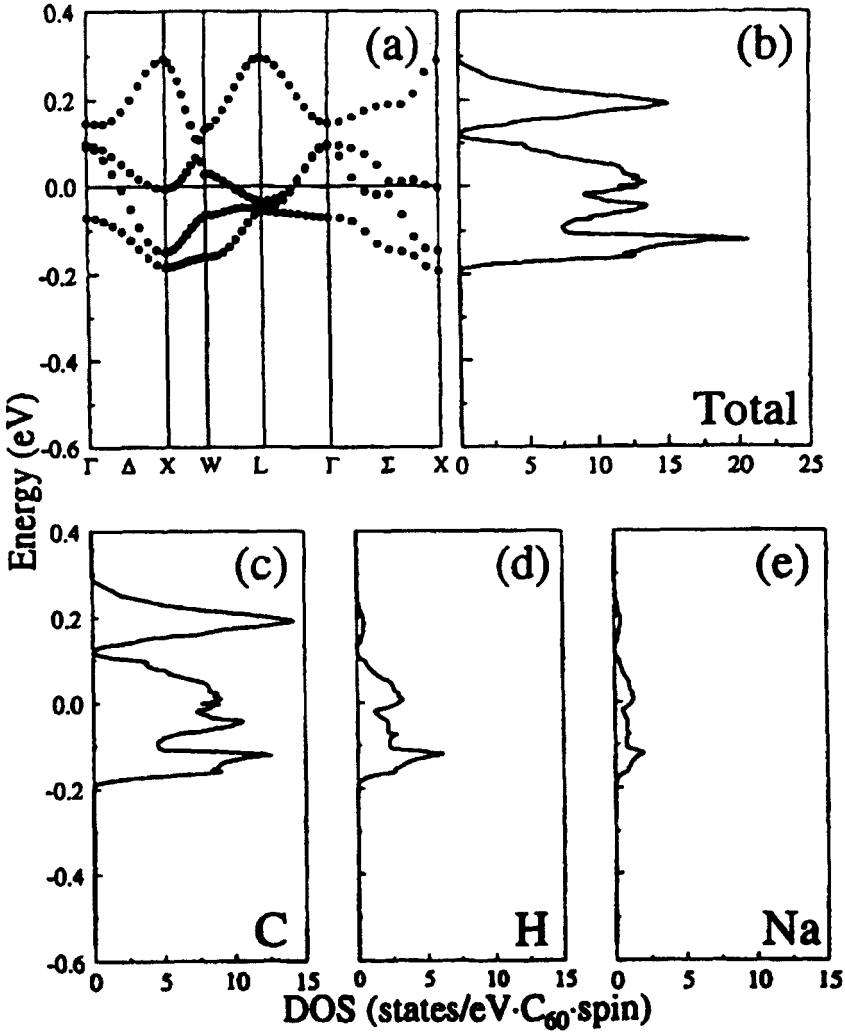

FIGURE 5 Electronic structure of Na_3HC_{60}: band structure (a), total density of states (b) and partial densities of states of C (c), H (d) and Na (e). The zero energy indicates the Fermi level

The electrons in the H-derived band in Na_3HC_{60} contribute to conduction, while the contribution of the electrons in $Na_4HC_{60}$is negligible. However, $Na_xH_yC_{60}$ has a composition of $Na_{3.6}HC_{60}$ from the experiments. There is a possibility that $Na_xH_yC_{60}$ crystallizes in the solid solution of Na_3HC_{60} and

