Tonkie Khimicheskie Tekhnologii = Fine Chemical Technologies.
2024;19(3):258–266 ISSN 2686-7575 (Online)

Chemistry and technology of inorganic materials

Химия и технология неорганических материалов

UDC 54.061
https://doi.org/10.32362/2410-6593-2024-19-3-258-266
EDN KYWKWJ

Solid solutions in disulfide systems Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2, and Re(IV)S_2–W(IV)S_2

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Abstract

Objectives. Chalcogenides of transition elements with low oxidation states, as well as their substituted derivatives, remain a poorly studied class of chemical compounds. Rhenium disulfide has many distinctive features and great application potential as a new two-dimensional semiconductor. This is due to its unusual structure and unique anisotropic properties. The presence of weak interlayer bonding and a unique distorted octahedral (1T) structure suggests the possibility of creating new phases on its basis. The aim of this work is to obtain and study phases in systems Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2, and Re(IV)S_2–W(IV)S_2.

Methods. The samples were obtained by high-temperature solid-phase ampoule synthesis in a vacuum. The study was carried out using X-ray phase analysis and X-ray photoelectron spectroscopy.

Results. The regions of existence of solid solutions, intercalates and two-phase regions in the resulting systems were established. Diffraction patterns were obtained for the new phases and the crystal lattice parameters were calculated. Based on data relating to the binding energies of core electrons with the nucleus, the study showed the valence states of the elements after synthesis. The study also confirmed that all phases obtained as a result of synthesis contain transition elements in the oxidation state (IV).

Conclusions. Intercalated solid solutions are formed in areas rich in rhenium, while in areas close to titanium and molybdenum disulfides, intercalated phases are attained. In the ReS_2–WS_2 system there is a region of solid solutions, including 30, 50, and 70 mol % rhenium disulfide. Their structure is a polymorphic modification of the structure of the original components. The presence of rhenium, molybdenum, and tungsten in these phases in the oxidation state (+IV) was confirmed. The data obtained on phase formation in dichalcogenide systems can be practically used in the creation of materials with unique electronic, magnetic, and optical properties with a wide range of applications.

Keywords

solid solutions, intercalates, transition metal disulfides, phase formation, crystal lattice, binding energies

Submitted: 07.11.2023
Revised: 08.12.2023
Accepted: 11.04.2024

For citation

Efremova E.I., Lazov M.A., Kobrin M.R., Fomichev V.V. Solid solutions in disulfide systems Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2, and Re(IV) S_2–W(IV)S_2. *Tonk. Khim. Tekhnol. = Fine Chem. Technol.* 2024;19(3):258–266. https://doi.org/10.32362/2410-6593-2024-19-3-258-266

НАУЧНАЯ СТАТЬЯ

Твердые растворы в системах дисульфидов Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2 и Re(IV)S_2–W(IV)S_2

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Аннотация

Цели. Халькогениды переходных элементов с низкой степенью окисления, а также их замещенные производные до сих пор являются малоизученным классом химических соединений. В качестве нового двумерного полупроводника дисульфид рения имеет множество отличительных особенностей и обладает большим потенциалом для применения благодаря своей необычной структуре и уникальным анизотропным свойствам, а наличие у данного соединения слабой межслойной связи и уникальной искаженной октаэдрической (1T) структуры позволяет предположить возможность создания новых фаз на его основе. Цель данной работы — получение и исследование фаз в системах Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2 и Re(IV)S_2–W(IV)S_2.

Методы. Образцы были получены методом высокотемпературного твердофазного ампульного синтеза в вакууме. Исследование проводили методами рентгенофазового анализа и рентгеновской фотоэлектронной спектроскопии.

Результаты. Установлены области существования твердых растворов, интеркалатов и двухфазных областей в полученных системах. Для новых фаз получены дифрактограммы и рассчитаны параметры кристаллической решетки. По данным энергий связи остовных электронов с ядром показано, в каких валентных состояниях находятся элементы после синтеза, подтверждено, что все полученные в результате синтеза фазы содержат переходные элементы в степени окисления (IV).

