

PRODUCTION OF MoO₂Cl₂

[0001] The invention relates to methods for producing molybdenum dioxide dichloride (MoO₂Cl₂) and to MoO₂Cl₂ obtainable by such methods, to the use thereof and to electronic components.

Prior art

[0002] Molybdenum-containing layers are frequently used in electronic components, for example in so-called flash memories. Such molybdenum-containing layers are often produced by means of vapor deposition. In such a vapor deposition, MoO₂Cl₂ is increasingly being used as a precursor for the molybdenum. In order to achieve advantageous electronic properties of the molybdenum-containing layers, the use of particularly pure MoO₂Cl₂ is desirable. In particular, the MoO₂Cl₂ should be as free as possible of metal impurities which can impair the electronic properties. A particular focus here lies on tungsten as an impurity, since tungsten can particularly impair the electronic properties of a molybdenum-containing layer. Accordingly, an MoO₂Cl₂ that is as tungsten-free as possible is particularly desirable.

[0003] The production of MoO₂Cl₂ is known per se. For example, Schrock et al. (J. Am. Chem. Soc., 1990, 112, 3875-3886) describe the production of MoO₂Cl₂ from molybdenum(IV) oxide (MoO₂) and chlorine gas (Cl₂). The reaction in the reaction vessel takes place at 160°C. A further active temperature setting of the test apparatus does not take place. The Cl₂ gas flow set is not specified, and the purity of the MoO₂Cl₂ produced is not determined.

[0004] A method for the preparation of molybdenum powder is known from CN 102632245 A, in which MoO₂Cl₂ is formed as an intermediate by reacting MoO₂ with Cl₂. No information is given regarding the atmosphere under which the reaction is carried out. No information is given regarding the purity of the MoO₂Cl₂ produced as an intermediate, either. The MoO₂Cl₂ produced as an

intermediate is subsequently reacted with aqueous ammonia to give ammonium paramolybdate. This is converted into molybdenum(VI) oxide (MoO_3), from which molybdenum powder is ultimately obtained.

5 [0005] EP 3 656 741 A1 discloses a method for the production of molybdenum oxide chlorides from a molybdenum oxide and Cl_2 under reduced pressure. In addition to MoO_2Cl_2 , the molybdenum oxide chlorides produced also comprise molybdenum oxide trichloride (MoOCl_3) and molybdenum oxide tetrachloride (MoOCl_4). In the exemplary embodiments, MoO_2Cl_2 is produced by reacting
10 MoO_3 with Cl_2 . The purity of the MoO_2Cl_2 produced is limited to 99.9995 wt.%.

[0006] The methods used in the prior art for producing MoO_2Cl_2 are in some cases still in need of improvement.

15 Object of the invention

[0007] The invention is based on the object of providing a method for producing MoO_2Cl_2 which, at least partially, and as fully as possible, overcomes the disadvantages of the prior art.

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[0008] It is a particular object of the invention to provide a method for producing MoO_2Cl_2 , which method gives particularly pure MoO_2Cl_2 , in particular MoO_2Cl_2 with a low tungsten content.

25 [0009] The invention is also based on the object of providing a method for producing a molybdenum-containing layer which, at least partially, and as fully as possible, overcomes the disadvantages of the prior art.

[0010] It is furthermore an object of the invention to provide MoO_2Cl_2 which at
30 least partially, and as fully as possible, overcomes the disadvantages of the prior art and in particular to provide MoO_2Cl_2 with improved purity.

[0011] It is also an object of the invention to provide the use of MoO_2Cl_2 as a precursor for producing a molybdenum-containing layer, which at least partially, and as fully as possible, overcomes the disadvantages of the prior art.

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[0012] It is also an object of the invention to provide an electronic component having a molybdenum-containing layer, which at least partially, and as fully as possible, overcomes the disadvantages of the prior art.

10 Disclosure of the invention

[0013] Surprisingly, the objects of the invention are achieved by methods, products and uses according to the patent claims.

