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RESEARCH OF ELECTROMAGNETIC SHIELDING PROPERTIES OF SINGLE-WALLED CARBON NANOTUBES THIN TRANSPARENT FILMS

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The paper presents the results of studying the shielding properties of thin transparent films in single-walled carbon nanotubes on flexible substrates of polyethylene terephthalate. The films were formed by spraying colloidal solution on single-walled carbon nanotubes. The film thickness was determined by the volume of the sprayed colloidal solution and was measured using transmission electron microscopy in a cross-section mode. The morphology and structural quality of the films were studied by electron microscopy, optical spectroscopy, and Raman spectroscopy. The results showed the high structural quality of the material. According to Raman spectroscopy, the ratio of peaks intensities G / D is 23.4, which is the evidence of a significant predominance of carbon in the sp^2 hybridization. It is typical for graphite-like systems and, in particular, carbon nanotubes. The spectral dependences of the transmission and reflection coefficients of radio waves in the K range of 18–26.5 GHz were studied. Absorption of radiation is the dominant shielding mechanism. Increasing the film thickness from 15.9 to 56.1 nm is accompanied by decreasing the surface resistance from 971 to 226 Ohm / sq, while optical transparency decreases from 93.58 to 76.71 %. Shielding efficiency increases from 2.29 to 6.6 dB, increasing the proportion of absorbed radiation from 34.6 to 51.2 % at a frequency of 18 GHz. This indicates the prospects for the use of films as electromagnetic shielding and anti-icing coatings in the aerospace industry.

Keywords: single-walled carbon nanotubes, thin films, electromagnetic shielding.

ИССЛЕДОВАНИЕ РАДИОЭКРАНИРУЮЩИХ СВОЙСТВ ТОНКИХ ПРОЗРАЧНЫХ ПЛЕНОК ОДНОСТЕННЫХ УГЛЕРОДНЫХ НАНОТРУБОК

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Представлены результаты исследования экранирующих свойств тонких прозрачных пленок одностенных углеродных нанотрубок на гибких подложках из полиэтилентерефталата. Пленки формировались спрей распылением коллоидного раствора одностенных углеродных нанотрубок. Толщина пленок задавалась объемом распыляемого коллоидного раствора и измерялась при помощи просвечивающей электронной микроскопии в режиме cross-section. Морфология и структурное качество пленок были изучены методами электронной микроскопии, методами оптической спектроскопии и спектроскопии комбинационного рассеяния света. Результаты позволяют судить о высоком структурном качестве материала. Согласно данным спектроскопии комбинационного рассеяния, соотношение интенсивностей пиков G/D составляет 23,4, что является свидетельством существенного преобладания углерода, находящегося в состоянии sp^2 гибридизации. Это характер-

но для графитоподобных систем и, в частности, углеродных нанотрубок. Изучены спектральные зависимости коэффициента прохождения и отражения радиоволн в К-диапазоне 18–26,5 ГГц. Доминирующим механизмом экранирования является поглощение излучения. Увеличение толщины пленки с 15,9 до 56,1 нм сопровождается снижением поверхностного сопротивления с 971 до 226 Ом/кв, оптическая прозрачность при этом снижается с 93,58 до 76,71 %. Эффективность экранирования увеличивается с 2,29 до 6,6 дБ, повышая долю поглощенного излучения с 34,6 до 51,2 % на частоте 18 ГГц. Это говорит о перспективах применения пленок в качестве радиоэкранирующих и антиобледенительных покрытий в аэрокосмической отрасли.

Ключевые слова: одностенные углеродные нанотрубки, тонкие пленки, электромагнитное экранирование.

Introduction. Single-walled carbon nanotubes (SWCNTs) are promising for the aerospace industry due to the unique combination of high mechanical strength (Young's modulus ~ 1TPa), high electrical conductivity, as well as low density and, as a consequence, low weight of the finished product. The listed properties imply broad prospects for the use of SWCNTs as a reinforcing material for polymer composites, light and strong electrical cables, and functional layers for transparent electric-heated and electromagnetic shielding structural elements. The function of electromagnetic shielding is of interest, in particular, for solving the problem of protecting information. As far back as the 80s of the 20th century the fundamental possibility of intercepting and decrypting information contained in radiation from a computer monitor was shown. This fact increases the priority of applying various measures to protect a wide variety of electronic objects from unauthorized removal of the information contained in them, or possible external influences. Shielding output devices is the main solution to protecting information. The reality of such a scenario has been experimentally proven. In Russia this channel of information leakage is called SEMRP (Side Electromagnetic Radiation and Pickup). In the USA the standard is called TEMPEST (Transient Electromagnetic Pulse Emanation Standard).