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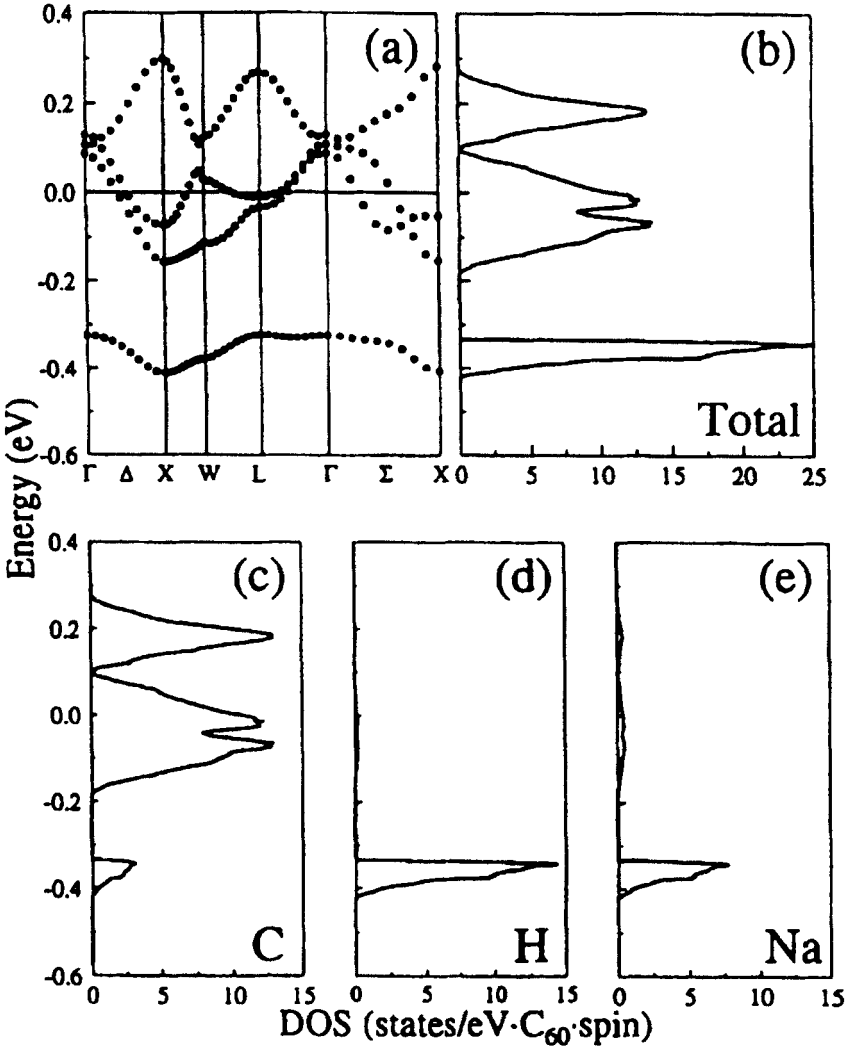

FIGURE 6 Electronic structure of Na_4HC_{60}: band structure (a), total density of states (b) and partial densities of states of C (c), H (d) and Na (e). The zero energy indicates the Fermi level

Na_4HC_{60}, and possesses the intermediate electronic structure between $Na_3HC_{60}$and Na_4HC_{60}.

In conclusion, hydrogen in $Na_xH_yC_{60}$plays a very important role as a spacer on the stabilization of the SC phase down to low temperature. Besides, hydrogen exists as not a neutral spacer but a fractionally charged spacer and correlates with

the electronic state. In this sense, $Na_xH_yC_{60}$ is a new type of C_{60} based superconductor.

BONDING STATES OF RARE GASES IN SOLID C_{60}

A characteristic interaction was found to exist between C_{60} and rare gas elements under the conditions of ambient temperature and pressure by means of mass-analyzed thermal desorption and x-ray photoelectron spectroscopy (XPS).

C_{60} (Hoechst, 99.98% purity) transferred *in situ* to an ultra-high vacuum system and heated at 623 K was exposed to rare gases (Nippon Sanso, >99.9999% purity) of 1 to 1.4 atm, at room temperature to 473 K and for 1 to 10 days. After the sample was cooled to liquid nitrogen temperature, the sample tube was evacuated to ultra-high vacuum. The thermal desorption spectra were measured by using two mass-spectrometers with the temperature-rise rate of 5 K/min. [5][7][11] A Pyrex glass tube in which a sample of C_{60} exposed to rare gas had been sealed was broken in a glove bag attached to a specimen-introduction device of x-ray photoelectron spectroscopy machine, VG ESCA LAB Mk II, and the sample was pressed onto an electrically-conductive adhesive tape on a sample stage. An MgK_α line was used as probe for x-ray photoelectron spectrum measurements.