Выводы. В богатых рением областях образуются твердые растворы по типу внедрения, в то время как в областях, близких к дисульфидам титана и молибдена, реализуются интеркалированные фазы. В системе ReS_2–WS_2 существует область твердых растворов, включающая 30, 50 и 70 мол. % дисульфида рения, структура которых является полиморфной модификацией структуры исходных компонентов. Подтверждено присутствие рения, молибдена и вольфрама в этих фазах в степени окисления (+IV). Полученные данные о фазообразовании в системах дихалькогенидов могут быть практически использованы при создании материалов, обладающих уникальными электронными, магнитными и оптическими свойствами с обширной областью применения.

Ключевые слова

твердые растворы, интеркалаты, дисульфиды переходных металлов, фазообразование, кристаллическая решетка, энергии связи

Поступила: 07.11.2023
Доработана: 08.12.2023
Принята в печать: 11.04.2024

Для цитирования

Ефремова Е.И., Лазов М.А., Кобрин М.Р., Фомичев В.В. Твердые растворы в системах дисульфидов Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2 и Re(IV)S_2–W(IV)S_2. *Тонкие химические технологии.* 2024;19(3):258–266. https://doi.org/10.32362/2410-6593-2024-19-3-258-266

INTRODUCTION

Layered transition metal dichalcogenides are a relevant area of numerous scientific studies. This is due to the variety of physical and physicochemical properties of materials based on these compounds [1–3]. However, transition element chalcogenides with low oxidation degree, as well as their substituted derivatives, are still an understudied class of chemical compounds [4].

Transition element dichalcogenides are of interest due to their layered structure and a wide range of electrical properties from semiconducting in group 4 (d0) to metallic in group 5 (d1), then back to semiconducting in group 6 (d2). Here d0, d1, and d2 are the number of free valence electrons which remain after bond formation in dichalcogenides [5]. Dichalcogenides can also act as superconductors [6]. Furthermore, dichalcogenides are known for their segmentoelectric, catalytic and optical properties, and also have a forbidden zone of tunable widths [7].

Solid solutions and doped compounds of transition metal disulfides are important objects of research in chemical technology (materials science). This is due to the variety of electronic, magnetic and optical properties for which they are used in electronics (second harmonic generators, field-effect transistors), catalysis and in power engineering (solar cells) [2]. The combination of different properties (e.g., semiconductor and dielectric properties) can lead to interesting results and promising applications of the materials [9, 10]. Transition metals and their sulfides can be used as additives designed to improve the properties of the original layered materials. For example, rhenium sulfide is used as an additive to a photocatalyst for hydrogen evolution, or to cadmium sulfide, in order to increase the speed of this process. In the future this will allow us to completely move away from catalysts based on noble metals [11–13].

Rhenium disulfide (ReS_2) forms two-dimensional (2D) layered crystals [14]. As a novel 2D semiconductor, rhenium disulfide has many distinctive features. It also has great potential for applications due to its unusual structure and unique anisotropic properties [15]. Due also to its crystal structure, rhenium disulfide has many distinctive characteristics when compared with titanium, molybdenum and tungsten dichalcogenides: it has weak interlayer bonding and a unique distorted octahedral structure (structure type 1T). Due to such distinctive characteristics, bulk and monolayer ReS_2 have almost identical zone structures, and both of them belong to direct-gap semiconductors [16, 17][1].

Combination of the properties of 4th and 6th groups of disulfides (titanium, molybdenum, tungsten) with the properties of rhenium disulfide in a single structured phase of the host-guest type, can be assumed to result in a change in their properties.

The aim of the present work is the synthesis of new phases: solid solutions in the $Re(IV)S_2$–$Ti(IV)S_2$, $Re(IV)S_2$–$Mo(IV)S_2$, and $Re(IV)S_2$–$W(IV)S_2$ systems.

MATERIALS AND METHODS

The following initial substances were used in the work: rhenium—metal powder with the content of the main component not less than 99.9% of rhenium (GOST 25278.16-87[2]), titanium—metal powder, chemically pure (Company Standard Technical Specification KOMP 3-272-10), tungsten—metal powder, chemically pure (Company Standard Technical Specification KOMP 3-684-13), elemental sulfur, especially pure (*Component-Reactive*, Russia, TU 3-304-10), and molybdenum disulfide (*Spets Metal Master*, Russia, TU 48-19-133-90). Other dichalcogenides were obtained by direct interaction of powders of corresponding metals (titanium, tungsten, and rhenium) and elemental sulfur.