15 [0014] The invention relates to a method for producing MoO_2Cl_2 under inert conditions, comprising the steps of:

- (i) charging a reaction vessel with MoO_2 ,
- (ii) reacting the MoO_2 with supplied Cl_2 in the reaction vessel at a first
- 20 temperature T_1 to give gaseous MoO_2Cl_2 ,
- (iii) transferring the gaseous MoO_2Cl_2 into a receiving vessel,
- (iv) resublimating the gaseous MoO_2Cl_2 in the receiving vessel to give solid MoO_2Cl_2 at a second temperature T_2 that is lower than T_1 , and
- (v) recovering solid MoO_2Cl_2 with a purity determined by ICP-OES/MS of
- 25 99.9996 wt.% or more.

[0015] The method according to the invention is carried out under inert conditions and is preferably carried out continuously under inert conditions. Except for the Cl_2 supplied in step (ii), the method is thus carried out excluding

30 other reactive gases or substances. Accordingly, the method is preferably carried out excluding oxygen and/or excluding air and/or excluding water. The

method is particularly preferably carried out in an atmosphere of pure Cl_2 or in an atmosphere of a $\text{Cl}_2\text{-N}_2$ mixture.

[0016] The method according to the invention is furthermore carried out with
5 MoO_2 as the only molybdenum-containing and/or the only oxidic starting material. In particular, no other molybdenum oxide, such as MoO or MoO_3 , is used in the method according to the invention. However, the MoO_2 charge may contain impurities, in particular metal impurities such as tungsten (W). It is also preferred according to the invention that, in addition to MoO_2Cl_2 , no other
10 molybdenum oxide chloride is produced in the method according to the invention, in particular no MoOCl_3 and/or no MoOCl_4 .

[0017] The purity of the MoO_2Cl_2 recovered in step (v) is determined by means of coupled ICP-OES/MS. An inductively coupled plasma (ICP) is used as the
15 excitation source. Firstly, the MoO_2Cl_2 is introduced into an inert gas flow (e.g. argon stream), to which high-frequency energy is then transmitted electrodelessly by inductive coupling. The frequencies used for this purpose are typically in the range of 25 and 100 MHz. The generators used typically have powers of between 1 and 10 kW. The inert gas flow is heated to
20 temperatures of typically up to 10,000 K. The MoO_2Cl_2 is atomized or ionized as a result. The resulting free atoms and/or ions are analyzed optically by means of atomic emission spectrometry (OES, *Optical Emission Spectrometry*) as well as by mass spectrometry (MS).

[0018] Elements in solutions can generally be detected and quantified by
25 means of optical emission spectrometry with inductively coupled plasma (ICP-OES; also ICP-AES, ICP-atomic spectrometry). The solution to be measured is vaporized as described above and the aerosol formed is introduced into a high-frequency plasma in which the solution components are atomized and
30 partially ionized. The characteristic emission lines of the atoms and ions are decomposed with a monochromator or polychromator and the line intensities are detected with a suitable detector. The measurement can be carried out

axially and/or radially. According to the invention, the measurement is preferably carried out axially, since the axial measurement is more sensitive. For quantitative determination, a calibration can be carried out with suitable standard solutions since there is a linear correlation between the signal intensities of the emission and the concentrations of an element. According to the invention, it is preferred that the measuring principle of the ICP-OES complies with DIN EN ISO 11885 (1997) / DEV E22 and/or the guidelines for chemical analysis, determination of elemental composition in environmental samples by ICP-OES, Fraunhofer-Institut für Molekularbiologie und Angewandte Oekologie [*Fraunhofer Institute for molecular biology and applied ecology*], Auf dem Aberg 1, D-57392 Schmallenberg, Germany.

[0019] Elements in solutions can generally be detected and quantified by means of mass spectrometry with inductively coupled plasma (ICP-MS). The solution to be measured is vaporized as described above and introduced into a high-frequency plasma in which the solution components are atomized and ionized. The ions are extracted from the high-frequency plasma by a pinhole aperture system and separated according to their mass-to-charge ratio. After separation, they are registered in a detector and the resulting signals are evaluated using the device software. A quantitative determination is possible after calibration with standard solutions, since there is a linear correlation between the signal intensities of the ions and the concentrations of an element. According to the invention, it is preferred that the measuring principle of the ICP-MS complies with DIN EN ISO 17294-2:2017-01 (Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) – Part 2: Determination of selected elements, including uranium isotopes (ISO 17294-2:2016); German version EN ISO 17294-2:2016) and/or the guidelines for chemical analysis, determination of elemental composition in environmental samples by ICP-MS, Fraunhofer-Institut für Molekularbiologie und Angewandte Oekologie [*Fraunhofer Institute for molecular biology and applied ecology*], Auf dem Aberg 1, D-57392 Schmallenberg, Germany.