Traditionally thin layers of transparent conducting oxides such as In_2O_3: Sn [1] and ZnO: Al [2] act as shielding films.

Carbon nanomaterials, such as carbon nanotubes (single-walled [3] and multi-walled), graphene [4; 5], reduced graphene oxide [6], as well as polymers with conjugated bonds [7] are promising in solving this problem. As a result active study of their shielding properties is currently underway.

In [3] the effectiveness of shielding electromagnetic radiation of the HF and microwave range by SWCNT films with a thickness of 10 nm to 10 μm was studied. It was shown that the shielding efficiency of a SWCNT film with a thickness of 100 nm is about 40 dB at a frequency of 10 GHz, however, the optical transparency of such a film is about 50%, which is significantly lower than operational requirements (> 80 %). In the case of a graphene monolayer the shielding efficiency is 2.27 dB with a surface resistance of 635 Ohm / sq and optical transparency of 97.8 % [4]. The graphene monolayer has fairly low shielding efficiency, but due to its high transparency it can be a part of a composite coating, for example in combination with metal grids or nanowires.

The method of forming films of single-walled carbon nanotubes. The paper studies the shielding ability of thin SWCNT films (OCSiAl, Novosibirsk) on polyethylene terephthalate substrates. The application of thin SWCNT films was carried out by the spray method; a detailed description is given in [8; 9]. The principle is as follows: the compressed air from the compressor is supplied to the nozzle (airbrush) under pressure of 6 atm (0.6 MPa), spraying the SWCNT colloidal dispersion (the preparation procedure is described in detail in [9]) onto heated polyethylene terephthalate substrates (manufactured by Hi- Fi, Japan, thickness 50 μm) – as the main substrate and a base alkaline glass, 1 mm thick for spectroscopic measurements. The substrate temperature is controlled by the heating element and in this work was 120 °C, this is enough for the microdrops of the colloidal solution to evaporate without being able to coalesce on the substrate.

The thickness of the SWCNT films was determined by the volume of the sprayed SWCNTs dispersion; four volumes of SWCNT ink were used in the work: 1, 2, 3, and 4 ml. After spraying the colloidal solution the films were washed with distilled water for one hour, and then dried at 100 °C for one hour in order to remove residual water and form a coherent film of nanotubes.

Studying the morphology and thickness of SWCNT films by scanning and transmission electron microscopy. The morphology and thickness of SWCNT films was studied by electron microscopy. The study of morphology was carried out using a Hitachi S5500 scanning electron microscope (Center of collective uses FRC KSC SB RAS). Fig. 1, *a*, *b*, c show films obtained by spraying 1 and 3 ml of SWCNT dispersion, respectively. It is seen that with an increase in the volume of sprayed dispersion the density of the SWCNT film increases.

The thickness of SWCNT films was measured by transmission electron microscopy (TEM) using a Hitachi HT7700 transmission electron microscope (Center of collective uses FRC KSC SB RAS) in a cross-section mode according to the technique described in [10].

Measuring the SWCNT films average thickness in the cross-section mode made it possible to determine the average film thickness for all volumes of sprayed dispersion. Film thickness for 1; 2; 3 and 4 ml of the dispersion are 15.3; 32.9; 44.9 and 56.1 nm, respectively. According to the straight line tangent, the direct dependence of the film thickness on the sprayed colloid volume can be described by the equation $h = 14{,}56 \cdot V$, which allows accurate characterizing the parameters of SWCNT films for the selected technological mode.

Investigation of the structural and optoelectronic characteristics of thin SWCNT films. For SWCNT films all spectroscopic studies were carried out on alkaline glass, which excluded the contribution of the substrate to the spectra and thereby made the analysis more objective. The optical transparency of SWCNT films was studied by optical spectrophotometry using a Shimadzu UV-3600 spectrophotometer in the range 400–2500 nm.

