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A rhenium review – from discovery to novel applications

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ABSTRACT

Purpose: The article characterises rhenium in terms of its physiochemical properties, most popular methods of manufacturing and key applications. The examples of rhenium at a nanometric scales are also presented, taking into account the latest literature reports in this field. The objective of the article is also to present advanced nanocomposite materials consisting of nanostructured rhenium permanently attached to selected carbon nanomaterials - Single Walled Carbon NanoTubes (SWCNTs), Double Walled Carbon NanoTubes (DWCNTs), Multi Walled Carbon NanoTubes (MWCNTs) and Single Walled Carbon Nanohorns (SWCNHs).

Design/methodology/approach: The article delineates various manufacturing methods at a mass and nanometric scale. It also describes a custom fabrication method of carbon-rhenium nanocomposites and the results of investigations performed in a transmission electron microscope (TEM) for nanocomposites of the following type: MWCNTs-Re, SWCNTs/DWCNTs-Re, SWCNTs-Re and SWCNHs-Re.

Findings: Rhenium has been gaining growing importance in industry for years, and its applications are very diverse, including: heat resistant alloys, anti-corrosive alloys, rhenium and rhenium alloy coatings, elements of electrical equipment, radiotherapy, chemistry and analytical technology and catalysis. Carbon-metallic nanocomposites are currently enjoying strong attention of research institutions.

Research limitations/implications: The development and optimisation of fabrication processes of materials containing carbon nanotubes or carbon nanotubes coated with metal nanoparticles, especially rhenium, is a weighty aspect of advanced materials engineering.

Practical implications: Newly created nanocomposite materials, developed as a response to the market demand, are interesting, state-of-the-art materials dedicated to various applications, especially as gas or fluid sensors, and as materials possessing catalytic properties.

Originality/value: The article describes nanocomposites of the following types: MWCNTs-Re, SWCNTs/DWCNTs-Re, SWCNTs-Re, SWCNHs-Re, created as a result of high-temperature reduction of a precursor of rhenium ($HReO_4$ or NH_4ReO_4) to metallic rhenium. This metal is deposited on carbon nanomaterials as nanoparticles, or inside of them as nanoparticles or nanowires whose size and dispersion are dependent upon the conditions of a technological process.

Keywords: Rhenium; Re nanoparticles; Re nanowires; Carbon-metallic nanomaterials

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Rhenium was discovered in 1925 and – due to its unique physiochemical properties – is regarded as an exceptionally precious metal. Although the content of rhenium in the earth crust is very small and is estimated to be approx. 10^{-7} %, its industrial applications are in many cases irreplaceable [1-5]. It is thought that rhenium does not create own minerals, hence it does not occur independently in nature. This chemical element is most often accompanied by high-temperature sulphide ores, especially copper-molybdenite ores, and also exists in small amounts in minerals of lead, zinc, platinum or niobium. It is also a component of columbite, silicides, selenide and other minerals (maximum content of $21 \cdot 10^{-4}$ % Re). According to the literature data, the highest content of rhenium was detected in molybdenite in northern Sweden, and the content in this case was 0.05-0.25% [1,6-10].

2. Rhenium fabrication methods

Rhenium fabrication methods can be divided into several groups, whose characteristics are presented in Table 1. Rhenium production in Poland is a great achievement, especially that Poland is the only producer of this rare element from its own sources. After several years of extensive research and development works undertaken by scientists from the Institute of Non Ferrous Metals in Gliwice and industrial specialists, mass production of rhenium was launched in Poland in 2007. The KGHM Polska Miedź S.A. company uses acid industrial effluent, coming from cooper ores extracted by the company, in the manufacturing process. Crystalline ammonium perrhenate is obtained in the first place, which is next sintered in the form of so-called pellets and is reduced to metallic rhenium (Fig. 1) [2-4]. Global rhenium deposits can be found in particular in Chile, United States, Canada, Kazakhstan, Russia, Uzbekistan and Peru [1,8-9].