[0020] Coupling of the two analysis methods OES and MS makes it possible to analyze a wide range of elements. For example, lighter elements are more difficult to determine with OES. However, the simultaneous use of MS allows them to be determined. Coupling of the analysis methods is therefore required.

5 The elemental proportions determined with the two analysis methods are consistent for the same elements, i.e., they provide consistent results within the scope of the usual measurement accuracies.

[0021] According to the invention, it is preferred that devices from Thermo

10 Fischer with the designations iCap, iCap Duo or iCap RQ are used for the coupled ICP-OES/MS.

[0022] Determination via ICP-OES/MS makes it possible to determine impurities in the recovered MoO_2Cl_2 , in particular metal impurities such as

15 tungsten, in the ppb range ("*parts per billion*"). The ppb values or ppb limits specified herein correspond to the respective detection limit of the impurities.

[0023] The method according to the invention makes it possible to produce particularly pure MoO_2Cl_2 , in particular makes it possible to produce MoO_2Cl_2

20 with a low tungsten content. According to the invention, it is preferred that the purity of the MoO_2Cl_2 recovered in step (v) is 99.99969 wt.% or more. Such pure MoO_2Cl_2 makes it possible to produce molybdenum-containing layers with further improved electronic properties.

25 [0024] According to the invention, it is further preferred that the solid MoO_2Cl_2 recovered in step (v) has a tungsten content, again determined by ICP-OES/MS, of ≤ 200 ppb and particularly preferably of ≤ 50 ppb. Tungsten as an impurity may particularly impair the electronic properties of molybdenum-containing layers. Due to the low tungsten contents according to the invention,

30 such impairments can be avoided and the electronic properties can be further improved.

[0025] In addition to the tungsten, the MoO_2Cl_2 may optionally also contain very small amounts of other impurities, in particular (semi)metal impurities, such as Ag, Al, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Fe, Ga, Ge, Hf, In, Ir, K, La, Li, Mg, Mn, Na, Nb, Ni, Pb, Pd, Pt, Rb, Rh, Ru, Sb, Se, Sn, Sr, Te, Th, Ti, Tl, U, V, W, Zn and Zr. According to the invention, it is preferred in this case that the MoO_2Cl_2 recovered in step (v) has, with respect to each individual one of these impurities considered independently of one another, a content of ≤ 300 ppb, more preferably of ≤ 200 ppb, even more preferably of ≤ 100 ppb, particularly preferably of ≤ 50 ppb and most preferably of ≤ 20 ppb. Thanks to the low contents according to the invention of such impurities in the MoO_2Cl_2 , the electronic properties of molybdenum-containing layers obtained therefrom can be further improved.

[0026] According to the invention, it is further preferred that in step (ii) the first temperature T_1 is $\geq 170^\circ\text{C}$, more preferably ≥ 170 to $\leq 600^\circ\text{C}$, even more preferably ≥ 200 to $\leq 500^\circ\text{C}$, particularly preferably ≥ 350 to $\leq 450^\circ\text{C}$ and most preferably 400°C . Such temperatures make it possible to achieve a compromise between increased reaction rates by elevated temperatures on the one hand and unnecessary energy consumption by excessively high temperatures on the other hand. In a preferred embodiment, it is also possible for the reaction vessel to be divided into different physical and/or theoretical reaction zones in which the temperature T_1 is set differently within the aforementioned temperature ranges.

[0027] In step (ii), use is preferably made of a reaction vessel, the temperature of which can be controlled or regulated. For example, use is made of a rotary kiln, a static kiln, or a downpipe kiln. According to the invention, a rotary kiln is preferred which has different reaction zones that can be heated (and cooled) differently. Such a kiln makes it possible to set one or more temperature gradients in the kiln, which can contribute to a further improvement in the purity of the MoO_2Cl_2 produced in step (ii).