Fig. 2, *a* shows the spectral transmission for an alkaline glass substrate and a 44.5 nm thick SWCNT film. The graph shows three absorption peaks characteristic of SWCNTs, which correspond to electronic transitions between the Van Hove features for semiconductor (S_{11} ~ 1836 nm and S_{22} ~ 1064 nm) and metal (M_{11} ~ 786 nm) SWCNTs [11]; the position of these peaks depends on the SWCNTs diameter, with decrease in diameter all three peaks experience a blue shift.

Fig. 2, *b* shows the dependences of the specific surface resistance and optical transmission at a wavelength of 550 nm on the thickness of the SWCNT film. The sheet resistance of the films is 971; 607; 379 and 226 Ohm / sq, respectively, and the optical transmission minus the reflection from the glass substrate is 93.6; 88.5; 81.9 and 76.7 %, respectively.

The structural quality of SWCNTs can be most fully characterized using Raman spectroscopy. Atomic vibrations in SWCNTs were studied using Raman spectroscopy.

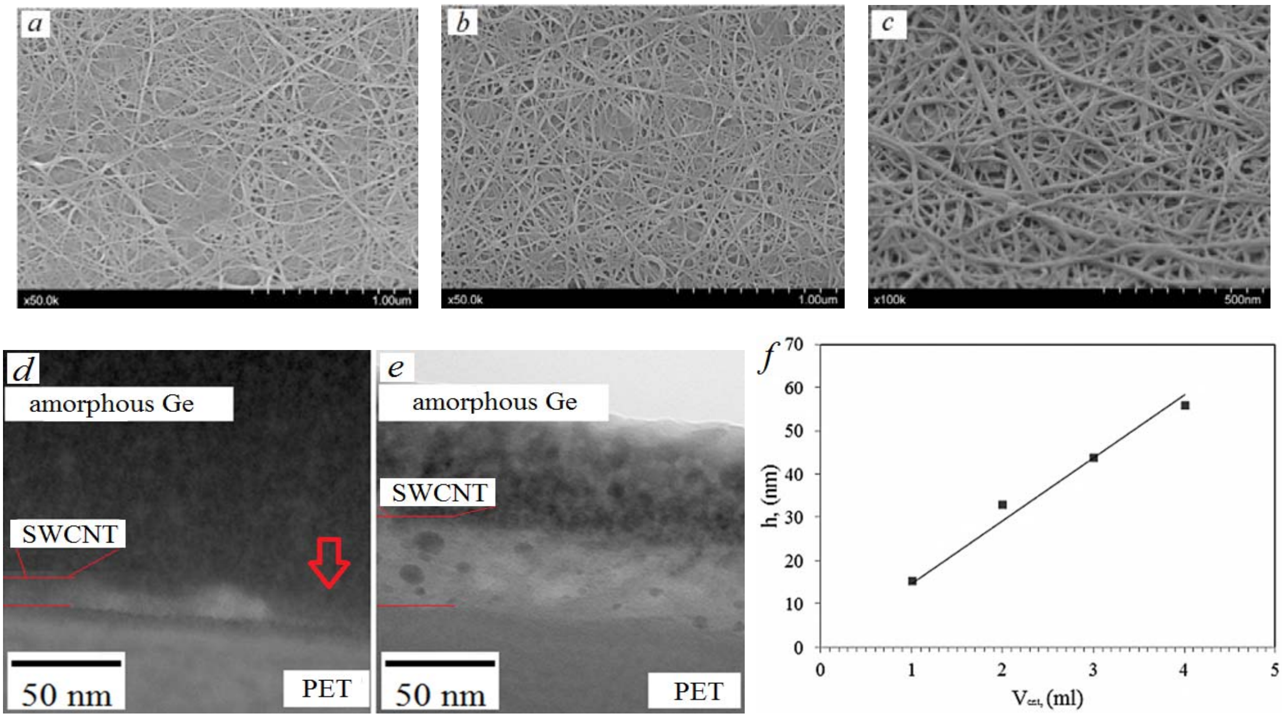

Fig. 1. SEM images of SWCNT films of various thicknesses: 1 ml (*a*) and 3 ml (*b* and *c*) of dispersion; TEM image of SWCNT films of various thicknesses: 1 ml (*d*) and 3 ml (*e*) of dispersion. Dependence of the SWCNT films thickness on the volume of sprayed dispersion (*f*)