Table 1.
Examples of metallic rhenium manufacturing methods (own study based on [1,10])

Method	Characteristics of the method	Input material
Reduction of rhenium compounds with hydrogen	The source of rhenium is potassium or ammonium perrhenate. The process temperature is 600-800°C. Reduction with hydrogen occurs optionally in accordance with the following chemical reactions: $\mathbf{2KReO_4 + 7H_2 \rightarrow 2Re + 2KOH + 6H_2O}$ or $\mathbf{2NH_4ReO_4 + 7H_2 \rightarrow 2NH_3 + 2Re + 8H_2O}$ Perrhenate can be reduced with hydrogen also at lower temperatures of 200-300°C at the pressure of 2.03-10.13 MPa	Metal in form of powder
Electrolytic precipitation of rhenium from solutions	Cathodic precipitation of rhenium from salt solutions, during which potassium perrhenate and sulphuric acid often form part of the bath. The recommended electrolysis temperature is 25-90°C. An anode is made of platinum and cathode is made of rhenium. Rhenium is produced with the electrolytic method as a result of the following chemical reactions: $\mathbf{ReO_4 + 8H + 7e \Leftrightarrow Re + 4H_2O}$ $\mathbf{7H^+ + 7e \Leftrightarrow 7H}$ $\mathbf{ReO_4 + 4H_2O \Leftrightarrow Re + 8OH}$ $\mathbf{Re^{7+} + 7e \Leftrightarrow Re}$	Re sediments are brittle and foamy, usually in form of coating
Thermal dispersion of rhenium halides	Pairs of chlorides or other rhenium halides are passed over an item heated to incandescence by way of resistance for coating deposition. The process temperature is 1000-1800°C. Thermal decomposition may be conducted in vacuum or at atmospheric pressure in a protective atmosphere of H_2+Ar	Solid, glossy and compact metallic coatings