[0028] According to the invention, it is further preferred that in step (iv) the second temperature T_2 is $\leq 160^\circ\text{C}$, more preferably ≥ 10 to $\leq 120^\circ\text{C}$, even more preferably ≥ 20 to $\leq 90^\circ\text{C}$, particularly preferably ≥ 25 to $\leq 70^\circ\text{C}$ and most preferably 70°C . Such temperatures make it possible to achieve a compromise
5 between high yields of MoO_2Cl_2 and low incorporation of impurities into the MoO_2Cl_2 during resublimation.

[0029] According to the invention, it is particularly preferred to vary the second temperature T_2 between the beginning and the end of the method according
10 to the invention. In this case, it is particularly preferred to lower the temperature T_2 in the receiving vessel from a higher temperature to a lower temperature, preferably from 90°C to 20°C , more preferably from 70°C to 25°C . Such temperature control makes it possible to set a particularly high purity of the MoO_2Cl_2 during the resublimation in step (iv).

15 [0030] In step (iv), use is preferably made of a receiving vessel, the temperature of which can be controlled or regulated. Use is for example made of a receiving flask. Alternatively, use can also be made of a cold trap on which gaseous MoO_2Cl_2 resublimates. The resublimated MoO_2Cl_2 can be removed
20 from the receiving flask in the usual way or can be thermally detached from the cold trap.

[0031] According to the invention, it is particularly preferred that the first temperature T_1 and the second temperature T_2 are set simultaneously as
25 specified above. Accordingly, the following temperature pairs or temperature controls are particularly preferred:

$T_1 \geq 170^\circ\text{C}$ and $T_2 \leq 160^\circ\text{C}$;
 $T_1 \geq 170$ to $\leq 600^\circ\text{C}$ and $T_2 \geq 10$ to $\leq 120^\circ\text{C}$;
30 $T_1 \geq 200$ to $\leq 500^\circ\text{C}$ and $T_2 \geq 20$ to $\leq 90^\circ\text{C}$;
 $T_1 \geq 350$ to $\leq 450^\circ\text{C}$ and $T_2 \geq 25$ to $\leq 70^\circ\text{C}$;
 $T_1 = 400^\circ\text{C}$ and $T_2 = 70^\circ\text{C}$;

$T_1 \geq 350$ to $\leq 450^\circ\text{C}$, and T_2 is 90°C at the start of the method, and is lowered to 20°C by the end of the method;

$T_1 = 400^\circ\text{C}$ and T_2 is 70°C at the start of the method and is lowered to 25°C by the end of the method.

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[0032] These temperature pairs or temperature controls make it possible to achieve a compromise between increased reaction rate, reduced energy consumption, high yield and low incorporation of impurities.

10 [0033] According to the invention, it is also preferred for step (iii) to be carried out at a temperature of $\geq 160^\circ\text{C}$. Such a temperature makes it possible to prevent premature resublimation of MoO_2Cl_2 and thereby to increase the yield in the receiving vessel.

15 [0034] According to the invention, it is moreover preferred that, in step (ii), the gas flow of the Cl_2 is 0.01 to 1 L/min, more preferably 0.05 to 0.8 L/min. Such a gas flow makes it possible to achieve a compromise between increased reaction rate and increased yield on the one hand, and unnecessary use of chlorine with a possibly subsequent problem of disposal of unused chlorine,
20 on the other hand.

[0035] According to the invention, it is moreover preferred that an inert gas, preferably nitrogen (N_2), is additionally supplied in step (ii). Such an additionally supplied inert gas, in particular N_2 , makes it possible to carry out
25 the method according to the invention even more safely under inert conditions. It is particularly preferred according to the invention that the gas flow of the inert gas, in particular of the N_2 , is 0.05 to 0.4 L/min, more preferably 0.1 to 0.3 L/min and particularly preferably 0.2 L/min, wherein the choice of the gas flow depends on the kiln size and configuration and should be correspondingly
30 adapted thereto. Such a gas flow of the inert gas, in particular of the N_2 , makes it possible to even better maintain the inert conditions without using unnecessarily large amounts of inert gas. In addition, such a gas flow can

enable improved transport of the gaseous MoO_2Cl_2 from the reaction vessel to the receiving vessel. In addition to N_2 , other inert gases can also be used, for example argon (Ar). The use of Ar can lead to a further improved purity of the MoO_2Cl_2 . According to the invention, the use of N_2 is most preferred due to its
5 lower costs.