Figure 7 shows the thermal desorption spectra for C_{60} exposed to He, Ne and Ar. Desorption peaks appeared in the low temperature range of 70–300 K as well as in the high temperature range of 450–900 K. This is a first report on the observation of thermal desorption spectra of He, Ne and Ar in the temperature region higher than room temperature. The content of rare gas in the sample was determined from the amount of the rare gas integrated over the entire desorption spectrum, the relative sensitivity of the mass analyzer and the pumping speed. Exposure to rare gases at 393 K and 1.3 atm for 3 days gives the compounds; $C_{60}He_{0.39}$, $C_{60}Ne_{0.074}$ and $C_{60}Ar_{0.09}$. The contents of the rare gases are not in impurity level but in stoichiometric level. The content of He in the compound is more than those of Ne and Ar in their compounds, which is attributed to the size effect of rare gas on its migration into the lattice of C_{60}. The desorption peak of He was observed at 509 K for $C_{60}He_{0.39}$, that of Ne at 706 K for $C_{60}Ne_{0.074}$, and that of Ar at 511 K for $C_{60}Ar_{0.09}$.

Figure 8 shows the x-ray photoelectron spectra in the C1s and Ar regions. The C1s peaks for C_{60} and C_{60} exposed to He, Ne and Ar were observed at 282–285 eV, and the shake-up satellites based on a $\pi - \pi^*$ transition appeared at *ca.* 5 eV apart on their higher binding-energy side. A weak peak at 275 eV on the lower binding energy side is ascribed to an x-ray impurity of an MgKa probe. Exposure to Ar gives a new peak which was obviously observed at 269 eV and is assigned

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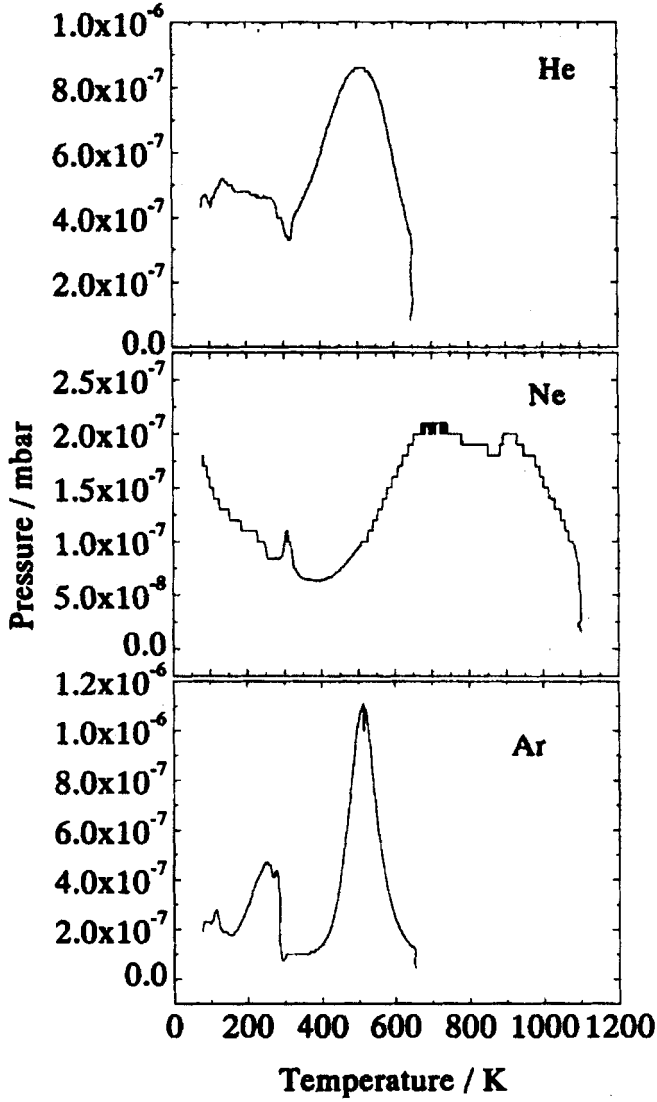