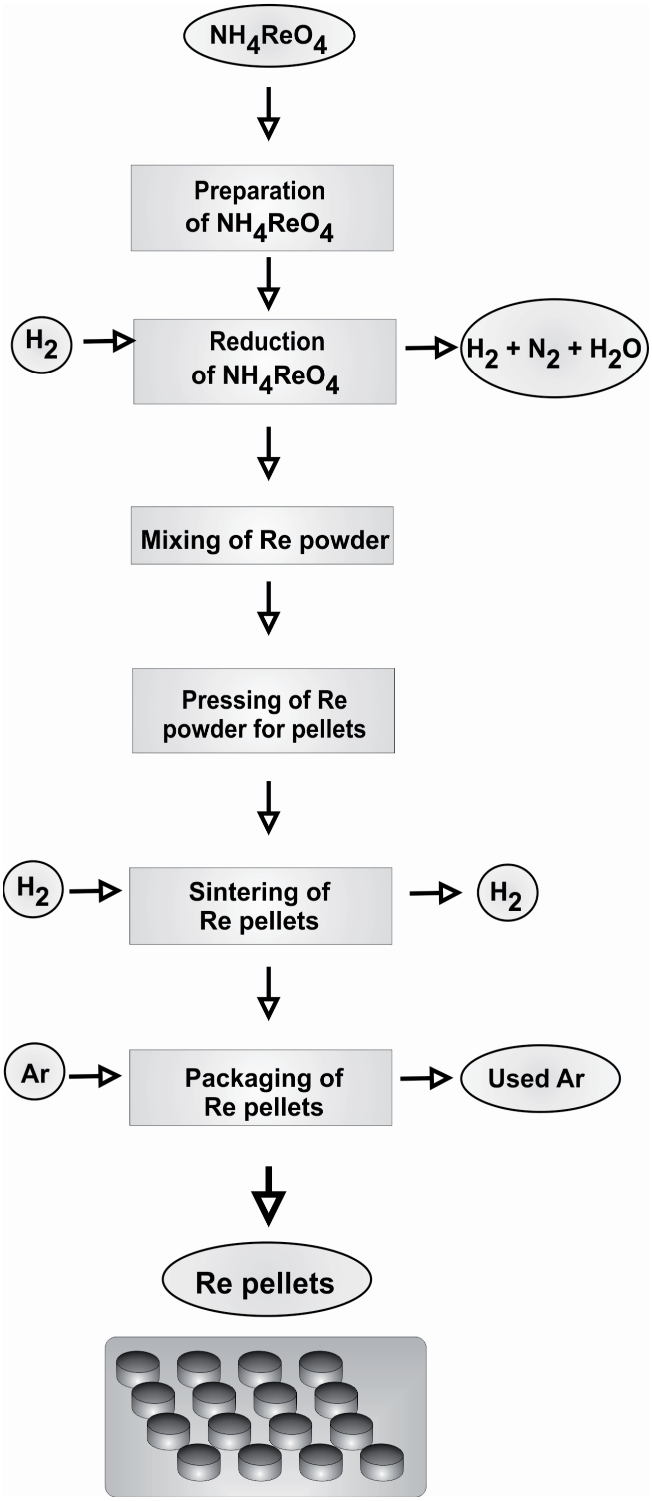

Fig. 1. Stages of metallic rhenium manufacturing in form of pellets at KGHM Polska Miedź S.A. company (based on [3,4])

3. Physiochemical properties of rhenium

Rhenium, being a group 7 element of the periodic table, has similar physical properties to group 6 metals, i.e. molybdenum and wolfram. On the other hand, it exhibits characteristics typical for platinum group metals (Pt, Ru, Os) [1,10-11]. Rhenium is a heavy metal with the density of 21.02 g/cm^3, with its solid state similar to steel. It features very high melting point and boiling point of, respectively, 3182°C and 5597°C. It is also a very hard chemical element and is wear resistant, and as a component of superalloys it improves their heat resistance and strength. An additive of Re in heavy alloys with tungsten and molybdenum causes the occurrence of so-called "rhenium effect", described by Savitski [12-16]. It is exhibited by significant changes in the alloy microstructure shown in Figure 2. The positive activity of Re in heavy sinters makes it particularly suitable for applications in cores of armour-piercing sabot shells [12]. Table 2 lists the key physiochemical properties of rhenium. In terms of chemical properties, rhenium is similar to magnesium and other group 7 elements. In terms of oxidisation resistance, rhenium shows higher stability than, e.g. tungsten.

Solid rhenium is stable at room temperature, but it starts to oxidise at temperature above 300°C. Rhenium dissolves in nitric acid, it reacts weakly with heated concentrated sulphuric acid, but does not react with sulphur acid and hydrofluoric acid. Rhenium does not react with nitrogen nor hydrogen, but strongly absorbs hydrogen when occurring in the form of powder [1,10].

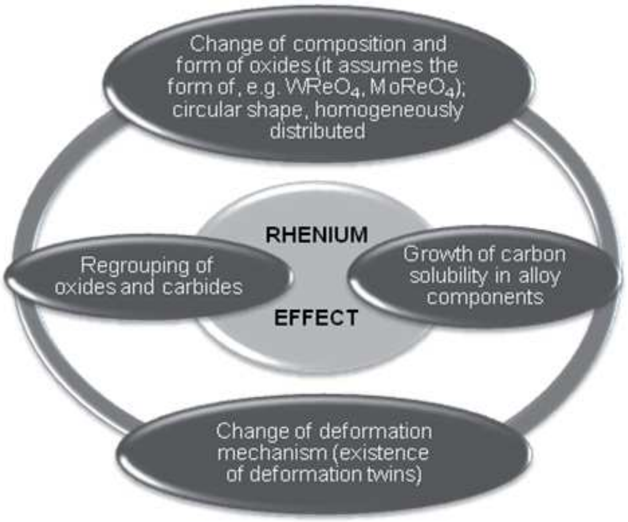

Fig. 2. "Rhenium effect" existence (own study based on [12-16])

Table 2.
Physiochemical properties of rhenium (own study based on [1,17-20])