The synthesis of rhenium disulfide was carried out by the method described above [4]. The suspensions of metallic rhenium and elemental sulfur were placed in a quartz ampoule, vacuumized (pressure 10^{-5} atm), sealed, and placed in a muffle furnace. Synthesis was carried out in the following mode. Over a period of 5 h the ampoule with the reaction mixture was gradually heated to a temperature of 1000°C and kept at this temperature for 20 h. Then the ampoule with the sample was cooled in the furnace for a day. The yield of the product by sulfur is stoichiometric. Titanium and tungsten disulfides were prepared by a similar method.

The synthesis of samples in the double disulfide systems $Re(IV)S_2$–$Ti(IV)S_2$, $Re(IV)S_2$–$Mo(IV)S_2$, and $Re(IV)S_2$–$W(IV)S_2$ was carried out in the following way. The disulfides were mixed in stoichiometric amounts. Then the resulting charge was homogenized in a ball mill for 30 min and then sealed in a vacuum-quartz ampoule. The synthesis was carried out in a muffle furnace and heated to 1100°C for 10 h. It was then kept at this temperature for 48 h, and then spontaneously cooled. Samples of double sulfides were prepared with the ratio of the main component from 0 to 100%.

X-ray studies were performed on a XRD-6000 diffractometer (*Shimadzu*, Japan) (Cu-K_α radiation, $2\theta = 10°–60°$, imaging step 0.2 deg/min, exposure 10 s). A ICDD-JCPDS[3] card index was used to identify the phases (rhenium disulfide (card 89-0341), trigonal crystal lattice; titanium disulfide (card 15-853), hexagonal crystal lattice; molybdenum disulfide (card 17-744), hexagonal crystal lattice; tungsten disulfide (card 8-237), hexagonal crystal lattice).

Studies of the electronic structure and composition by X-ray photoelectron spectroscopy were carried out on a Kratos AXIS Ultra DLD electron spectrometer (*Kratos Analytical*, United Kingdom) with a monochromatized X-ray source Al Kα ($h\nu$ = 1486.6 eV, energy resolution 0.5 eV).

RESULTS AND DISCUSSION

Phase formation in the ReS_2–TiS_2 system

Diffractograms of some samples of the system in the titanium-rich region are shown in Fig. 1. The calculated unit cell parameters of these phases are presented in Table 1. It should be noted that the nature of the X-ray

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[1] BE Lookup Table for Signals from Elements and Common Chemical Species. URL: https://xpslibrary.com/wp-content/uploads/2019/07/BE_Lookup_table.pdf. Accessed October 18, 2022.

[2] GOST 25278.16-87. State Standard of the USSR. Alloys and foundry alloys of rare metals. Methods for determination of rhenium. Moscow: Izdatel'stvo standartov; 1998.

[3] International Center for Diffraction Data (former name—Joint Committee on Powder Diffraction Standards). https://www.icdd.com/. Accessed December 28, 2023.

diffractograms of the initial components, namely the insufficient number of reflexes (for example, the number of reflexes is four for rhenium disulfide according to the ICDD-JCPDS card catalog), may lead to an error in the calculation of lattice parameters of solid solutions. However, this does not affect the determination of phase formation of the synthesized samples. The results of X-ray phase analysis established the invariability of lattice parameters in this region of rhenium disulfide concentrations, allowing us to speak about the formation of solid solutions of $Re_xTi_{(1-x)}S_2$ composition in the range $0 < x \leqslant 0.04$. With increasing Re content, the existence of a two-phase region with the presence of TiS_2 was noted.