[0036] According to the invention, it is also preferred that, in step (ii), the gas flow of the Cl_2 is 0.01 to 1 L/min and more preferably 0.05 to 0.8 L/min and at the same time N_2 is supplied with a gas flow of 0.05 to 0.4 L/min and more
10 preferably of 0.1 to 0.3 L/min (0.01 to 1 L/min of Cl_2 and 0.05 to 0.4 L/min of N_2 ; 0.05 to 0.8 L/min of Cl_2 and 0.05 to 0.4 L/min of N_2 ; 0.01 to 1 L/min of Cl_2 and 0.1 to 0.3 L/min of N_2 ; or 0.05 to 0.8 L/min of Cl_2 and 0.1 to 0.3 L/min of N_2). According to the invention, it is most preferred that, in step (ii), Cl_2 is supplied with a gas flow of 0.8 L/min and N_2 is supplied with a gas flow of 0.2
15 L/min. Such gas flows make it possible to achieve a yet further improved compromise between reaction rate, increased yield, unnecessary use of Cl_2 and associated disposal problems, and maintenance of the inert conditions without excessive use of inert gas and improved transport of the gaseous MoO_2Cl_2 .

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[0037] According to the invention, it is also preferred that the reaction vessel, the receiving vessel and optionally a transition region, optionally arranged between the reaction vessel and the receiving vessel, be inertized before step (i) by flushing with an inert gas, preferably nitrogen. As is generally customary,
25 "*inertization*" is understood to mean setting inert conditions in an apparatus. By flushing with an inert gas, the inert conditions of the method according to the invention can be set particularly quickly and efficiently. According to the invention, it is also preferred that the method according to the invention does not comprise a step of pressure reduction, in particular does not comprise a
30 step in which the pressure in the reaction vessel and/or in the receiving vessel is reduced.

[0038] According to the invention, it is also preferred that the MoO_2Cl_2 obtained in step (v) is further purified in a subsequent step (vi) by sublimation and resublimation. Alternatively, other purification methods, such as recrystallization, can also be used in a step (vi) in order to further purify the MoO_2Cl_2 obtained in step (v). This makes it possible to provide an MoO_2Cl_2 with yet further improved purity. Such an MoO_2Cl_2 can be converted into a particularly pure molybdenum-containing layer with improved electronic properties in a subsequent method, in particular in a method of vapor deposition. According to the invention, it is accordingly preferred that the method according to the invention does not have a step in which produced solid MoO_2Cl_2 is processed in a wet-chemical manner, in particular does not have a step of reacting produced solid MoO_2Cl_2 with aqueous ammonia. Such a reaction would typically result in the formation of hydrogen chloride (HCl). However, the formation of HCl is to be avoided according to the invention so that there are no problems with the disposal thereof and no risk of undesired corrosion of the apparatus used, including the reaction vessel and the receiving vessel, by the HCl.

[0039] The invention also relates to a method for producing a molybdenum-containing layer, which comprises a method according to the invention, wherein subsequently thereto, i.e. after step (v), or, if performed, after step (vi), the MoO_2Cl_2 obtained in step (v) or step (vi) is used in a vapor deposition as a precursor in order to form the molybdenum-containing layer. According to the invention, it is preferred that a chemical vapor deposition (CVD) or an atomic layer deposition (ALD) be used for the vapor deposition.

[0040] Chemical vapor deposition, or CVD, is a gas phase reaction that generally takes place at or near a surface of a substrate. Reactants involved in the reaction are fed simultaneously in the form of gases to the substrate to be coated. The substrate is arranged in a reaction chamber and is heated. The usually preheated gases are thermally activated by the heated substrate and react with each another. In the process, the desired material is deposited and

chemically bonded. Chemisorption of the desired material occurs, i.e. of the molybdenum in the present invention.

5 [0041] The ALD process, also referred to as atomic layer deposition, is a modified CVD process. With the ALD process, the reaction or sorption at the surface ceases by itself once the surface is completely covered. This self-limiting reaction is carried out in several cycles with flushing steps in between. This makes it possible to achieve very precise layer thicknesses.