Property	Value	Property	Value
State of concentration at room temperature	solid body	Hardness at Brinell scale	165
Density	21.03 g/cm^3	Hardness at Rockwell A scale	52
Molar mass	186.207 g/mol	Hardness at Vickers scale	170
Melting point	3,182°C	Tensile strength	1070 MPa (620 MPa at 800°C)
Boiling point	5,597°C	Yield point	290 MPa
Atomic number	75	Elongation at rupture	15-25%
Electronegativity	1.9	Modulus of elasticity	469 GPa
Valence	±I, II, III, IV, V, VI, VII	Specific heat	138 J/(kg·K) (at 25°C)
Crystallographic system	hexagonal close packed	Heat conductivity	48 W/(m·K)
Electron configuration	$1s^2 2s^2 2p^6 3s^2 3p^6 3d^{10} 4s^2 4p^6 4d^{10} 4f^{14} 5s^2 5p^6 5d^5 6s^2$		

4. Rhenium applications in industry

Excellent physiochemical properties make rhenium suitable for applications in progressive industries. Statistics inform that rhenium is used in 80% as a component of superalloys. Products in the aviation and space industry are fabricated mainly using alloys containing Re, which cannot be replaced in this case [1,11,18-23]. Rhenium is used for production of components of jet engines, turbine blades, shields, combustion chambers, where one of the key parameters is high temperature. The production of thermoelements, especially thermocouples, and heating elements is an important field of applications for Re (working temperature reaching 2000°C). Rhenium is used for production of heat resistant and high-temperature resisting alloys (rhenium alloys with nickel, chromium, molybdenum, titanium) for protective coatings, as well as for elements of sea ships – due to resistance to the activity of air saturated with sea water. It is also used as a material for manufacturing incandescent parts, fibres and cathodes, electron guns, as it has lower volatility compared with tungsten, which is often used for this purpose; it also features higher resistance to high temperatures [3-5,10,24].

The use of rhenium in medicine must also be mentioned. The radioactive isotopes ^{186}Re and ^{188}Re are employed in diagnostics and for destruction of tumorous cells [24-25]. Rhenium and its alloys (e.g. rhenium-tungsten alloys) also work very well in the conditions of an electric arc, for this reason they are often utilised for elements of electrical equipment, including electric contacts [10]. The application areas of Re include also analytical chemistry, catalysis, whereas rhenates, perrhenates, rhenium halides, rhenium oxides and acids are used as impregnation solutions containing dissolved rhenium [26]. This element is a very important catalyst used for production of high-octane fuels. Bimetallic Pt/Re catalysts are successfully replacing other – monometallic catalysts. In case of lead-free fuel, they are used for improving the progress of chemical reactions at high temperature and at lower pressure, which is automatically increasing production process efficiency and improves fuel quality [5, 27]. Rhenium is manufactured under different forms: pellets, powders, rods, wires, plates, sheets and foils (Fig. 3) [28].

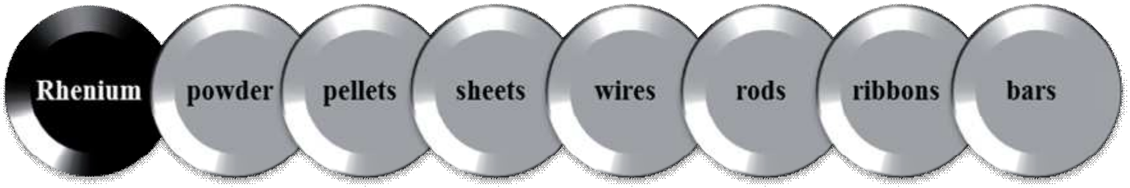

Fig. 3. Trade products manufactured from rhenium (based on [28])

5. Rhenium at nanometric scale

Examples are available in literature of rhenium fabrication, e.g. in form of nanoparticles [29], nanowires [30-32]. An interesting example can be found in a publication by Brorson et all [32] of the utilisation of multi-walled carbon nanotubes as a template for the manufacturing of ReS_2 nanotubes. A comprehensive patent also exists for the fabrication methods of nanomaterials based on Re and Re and other metals such as In, Sn, Sb, Pb and Bi (binary Re-metal nanostructures). The publication [26] presents the examples of the most popular rhenium precursors such as: NH_4ReO_4, $KReO_4$, $NaReO_4$. The examples of interesting forms of rhenium at a nanometric scale are presented in Table 3.