Analysis of Fig. 2 shows that compositions with rhenium disulfide content of 50% have two phases, and with a content of 60% and more—one phase. Thus, solid solutions are established in a wide region rich in rhenium. It can also be seen that in compositions with rhenium content of 60% and 70%, the reflexes 34° and 57.7° are present. These belong to the rhenium disulfide phase which is consistent with the data obtained from the JCPDS database (card 89-0341). Calculation of lattice parameters was performed for the solid solutions obtained (Table 1). When analyzing the results, it should be borne in mind that the ionic radii of all transition elements in the oxidation degree (IV), considered in this work, are approximately equal and are ~0.6 Å [18]. In this case, the consistency of the lattice parameters may indicate the formation of solid substitution solutions based on both titanium disulfide and rhenium disulfide. At the same time, it can be assumed that the changes observed in the *c* parameter indicate the formation of intercalated compounds based on rhenium disulfide. This is natural if we assume that the layers in the structure of rhenium disulfide are weakly connected and that the interlayer distance of rhenium disulfide is larger than that of titanium disulfide. Noticeable changes caused by the intercalation of rhenium in TiS_2 include the broadening

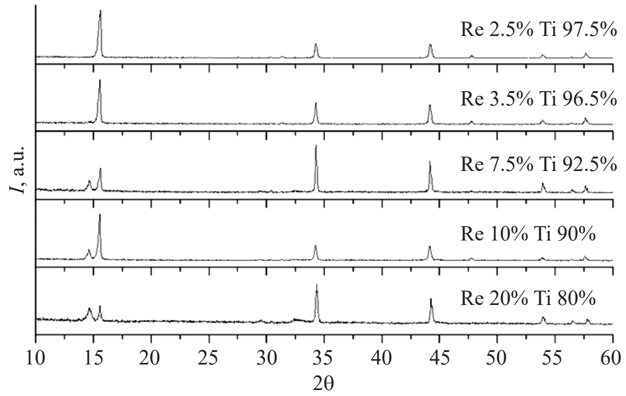


Fig. 1. Diffraction patterns of some samples of the system ReS_2–TiS_2, including solid solutions of composition substitution $Re_{1-x}Ti_xS_2$ (2.5 and 3.5% Re)

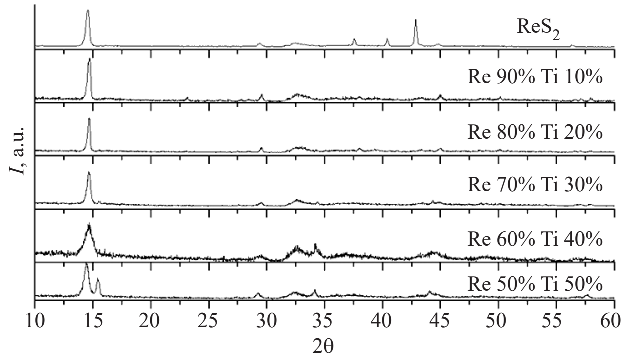


Fig. 2. Diffraction patterns of the original ReS_2 and solid solutions synthesized from it with the nominal composition $Re_{1-x}Ti_xS_2$

Table 1. Crystal lattice parameters and bond energies of some phases $Re_xTi_{(1-x)}S_3$

Composition	*a*, Å	*c*, Å	*V*, Å^3	Re bond energy, eV	Ti bond energy, eV
ReS_2, trigonal crystal lattice	6.35(5)	12.78(15)	434.49	41.00	–
ReS_2 95%	6.39(5)	12.23(15)	432.39	42.06	–
ReS_2 90%	6.41(5)	12.27(15)	436.21	–	–
ReS_2 80%	6.55(5)	12.76(15)	474.91	–	–
ReS_2 70%	6.41(5)	12.28(15)	437.66	–	–
ReS_2 3.5%	3.40(4)	5.69(6)	56.93	40.92	456.09
TiS_2, hexagonal crystal lattice	3.40(4)	5.69(6)	57.14	–	457.20

Note: a and *c* are lattice measurements, *V* is unit cell volume.

of the diffraction maxima (compared to the original ones) and a decrease in their relative intensity. This effect may be due to the appearance of microstresses in the host lattice or a decrease in the crystallite size.

X-ray photoelectron spectra were measured for titanium(IV) sulfide and samples of three compositions: $Re_{0.035}Ti_{0.965}S_2$, $Re_{0.95}Ti_{0.05}S_2$, and a sample from the two-phase region with rhenium(IV) sulfide content of 20% (Table 1). The Ti 2p line has an energy of 457.2 eV, corresponding to tetravalent titanium in the sulfides. In samples $Re_{0.035}Ti_{0.965}S_2$ and $Re_{0.95}Ti_{0.05}S_2$ both peaks of titanium were shifted. This suggests the formation of solid solutions in the considered systems. Other forms of rhenium in the samples were not observed.