FIGURE 7 Thermal desorption spectra for C_{60} exposed to He, Ne and Ar

to Ar2p. An Ar2p peak of Ar implanted into graphite has been reported to appear at 241.3 eV or 241.6 eV, and those of argon implanted into metals at 240.2–241.9 eV. [12] The Ar2p peak for $C_{60}Ar_x$ is located at *ca.* 26 eV higher than those for

graphite-Ar and metal-Ar systems. The energies for the peaks could not be determined accurately, because the peaks shifted depending on the charging-up of a sample during the measurement. The energy difference between the C1s and the Ar2p peaks is, however, expected to be definite, because they were observed under the same experimental conditions of a sample. Ar is considered to be occluded as a neutral atom in graphite-Ar and metal-Ar systems, and the energy difference between the C1s and the Ar2p peaks for the graphite-Ar system has been reported to be 43 eV, and the Ar2p peaks for the metal-Ar systems to show two-peak profiles due to a spin-orbit interaction. [14] The energy difference for $C_{60}Ar_x$ is 16 eV, and the Ar2p peak shows almost a single-peak profile. The changes of chemical shift and peak profile depend on the chemical bonding state. Since the C1s peaks were observed at 282–285 eV (3 eV difference), the remarkably reduced energy difference and the change of a peak-profile for $C_{60}Ar_x$ indicate that argon atoms in a lattice of C_{60} are in the bonding state.

Figure 9 shows XPS spectra in the valence band region. Exposure to Ar also gives a new peak which appeared at around 30 eV and is assigned to Ar3s. An Ar3s peak has been reported to appear at 22 eV for the argon implanted into metals, [12] which is located at *ca.* 8eV lower than that for $C_{60}Ar_x$. This energy difference as *ca.* 8 eV is larger than the energy differences of *ca.* 3 eV among carbon peak position for C_{60}, C_{60}-He, C_{60}-Ne and C_{60}-Ar systems. Carbon peak profiles for C_{60}-He and C_{60}-Ne systems are also different from that for pristine C_{60}. The XPS spectra of He and Ne have not been reported because of negligibly small photoelectron of He and Ne by x-ray. Therefore, the peak assignment is difficult at the present. The changes in peak profiles for carbon indicate the chemical bonding at least. The energy difference and change in peak profile also suggest that argon atoms in a lattice of C_{60} are in the bonding state. Since the energy difference of carbon valence bands were also observed to be ca. 3 eV as described in the C1s spectra, the observed energy difference of Ar3s as ca. 8 eV is larger than that in the carbon valence band. This indicates that Ar has the positive charge.

The mass-analyzed thermal desorption spectra above room temperature region and the XPS spectra show single-peak profiles. If the interaction of rare gases in a lattice of C_{60} occurs through a simple adsorption, the amount of desorption gas has to be in the order as Ar > Ne > He because their van der Waals forces decrease along this sequence. The experimental results, however, show the reverse order, so that it is reasonable to conclude that the interaction of He, Ne and Ar is caused not by adsorption but by bonding in the lattice. Two experimental results support the bonding state. (1) A hydrogen desorption peak for the KC_8H_x (x~0.6) ternary system appears at 512 K, [11] and desorption peaks for Na-H-C_{60} at around 650 and 900 K, [5][7] in which hydrogen has been concluded