Table 3.
Selected examples of rhenium or rhenium compounds production in form of nanoparticles or other nanoforms (own study based on [29-38])

Nanomaterial type	Notes	Method	Source
Re nanoparticles	Production of nanoparticles due to the milling of rhenium precursor	Mechanochemical synthesis	[29]
Re nanowires	Re-Sn nanowires fabricated by deposition of a rhenium layer on the previously produced zinc nanowires, owing to which a core/shell structure was achieved	Electrodeposition	[30]
Re nanowires	Fabricated by selecting etching of the produced eutectic NiAl-Re alloy (1.5% of Re atomically); it was shown that the yield point of Re nanowires (measurement made with AFM probe blade) is 100 times higher than a value measured for mass rhenium; it was calculated that the ratio of distance between the Re nanowires growing vertically from the substrate and their diameter is 9.1	Selective etching of eutectic alloy containing Re	[31-34]
Single-walled $ReSe_2$ nanotubes	$ReSe_2$ nanotubes were produced as a result of ammonium perrhenate reaction with the previously prepared Sn nanowires (Se nanowires acted as a template) in solvothermal conditions at 135°C, the reaction lasted 6 days. The reaction takes place according to the following formula: $2NH_4ReO_4 + 5Se /2ReSe_2 + H_2SeO_3 + 2NH_3 + 5/2O_2$	Chemical synthesis in solvothermal conditions	[35]
ReS_2 nanotubes	Carbon nanotubes were used as a template, immersed in a medium containing: $ReCl_5$ dissolved in ethanol and NH_4ReO_4 dissolved in water. The suspension was treated with ultrasounds, then a solvent was evaporated and the powder obtained was heated at the temperature of 1000°C for 3 hours in the H_2S atmosphere. The interval between neighbouring walls of carbon nanotubes was 0.5-0.7 nm	Procedure consisting of several stages using carbon nanotubes as a template	[36]
Multi-walled ReS_2	Fullerene-like nanostructures comprised of ReO_x nanoparticles surrounded with ReO_2 fullerene. They were manufactured by xanthating with the use of H_2S, rhenium compound – ReO_2, similar to manual and mechanical milling	Procedure consisting of several stages	[37-38]

6. Rhenium as component of carbon-metallic nanocomposites

Information concerning the use of rhenium at a nanometric scale in combination with carbon nanotubes is limited to occasional publications. A review of literature shows that interesting results of investigations were published concerning core filling of single-walled nanotubes with Re nanocrystals. The investigations were conducted by a group of scientists headed by Zhang [39]. A group of Russian researchers tackled the topic of fabrication of multi-walled carbon nanotubes decorated with Re with the CVD method [40]. Efforts were also made to decorate graphene with rhenium occurring in form of ultra-small rhenium clusters consisting of 2-13 atoms of Re [41].

Considering the interesting perspectives and the fact that the topic is new, own research was undertaken [42-44] to investigate and describe the structure of carbon-metallic nanocomposites consisting of nanostructured rhenium permanently attached to carbon nanomaterials, in the form of single-walled (SWCNTs), double-walled (DWCNTs) or multi-walled carbon nanotubes (MWCNTs) or single-walled carbon nanohorns (SWCNHs). Transmission Electron Microscopy (TEM) was employed for this purpose and the results are presented in Figures 4-9. It was found in the course of research works that implementation conditions for particular stages of the manufacturing process of newly created nanocomposites composed of nanostructured rhenium permanently attached to carbon nanomaterials, have a significant effect on the structure of the created carbon-metallic nanocomposites. The following are the stages, in particular: (1) functionalisation of carbon nanomaterials; (2) impregnation of carbon nanomaterials in rhenium precursor; (3) reduction of rhenium precursor as a result of heating at the temperature of 800°C and in the atmosphere of hydrogen. The type of the carbon nanomaterial used is conditioning the structure of newly developed nanocomposites. Different carbon nanomaterials feature different chemical reactivity, hence they exhibit different resistance to intensive treatment when nanocomposites are produced from them [44].