Phase formation in the ReS_2–MoS_2 system

Figure 3 shows the diffractograms of samples of the rhenium disulfide-molybdenum disulfide system with a different ratio of components (from 20 to 90%). The results of calculation of crystal lattice parameters are presented in Table 2. The table shows that the parameters of samples with different sulfide content differ from those of pure metal disulfides.

Analysis of the X-ray diffraction patterns enables two regions of solid solutions to be defined: on the basis of ReS_2—up to 35 mol % MoS_2, and on the basis of MoS_2—up to 20 mol % ReS_2. In the concentration range from 20 to 65 mol % MoS_2 a two-phase region is observed in the system. The MoS_2 based solid solutions show a change in both lattice parameters as the rhenium content increases. This may indicate the formation of a solid solution introduction. On the rhenium disulfide side, there is a sharp change in the *a* and *V* parameters for the phase containing 20 mol % MoS_2. This may suggest the formation of a solid solution of introduction on the basis of rhenium disulfide. Given weak interaction between ReS_2 layers, the introduction of molybdenum into the layer structure will naturally affect mainly the *a* parameter, while the *c* parameter may remain unchanged.

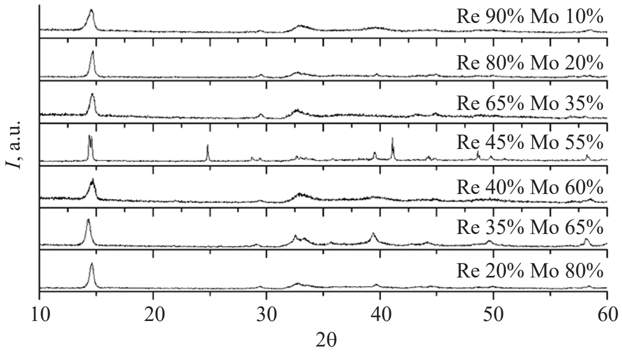


Fig. 3. Diffraction patterns of the ReS_2–MoS_2 system

Phase formation in the ReS_2–WS_2 system

According to the X-ray diffraction patterns of samples of ReS_2–WS_2 system (Fig. 4) and calculated parameters of crystal lattices (Table 3), a conclusion can be drawn about the formation of solid solutions in the region with the content of rhenium disulfide from 30 to 70%. Figure 4 shows that X-ray diffraction patterns of samples with rhenium disulfide content of 70, 50, and 30% are characterized by additional reflections. This may indicate polymorphism in this part of the double system. Since the presence of polymorphic modifications in this system is characteristic only for tungsten disulfide, it can be assumed that polymorphism refers to this sulfide [19] specifically. The decrease in the values of the crystal lattice parameters of solid solutions compared to those of rhenium disulfide can be related to the strengthening of interlayer interaction during the introduction of WS_2 into the layers and the alignment of their wavy structure.

Table 2. Crystal lattice parameters and bond energies of solid solutions formed in the ReS_2–MoS_2 system

ReS_2 composition	*a*, Å	*c*, Å	*V*, Å^3	Re bond energy, eV	Mo bond energy, eV
MoS_2, hexagonal crystal lattice	3.161(18)	12.290(4)	106.43	–	229.50
ReS_2 20%	3.165(25)	12.254(87)	106.30	42.67	227.21
ReS_2 35%	3.173(46)	12.328(16)	107.50	41.15	226.31
ReS_2 80%	6.749(03)	12.727(5)	501.99	41.61	226.32
ReS_2, trigonal crystal lattice	6.352(52)	12.779(44)	434.49	41.00	–

X-ray photoelectron spectra of some synthesized samples of double systems (Tables 1–3, Fig. 5) show the presence of transition elements (titanium, molybdenum, tungsten, and rhenium) in oxidation degrees (IV). Pure disulfides of rhenium, molybdenum, tungsten, and titanium were used as reference compounds in analyzing the spectra. This concurs well with the literature date (see Footnote 1). Certain changes in the binding energy of the backbone electrons of these elements are due to the formation of solid solutions. This is natural, bearing in mind that in individual disulfides the metal-to-metal interatomic distances are small and comparable to the interatomic distances in metals.