Рис. 1. СЭМ изображения пленок ОУНТ различной толщины: 1 мл (*a*) и 3 мл (*b* и *c*) дисперсии; ПЭМ изображение пленок ОУНТ различной толщины: 1 мл (*d*) и 3 мл (*e*) дисперсии. Зависимость толщины пленок ОУНТ от объема распыленной дисперсии (*f*)

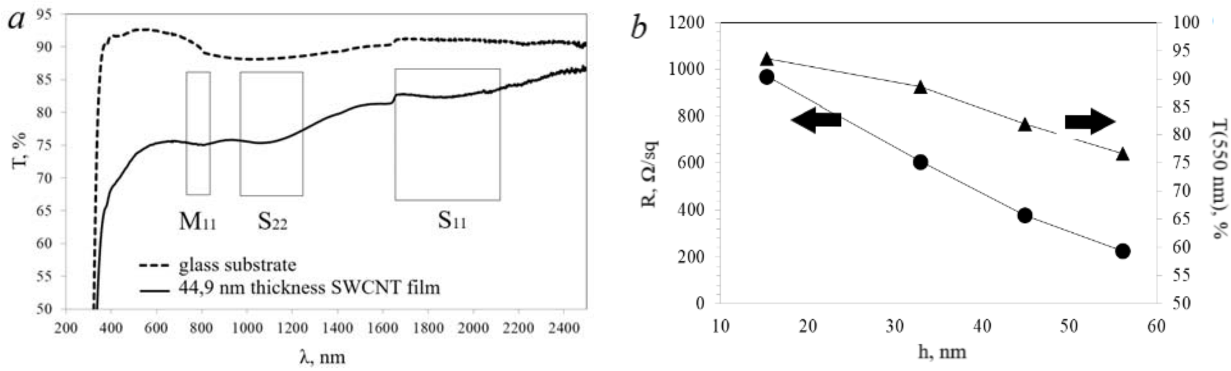

Fig. 2. Transmission of a SWCNT film 44.9 nm thick in the range 400–2500 nm (*a*); dependence of surface resistance and transmission of SWCNT films as a function of film thickness (*b*)

Рис. 2. Пропускание пленки ОУНТ толщиной 44,9 нм в диапазоне 400–2500 нм (*a*); зависимость поверхностного сопротивления и пропускания пленок ОУНТ в зависимости от толщины пленки (*b*)

Fig. 3 shows the Raman spectra for the films with thickness of 15.3 nm and 56.1 nm, obtained using a Horiba Jobin Yvon T64000 Raman spectrometer (Center of collective uses FRC KSC SB RAS).

Three peaks characteristic of SWCNTs can be distinguished in the spectra: the G-line characterizing the vibrations of the system of carbon sp^2 bonds (~ 1592.8 cm^{-1}) (graphite-like zone), the 2D line (~ 2678.7 cm^{-1}), indicating two-dimensionality of the material, which is the overtone of the D-line (defective zone) (~ 1332.5 cm^{-1}). The RBM peak (the breathing mode is a split peak at ~ 150.6 cm^{-1} and ~ 180.29 cm^{-1}) characterizes expansion-compression vibrations relative to the central axis of nanotubes [12]. It is also worth noting that the film thickness does not affect the position of the peaks. The low intensity of the D mode indicates low defectiveness of the nanotube material; the ratio of the G / D peaks intensities is 23.4.

In [9] we calculated the diameter of SWCNTs of this type; it amounted to 1.6–1.8 nm, which correlates with the results of [13].

Investigation of the electromagnetic shielding properties of SWCNT films. The reflection (S_{11}) and transmission (S_{21}) coefficients in the K range (18–26.5 GHz) were measured according to the scheme shown in fig. 4. Samples of SWCNT films were clamped using threaded connections between two waveguide-coaxial transitions of rectangular cross section with dimensions 5.5 × 11 mm. The signal was excited and analyzed using the R&S ZVA 50 vector network analyzer. Changing the level of the transmitted and reflected signal allows drawing conclusions about the magnitude of the shielding effect and its mechanism. It is worth noting that the approach involving the use of a waveguide, unlike the method of measurement in free space (using horns), allows estimating the level of reflection of the microwave signal from the sample.