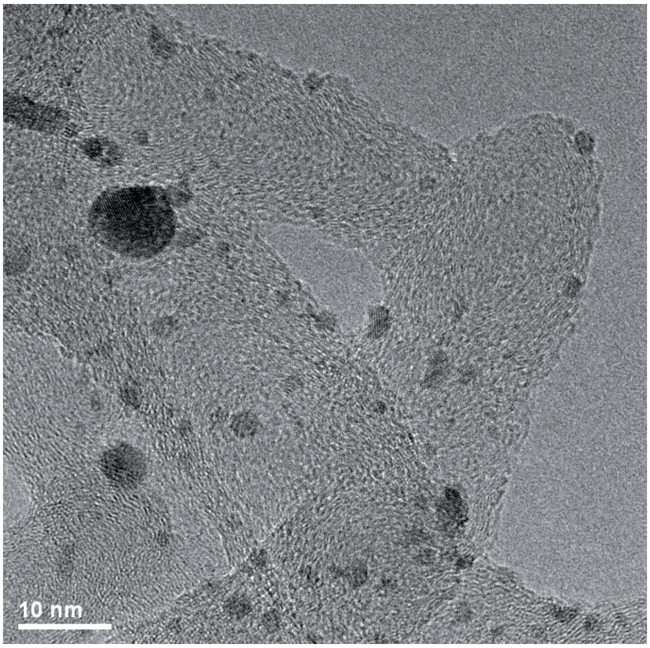

Fig. 5. Nanocomposite of MWCNTs-Re type; multi-walled carbon nanotubes with Re nanoparticles covering their external walls; HRTEM image in bright field [44]

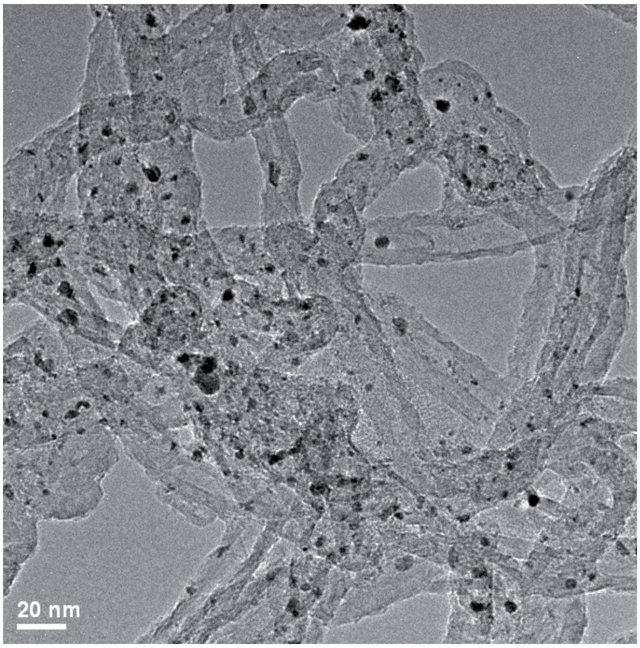

Fig. 4. Nanocomposite of MWCNTs-Re type; cluster of multi-walled carbon nanotubes with uniformly deposited rhenium nanoparticles; HRTEM image in bright field [44]