10 [0042] The invention also relates to MoO_2Cl_2 obtainable by a method according to the invention. Such MoO_2Cl_2 is a high-purity MoO_2Cl_2 and is particularly suitable as a precursor for producing high-purity molybdenum-containing layers with improved electronic properties, in particular improved storage properties, for example when used in flash memories such as a 3D-NAND.
15 Such an MoO_2Cl_2 is preferably an MoO_2Cl_2 with a low water content, particularly preferably an anhydrous or dry MoO_2Cl_2 .

[0043] The invention also relates to MoO_2Cl_2 which has a purity determined by ICP-OES/MS of 0.9996 wt.% or more and more preferably of 0.99969 wt.% or
20 more. In this case, it is particularly preferred that the MoO_2Cl_2 has a tungsten content determined by ICP-OES/MS of ≤ 200 ppb, more preferably of ≤ 100 ppb and even more preferably of ≤ 50 ppb. Such an MoO_2Cl_2 is also a high-purity MoO_2Cl_2 and is particularly suitable as a precursor for producing high-purity molybdenum-containing layers with improved electronic properties, in
25 particular improved storage properties, for example when used in flash memories such as a 3D-NAND. Such an MoO_2Cl_2 is preferably an MoO_2Cl_2 with a low water content, particularly preferably an anhydrous or dry MoO_2Cl_2 .

[0044] The invention also relates to the use of the MoO_2Cl_2 obtainable or
30 provided according to the invention as a precursor for producing a molybdenum-containing layer. In particular, the MoO_2Cl_2 can serve to produce

a molybdenum-containing layer by means of gas-phase thin-film methods such as the CVD or ALD described above.

5 [0045] The invention also relates to an electronic component comprising a molybdenum-containing layer produced according to the invention. Due to the improved purity of the MoO_2Cl_2 provided according to the invention, the molybdenum-containing layer is likewise particularly pure and is in particular virtually to completely free of tungsten impurities. The electronic component can thus be provided with improved electronic properties.

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[0046] According to the invention, it is preferred that the electronic component is what is referred to as a 3D-NAND. A 3D-NAND is a non-volatile memory ("*flash memory*") in which a plurality of memory cells are stacked vertically in a plurality of layers. In this case, the memory cells are connected in series. For
15 example, 16, 32, 64, 96, or 128 memory cells can be connected in order to achieve an increased storage density. By using a molybdenum-containing layer produced according to the invention in such a non-volatile memory, the electronic properties thereof can be improved, in particular the storage properties thereof can be improved.

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Exemplary embodiment

[0047] A rotary kiln having a pipe length of 1 m and a pipe diameter of 5 cm is used as the reaction vessel. The rotary kiln is connected via a transition region
25 to a product flask as receiving vessel. The rotary kiln, the transition region and the product flask can be heated separately. The rotary kiln is charged with 400 g of MoO_2 . Subsequently, an inert gas atmosphere is created throughout the entire apparatus by introducing nitrogen (0.4 L/min of N_2). The rotary kiln is then brought to a temperature (T_1) of 400°C, the transition region is brought to
30 a temperature of 160°C, and the product flask is brought to a temperature (T_2) of 70°C. After these temperatures have been reached, an N_2 flow of 0.2 L/min is set. Thereafter, Cl_2 is additionally introduced into the rotary kiln (0.8 L/min of

Cl₂). The MoO₂ charge is allowed to react with the introduced Cl₂ for two hours (2 h). Thereafter, the introduction of Cl₂ and the heating of rotary kiln and transition region are terminated, and the product flask is allowed to cool to 25°C. The N₂ flow is increased to 1 L/min and continued for 30 min. The introduction of N₂ is then terminated, and the product flask is removed from the apparatus in Ar countercurrent. The product flask is fed into a glove box with an inert gas atmosphere and unpacked there.