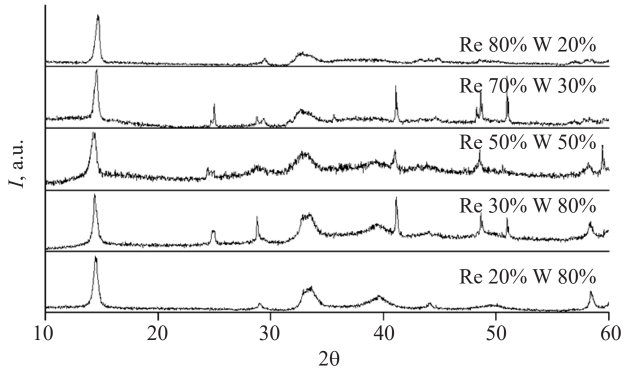
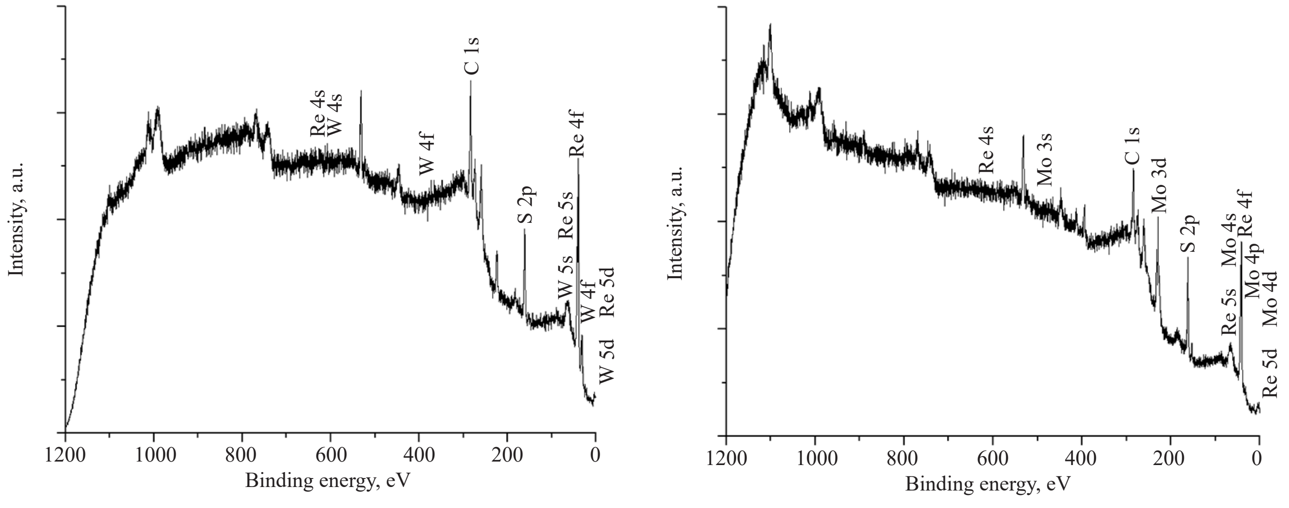


Fig. 4. Diffraction patterns of solid solutions in the ReS_2–WS_2 system

Table 3. Crystal lattice parameters and bond energies of solid solutions formed in the ReS_2–WS_2 system

ReS_2 composition	*a*, Å	*c*, Å	*V*, Å^3	Re bond energy, eV	W bond energy, eV
WS_2, hexagonal crystal lattice	3.15(6)	12.31(54)	153.76	–	32.00
ReS_2 20%	3.29(7)	12.4(87)	169.05	42.11	33.88
ReS_2 30%	5.75(62)	12.45(33)	519.29	41.27	32.97
ReS_2 70%	5.39(7)	12.64(56)	462.59	40.34	32.01
ReS_2 80%	5.71(98)	12.69(12)	521.08	42.62	34.67
ReS_2, trigonal crystal lattice	6.35(21)	12.78(65)	649.67	41.00	–



(a) (b)

Fig. 5. Survey spectrum of the ReS_2 35%–WS_2 65% solid solution (a) and the ReS_2 80%–MoS_2 20% solid solution (b)

Analysis of the chemical state of atoms (rhenium, molybdenum, and sulfur) on the surface of solid solution sulfides $Re_{0.8}Mo_{0.2}S_2$ consisted in a detailed study of the spectra of the electronic levels of Re 4f, Mo 3d, and S 2p. This enabled us to quantitatively characterize the binding energies of the backbone electrons with the nucleus.