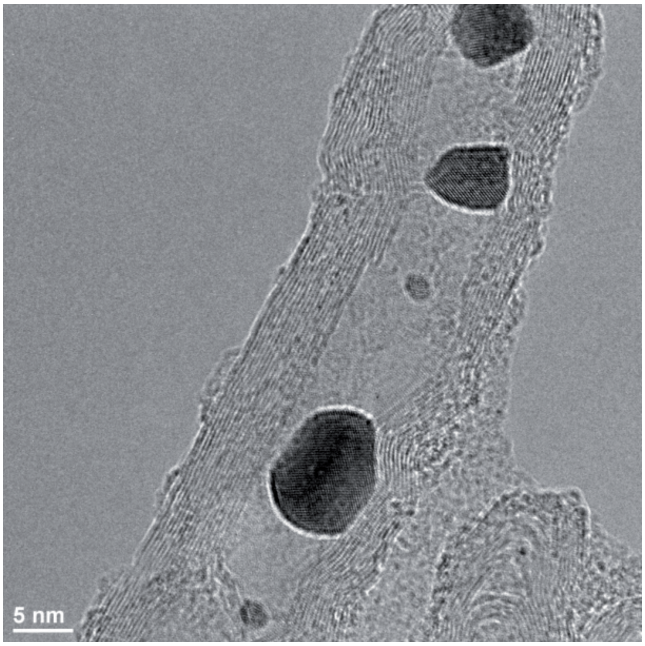

Fig. 6. MWCNTs-Re nanocomposite; a visible multi-walled carbon nanotube with Re nanoparticles in its core; HRTEM image in bright field [44]

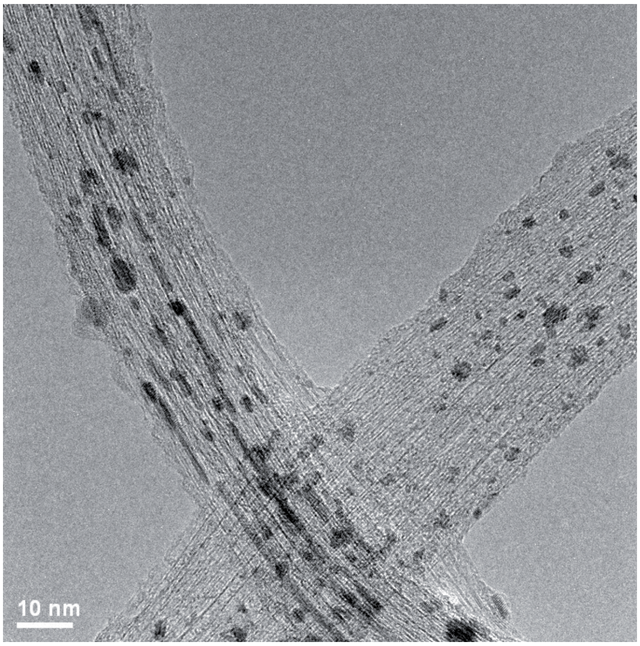

Fig. 7. SWCNTs-Re-type nanocomposite in form of SWCNTs bundles decorated with rhenium nanoparticles, with visible Re nanowires in the core of nanotubes; HRTEM image in bright field [44]

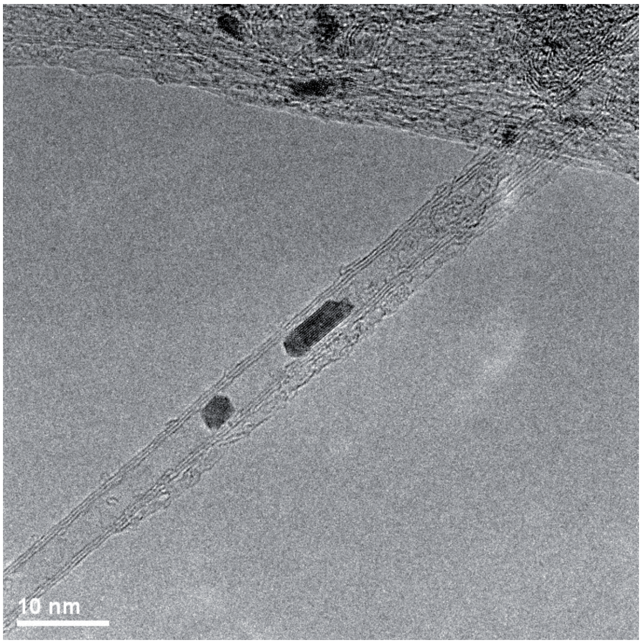

Fig. 8. SWCNTs/DWCNTs-Re-type nanocomposite; rhenium nanoparticles placed in the core of carbon nanotube; HRTEM image in bright field [44]