Fig. 5 shows the spectral dependences of the parameters S_{11} and S_{21} measured in dB which are reflection and transmission coefficients, respectively.

Thus, knowing the values of the coefficients S_{21} and S_{11} it is possible to calculate the transmission and reflection coefficients according to the following equations [14]:

$$T = \frac{P_t}{P_i} = 10^{(0.1 S_{21})}, \quad (1)$$

$$R = \frac{P_r}{P_i} = 10^{(0.1 S_{11})}.$$

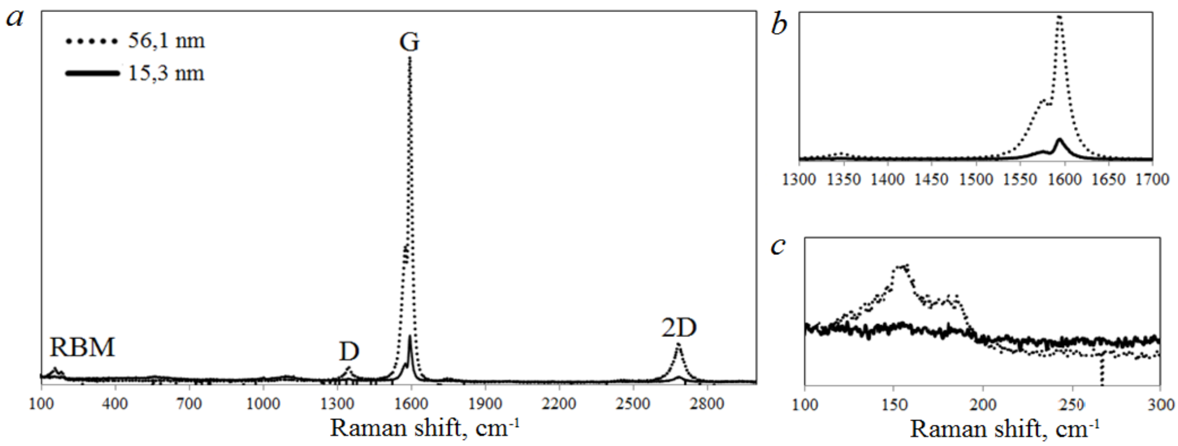

Fig. 3. Raman spectra of SWCNT films with thickness of 15 and 50 nm (*a*), enlarged fragments of D and G peaks (*b*) and RBM (radial breathing mode) (*c*)

Рис. 3. Спектры комбинационного рассеяния пленок ОУНТ толщиной 15 и 50 нм (*a*), увеличенные фрагменты D и G пиков (*b*) и дыхательной RBM моды (*c*)