[0048] Furthermore, a sample for determining the content of impurities is taken from the product flask. The determination in turn takes place by means of ICP-OES/MS. For this purpose, devices from Thermo Fischer with the designations iCap, iCap Duo or iCap RQ are used. The impurities indicated in Table 1 below were determined (the indicated ppb values corresponding to the respective detection limit):

Table 1

Element	Content in ppb	Element	Content in ppb
Ag	< 50	Mn	< 20
Al	< 50	Na	< 150
Au	< 50	Nb	< 100
B	< 50	Ni	< 50
Ba	< 50	Pb	< 20
Be	< 20	Pd	< 50
Bi	< 20	Pt	< 50
Ca	100	Rb	< 50
Cd	< 150	Rh	< 50
Ce	< 50	Ru	< 50
Co	< 50	Sb	< 20
Cr	< 50	Se	< 50
Cu	< 50	Sn	< 100
Fe	< 50	Sr	< 50
Ga	< 50	Te	< 100
Ge	< 20	Th	< 20
Hf	< 20	Ti	< 200
In	< 50	Tl	300
Ir	< 50	U	< 20
K	< 200	V	< 50

La	< 20		W	< 200
Li	< 20		Zn	< 50
Mg	< 50		Zr	< 50

[0049] The sum of the impurities is thus in total < 3070 ppb. The MoO_2Cl_2 produced accordingly has a purity of at least 99.9996930 wt.%.

Claims

1. A method for producing MoO_2Cl_2 under inert conditions, comprising the steps of:
 - 5 (i) charging a reaction vessel with MoO_2 ,
 - (ii) reacting the MoO_2 with supplied Cl_2 in the reaction vessel at a first temperature T_1 to give gaseous MoO_2Cl_2 ,
 - (iii) transferring the gaseous MoO_2Cl_2 into a receiving vessel,
 - 10 (iv) resublimating the gaseous MoO_2Cl_2 in the receiving vessel to give solid MoO_2Cl_2 at a second temperature T_2 that is lower than T_1 , and
 - (v) recovering solid MoO_2Cl_2 with a purity determined by ICP-OES/MS of 99.9996 wt.% or more.
2. The method according to claim 1, wherein, in step (v), solid MoO_2Cl_2 with a purity determined by ICP-OES/MS of 99.99969 wt.% or more is recovered.
3. The method according to at least one of the preceding claims, wherein, in step (v), solid MoO_2Cl_2 with a tungsten content determined by ICP-OES/MS of ≤ 200 ppb, preferably ≤ 50 ppb, is recovered.
4. The method according to at least one of the preceding claims, wherein the first temperature T_1 is $\geq 170^\circ\text{C}$, preferably 170 to 600°C , and/or the second temperature T_2 is $\leq 160^\circ\text{C}$, preferably 20 to 90°C .
5. The method according to at least one of the preceding claims, wherein step (iii) is carried out at a temperature of $\geq 160^\circ\text{C}$.
6. The method according to at least one of the preceding claims, wherein, in step (ii), the gas flow of the Cl_2 is 0.01 to 1 L/min, preferably 0.05 to 0.8 L/min.

7. The method according to at least one of the preceding claims, wherein an inert gas, preferably N₂, is additionally supplied in step (ii).
8. The method according to claim 7, wherein the gas flow of the inert gas is 0.05 to 0.4 L/min, preferably 0.1 to 0.3 L/min.
9. The method according to at least one of the preceding claims, wherein, prior to step (i), the reaction vessel and the receiving vessel are inertized by flushing with an inert gas, preferably N₂.
10. The method according to at least one of the preceding claims, wherein the MoO₂Cl₂ obtained in step (v) is further purified in a subsequent step (vi) by sublimation and resublimation.
11. A method for producing a molybdenum-containing layer, comprising a method according to at least one of the preceding claims, wherein subsequently the MoO₂Cl₂ obtained in step (v) or step (vi) is used in a vapor deposition as a precursor in order to form the molybdenum-containing layer.
12. MoO₂Cl₂ obtainable by a method according to at least one of claims 1 to 10.
13. MoO₂Cl₂ which has a purity determined by ICP-OES/MS of 0.9996 wt.% or more and preferably a tungsten content determined by ICP-OES/MS of ≤ 200 ppb.
14. The use of MoO₂Cl₂ according to claim 12 or 13 as a precursor for producing a molybdenum-containing layer.
15. An electronic component, in particular a 3D NAND, comprising a molybdenum-containing layer produced according to claim 11 or 14.