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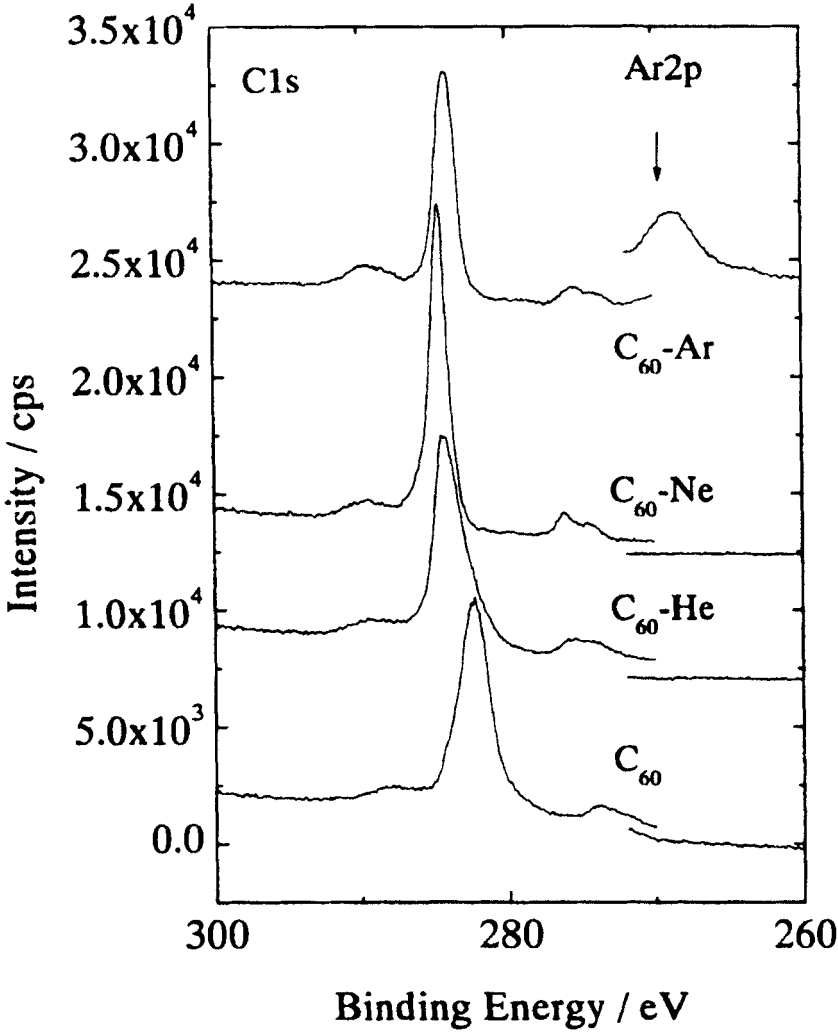

FIGURE 8 XPS spectra in the C1s region for C_{60} and C_{60} exposed to He, Ne and Ar

to exist as hydride or $H^{-\delta}$. These desorption temperatures are comparable to those of C_{60}-He, Ne and Ar systems. (2) A large isotopic effect is found for the C_{60}-He system: ^{4}He is desorbed at 509 K, while ^{3}He at around 420 K.

The impurities such as another chemical species and defects are difficult to think more, to have the content that helium reaches 40% to C_{60}. Also, the significant impurities are not found out even XPS and ESR measurements. The meas-