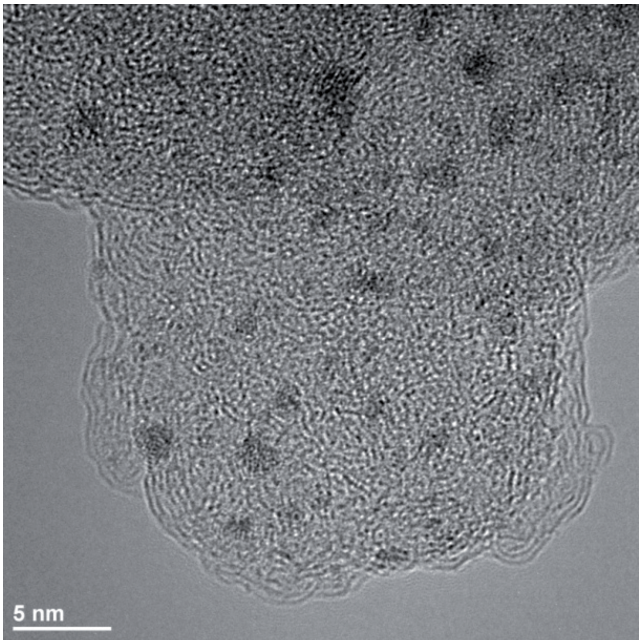

Fig. 9. SWCNHs-Re nanocomposite in form of cluster of single-walled carbon nanohorns decorated with rhenium nanoparticles; HRTEM image in bright field [44]

An inspiration for creating carbon-rhenium nanocomposites is a combination of their good properties enabling the future use of such materials in progressive industries. Nanocomposites, consisting of carbon nanotubes decorated with nanoparticles of metals, have a strongly expanded surface and are suitable, in particular, for uses as active elements of gas sensors or liquid sensors, especially for detecting biological substances in a human organism. The more expanded is the sensor surface, the greater active area in which reactions are occurring with the molecules of a given chemical substance. Catalysts whose task is to improve and accelerate chemical reactions are used in different forms. In case of the newly created nanocomposites presented in the article, carbon nanotubes act as a carrier, whose task is to expand an active area of a catalyst and to additionally stiffen and increase the resistance of the catalyst, while metal nanoparticles are acting as active centres.

7. Recapitulation

The article characterises physical and chemical properties of rhenium. Manufacturing methods are discussed and key applications are listed. The examples of rhenium at a nanometric scale are also presented, taking into account the latest literature reports in this field. Apart from strategic applications in the aviation and space sector, rhenium is utilised in the chemical and petrochemical branch as a catalyst in production of high-octane fuels. Single- and multi-walled carbon nanotubes have enjoyed major interest in the world of science and industry. Due to the presence of an empty core in their structure, they have low density (1-2 g/cm^3) and excellent strength properties. Given their excellent electrical properties, carbon nanotubes are finding applications in modern electronics, especially in transistors, memories and displays. Nanotubes and other interesting carbon nanomaterials work very well also as active elements of gas and liquid sensors. It is suitable to use rhenium, a rare chemical element possessing multiple good physiochemical properties, as a component of a carbon-metallic composite. Such a combination makes those modern materials appropriate for applications in progressive industries. A method was therefore elaborated of attaching nanostructured rhenium permanently to carbon nanomaterials, such as multi-walled nanotubes, single- and double-walled nanotubes and carbon nanohorns [44]. A structure of nanocomposites created as a result is dependent on the execution conditions of particular steps of the manufacturing process encompassing functionalisation in an oxidising substance, impregnation in a rhenium precursor and reduction of this precursor in an atmosphere of hydrogen at a high temperature. The current trends in research over improved or completely new nanomaterials applied in high-tech industry indicate the need and necessity of searching such materials which combine a strictly designed structure and pre-defined physiochemical properties. The most important criteria essential for a good quality of the established carbon-rhenium nanocomposites are found to be: homogeneous dispersion of rhenium nanoparticles, relatively low distribution of rhenium nanoparticles' diameters and no tendency to agglomerate rhenium nanoparticles.

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