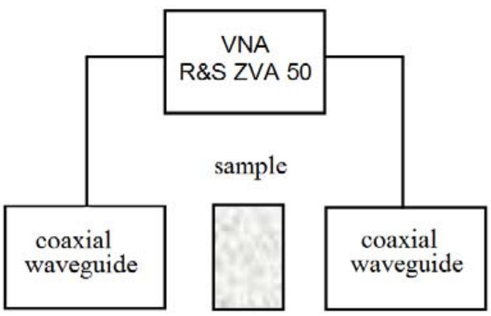

Fig. 4. Schematic representation of a measuring unit

Рис. 4. Схематическое изображение измерительной установки

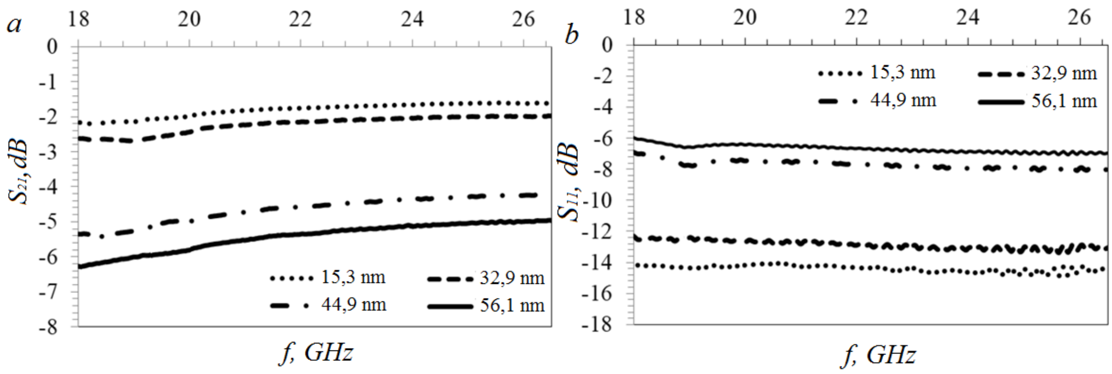

Fig. 5. Spectral dependences of parameters S_{21} (*a*) and S_{11} (*b*) for SWCNT films of various thicknesses

Рис. 5. Спектральные зависимости параметров S_{21} (*a*) и S_{11} (*b*) для пленок ОУНТ различной толщины

On the basis on the fact that the sum of all radiation components is 1, it is possible to calculate the fraction of absorbed radiation according to the following equation: $A = 1 - T - R$.

The dependence of transmission, reflection and absorption of SWCNT films of various thicknesses at the frequency of 18 GHz:

SWCNT film thickness, nm	T (18 GHz), %	R (18 GHz), %	A (18 GHz), %
15.3	60.8	4.1	34.6
32.9	54.8	5.8	39.4
44.9	29.2	20.4	50.4
56.1	23.6	25.2	51.2

The main model describing the relationship between the specific sheet resistances of thin films of non-magnetic materials is the continuous layer model [14]

$$SE(dB) = 20\lg\left(1 + \frac{Z_0}{2R_s}\right). \qquad (2)$$

Fig. 6 shows comparison of the experimentally obtained values of shielding efficiency at a frequency of 18 GHz from the sheet resistance of the coatings, calculated by the equation $SE(dB) = -10\lg T$ of the surface resistance of SWCNT films at the boundaries of the studied range, and approximation of the experimental points by equation 2.

It can be seen from the graph that the results obtained are in agreement with the model, as a result of which it can be assumed that the shielding efficiency can be increased by reducing the surface resistance of SWCNT films. Single-walled carbon nanotubes can be doped and acquire either a hole [15] (doping with electron acceptors such as: HNO_3, $FeCl_3$, $HAuCl_4$) or electron [16] conductivity (doping with electron donors: amines, phosphines, etc.). Initially SWCNT films have hole conductivity, as evidenced by the sign of the Seebeck coefficient (+40 mV at room temperature) [9]. As it can be seen from equation 2, lowering the sheet resistance due to doping to the value of 50 Ohm / sq helps to increase the shielding efficiency to the value of 13.5 dB or transmission of not more than 4.5 %, so the remaining 95.5 % of the power will be absorbed in the film and partially reflected back. This fact indicates the promise of using transparent SWCNT films in solving the problem of shielding microwave electromagnetic radiation.

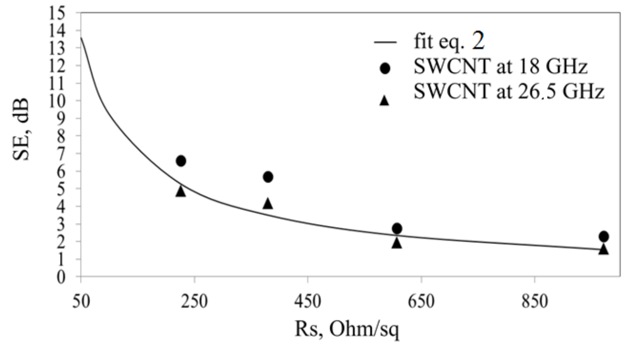

Fig. 6. Shielding efficiency as a function of sheet resistance for SWCNT films at the boundaries of the studied range

Рис. 6. Зависимость эффективности экранирования от величины поверхностного сопротивления для пленок ОУНТ на границах исследуемого диапазона

Conclusion: The electromagnetic shielding properties of thin SWCNT films on flexible PET (polyethylene terephthalate) substrates were studied. The contributions of reflection and absorption are determined. The absorption of radiation is the predominant factor in attenuation of radio emission in the studied K range (18–26.5 GHz). Increasing the film thickness from 15.9 nm to 56.1 nm increases the fraction of absorbed radiation from 34.6 to 51.2 % at the frequency of 18 GHz.

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