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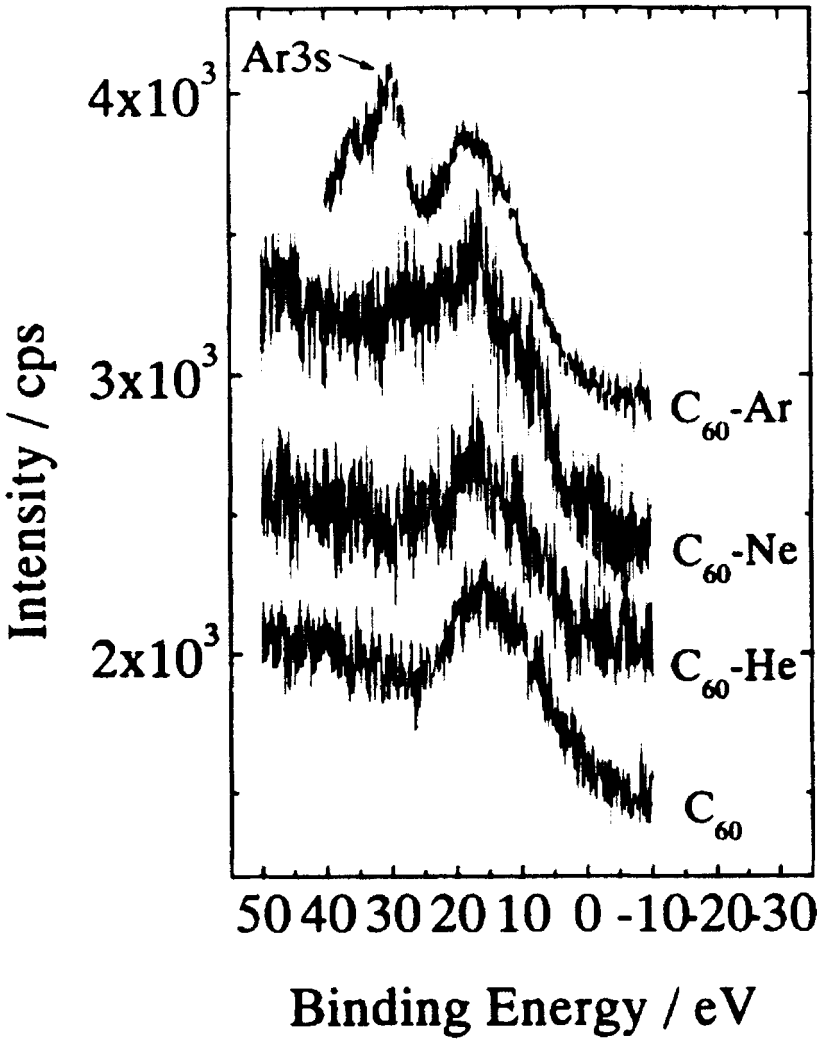

FIGURE 9 XPS spectra in the valence band region for C_{60} and C_{60} exposed to He, Ne and Ar

urements of compressibility for C_{60} using He, Ne and Ar as pressure media and the study of diffusion kinetics in solid C_{60} have been carried out under the conditions of high pressures at around several kbar. [13]-[16] In those study, van der Waals interaction is mainly discussed. However, the results obtained from the

mass-analyzed thermal desorption and the XPS spectra lead to the conclusion that the He, Ne and Ar atoms in a lattice of C_{60} are in the bonding state. [17]

DISCUSSION: HIDDEN SURFACE SCIENCE

More than 220 million chemical substances have been known and another million species are newly obtained every year by means of synthesis and extraction. Although these substances possess various structures and properties, they can be classified on the basis of their chemical bonds, i.e., three kinds of chemical bonds built by electromagnetic force – covalent, metallic, ionic – and a van der Waals bond.

Charge transfer complexes, from which most organic conductors and superconductors are derived, are formed through mixing of ionic and van der Waals bondings. Hydrogen bonds contributing to composition of various biosubstances are also regarded to be produced from a kind of charge transfer interaction.

Adsorption phenomena on solid surfaces in the field of chemistry and physics – chemisorption and physisorption – have been studied extensively, enriching "Surface Science". The force operating in these adsorptions is also generated from the hybridization of four kinds of bonds mentioned above.

The two studies described in the previous sections – the three-component molecular superconductors and the compounds formed between C_{60} and rare gases – provided certain experimental verification of the idea that a unique force derived from the hybridization of the four kinds of bonds is operating in intermolecular spaces of C_{60}. Hydrogen accommodated in a C_{60} molecular lattice for a superconductor of Na_4HC_{60} shows characteristics of $H^{-\delta}(\delta \sim 0.2)$. The hydride ions are expected to form a conduction band in the solid of Na_3HC_{60}. On the other hand, it could be inferred that some bonding state which is still out of our knowledge exists in $C_{60}X$ (X = He, Ar, Ne). This may suggest that a unique bonding field is generated among C_{60} molecules – not on the surface, but in the intermolecular spaces. We would like to name the science treating an energy field in the intermolecular spaces inside material where novel substances could be produced, as "Hidden Surface Science", and hope we will be able to develop the new science.

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