In the X-ray photoelectron spectrum of Re 4f spin-orbit splitting of the peak occurs, in all samples the element is characterized by the presence of two lines. This is consistent with the literature date (see Footnote 1). In samples containing molybdenum, the binding energies of both peaks shifted, possibly indicating the formation of solid solutions in the systems under consideration. Other forms of rhenium in the samples were not observed.

Molybdenum is present in the spectrum of solid solution with a rhenium content of 20% in two lines, and in three lines in the rest of the samples. This suggests the possible formation of the oxide form with Mo(IV) along with disulfide, as well as the presence of trace amounts of molybdenum in higher degrees of oxidation. The presence of these peaks prevents us from accurately determining the atomic ratio of elements in the samples obtained. Conclusions on energy redistribution were made only on the bonding energy. This corresponds to molybdenum in disulfide (Table 2). In the samples containing molybdenum and tungsten, the bond energies of both peaks shifted, possibly indicating the formation of solid solutions in the systems under consideration.

In all samples tungsten is represented by one line corresponding to the energy of the sulfide of the element with valence (IV). The samples show a change in the binding energy of the backbone electrons to the nucleus (Table 3), indicating redistribution of energy during the formation of solid solutions.

For the Re(IV)S_2–Ti(IV)S_2 system, the photoelectron spectra of three samples were investigated: intercalate of composition $Re_{0.035}Ti_{0.965}S_2$, solid solution of composition $Re_{0.95}Ti_{0.05}S_2$ and two-phase region with the content of rhenium(IV) sulfide 20%. The change of bonding energy allows us to judge about its redistribution, i.e. about the formation of solid solutions and intercalate compounds.

For titanium, the samples with rhenium also show doublets, one peak of which corresponds to titanium sulfide with changed value of binding energy. The second peak, absent in the pure titanium sulfide sample, may correspond to a more oxidized form of titanium which bonds to rhenium in a particular way. This data needs to be investigated in more detail. Conclusions on the energy redistribution were drawn only from the binding energy, corresponding to titanium in the disulfide.

CONCLUSIONS

The paper presents the results of the study of phase formation in the systems Re(IV)S_2–Ti(IV)S_2, Re(IV)S_2–Mo(IV)S_2, and Re(IV)S_2–W(IV)S_2. X-ray phase and X-ray photoelectron spectroscopic study of the isolated phases were carried out. Areas of existence of solid solutions in the systems were established. It was shown that in rhenium-rich areas, solid solutions of the embedding type are formed, while in areas close to titanium and molybdenum disulfides intercalated phases are attained. In the ReS_2–WS_2 system, there is a region of solid solutions including 30, 50, and 70 mol % of rhenium disulfide. The structure represents a polymorphic modification of the structure of the initial components (structural type CdI_2). The presence of rhenium, molybdenum and tungsten in these phases in oxidation degree (IV) was also confirmed.

Authors' contribution

All authors equally contributed to the research work.

The authors declare no conflicts of interest.

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[Original Russian Text: Golub A.S., Zubavichus Ya.V., Slovokhotov Yu.L., Novikov Yu.N. Single-layer dispersions of transition metal dichalcogenides in the synthesis of intercalation compounds. *Uspekhi khimii*. 2003;72(2): 138–158 (in Russ.).]

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Translated from Russian into English by H. Moshkov
Edited for English language and spelling by Dr. David Mossop

MIREA – Russian Technological University
78, Vernadskogo pr., Moscow, 119454, Russian Federation.
Publication date *June 27, 2024.*
Not for sale

МИРЭА – Российский технологический университет
119454, РФ, Москва, пр-т Вернадского, д. 78.
Дата опубликования *27.06.2024.*
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