Object of the present invention is to provide a process for the manufacture of purified fluorine. Another object of the present invention is to provide a process for the manufacture of purified fluorine which is especially suitable for the application in the semiconductor industry.

These objects and other objects as apparent from the description and the claims are achieved by the invention.

Consequently, in one aspect, the process of the present invention concerns a process for the manufacture of purified fluorine comprising a purification treatment wherein fluorine which contains solid impurities is subjected to a solid-removing treatment wherein the solid-removing treatment comprises at least one step of contacting the fluorine with liquid hydrogen fluoride, and a subsequent purification treatment comprising at least one step of removing hydrogen fluoride from the fluorine after contact with liquid hydrogen fluoride. Additionally, the fluorine may be subjected to at least one step of contacting it with an adsorbent (e.g. a solid sorbent) for HF, and it may additionally subjected to at least one step of passing it through a particle filter. The fluorine may be passed through a particle filter before and/or after the solid removing treatment and the purification treatment mentioned above. In the frame of the present disclosure~~invention~~, the term "solids" and particles" are interchangeable.

In another aspect, there is provided a method for the manufacture of a semiconductor, a micro-electromechanical system, TFT (flat panel display) or a solar cell comprising a step of etching an item with fluorine purified as defined herein.

In yet another aspect, there is provided a method for the manufacture of a semiconductor, a micro-electromechanical system, TFT (flat panel display) or a solar cell, said method comprising a step of depositing layers on items in a deposition chamber, wherein deposits form at least on a part of the interior of the deposition chamber, and further comprising contacting said deposition chamber with fluorine purified as defined herein to remove at least a part of the deposits in the interior of the deposition chamber.

The F_2 can be contacted with technical grade HF. If desired, purified HF can be applied.

Methods to purify hydrogen fluoride from phosphorous compounds, sulfur compounds, arsenic compounds, metals, hydrocarbons and water are well known. See for example US patent 5,362,469 and US patent 5,585,085. According to US patent 5,362,469, water, arsenic compounds, boron compounds, phosphorous compounds and sulfur compounds as well as carbon compounds and metal compounds are removed from HF by contacting liquefied hydrogen fluoride with lithium fluoride and elemental fluorine. The hydrogen fluoride is then distilled to provide pure hydrogen fluoride. According to US patent 5,585,085, water and hydrocarbons are removed from hydrogen fluoride by contacting it with elemental fluorine and subsequent distillation.

The fluorine may preferably be contacted with a purified hydrogen fluoride which is essentially free of at least phosphorous compounds, sulfur compounds and arsenic compounds. Hydrogen fluoride can be purified according to the process of US patent 5,362,469 and then has a content of water of less than 1 ppm.

CLAIMS

1. A method for the manufacture of a semiconductor, a micro-electromechanical system, TFT (flat panel display) or a solar cell comprising a step of etching an item with fluorine, or a method for the manufacture of a semiconductor, a micro-electromechanical system, a TFT (flat panel display) or a solar cell comprising a step of depositing layers on items in a deposition chamber wherein deposits form at least on a part of the interior of the deposition chamber, and contacting said deposition chamber, with fluorine purified according to a process wherein fluorine which contains solid impurities is subjected to a solid-removing treatment wherein the solid-removing treatment comprises at least one step of contacting the fluorine with liquid hydrogen fluoride, and wherein the fluorine is subsequently subjected to a purification treatment comprising at least one step of removing hydrogen fluoride from the fluorine after contact with liquid hydrogen fluoride, and wherein the fluorine is subjected to a step of contacting it with sodium fluoride, and wherein the fluorine is subjected to a step of passing it through a particle filter for the removal of entrained solids.

2. The process of claim 1 wherein the fluorine is contacted with liquid hydrogen fluoride in a jet gas scrubber.

3. The process of claim 1 wherein the fluorine is contacted with liquid HF by bubbling it through liquid HF in a vessel.

4. The process of anyone of claims 1 to 3 wherein the at least one step of removing hydrogen fluoride is a low temperature treatment.

5. The process of anyone of claims 1 to 4 wherein the purification treatment comprises: at least one step of a low temperature treatment, at least one subsequent step of contacting the fluorine after the low temperature treatment with an adsorbent for HF, and, after the contact of the fluorine with the adsorbent for HF, at least one subsequent step of passing the fluorine through a particle filter to provide highly pure fluorine.

6. The process of claims 4 or 5 wherein the particle filter to provide highly pure fluorine comprises pores having a diameter less than 1 μm.

7. The process of claim 6 wherein the particle filter comprises pores having a diameter less than 10 nm.

8. The process of any one of claims 1 to 7 wherein continuously or intermittently, at least a part of the liquid HF is withdrawn from the solid removing treatment, and wherein withdrawn HF is introduced into an electrolytic cell or into electrolytic cells for F_2 production.

9. The process of any one of claims 1 to 8 wherein the fluorine is electrolytically produced in one or more electrolytic cells from an electrolyte containing KF dissolved in HF.

10. The process of claim 9 wherein the fluorine electrolytically produced is passed through a particle filter and is subsequently subjected to the solid removing treatment and the purification treatment.

11. The process of claims 9 or 10 wherein the fluorine electrolytically produced is passed through a particle filter and subsequently subjected to the solid removing treatment comprising at least one step of contacting the fluorine with liquid hydrogen fluoride in a jet gas scrubber or by bubbling the fluorine through liquid HF in a vessel, subjecting the fluorine after the treatment with liquid HF to a low temperature treatment, subsequently contacting the fluorine with NaF as adsorbent for HF, and subsequently passing the fluorine through at least one particle filter having pores with a diameter of less than 1 μm.

12. The method of claim 4 wherein the pressure is equal to or greater than 1 and equal to or lower than 20 bar (abs), preferably equal to or lower than 12 bar (abs).

CLAIMS

1. A process for the manufacture of purified fluorine, comprising subjecting fluorine which contains solid impurities to a solid-removing treatment wherein the solid-removing treatment comprises at least one step of contacting the fluorine with liquid hydrogen fluoride, subsequently subjecting the fluorine to a purification treatment comprising at least one step of removing hydrogen fluoride from the fluorine after contact with said liquid hydrogen fluoride, optionally subjecting the fluorine to a step of contacting said fluorine with an adsorbent for HF; and optionally, subjecting the fluorine to a step of passing said fluorine through a particle filter for the removal of entrained solids.

2. The process of claim 1 wherein, in the solid-removing treatment, the fluorine is contacted with liquid hydrogen fluoride in a jet gas scrubber.

3. The process of claim 1 wherein, in the solid-removing treatment, the fluorine is contacted with liquid HF by bubbling it through liquid HF in a vessel.

4. The process of claim 1, wherein the at least one step of removing hydrogen fluoride is a low temperature treatment.

5. The process of claim 1, comprising at least one step of comprising at least one step of contacting the fluorine, after the solid-removing treatment with liquid HF, with an adsorbent for HF, and subsequently at least one step of passing the fluorine through a particle filter for the removal of entrained solids.

6. The process of claim 1, wherein the purification treatment comprises: at least one step of a low temperature treatment, at least one subsequent step of contacting the fluorine after the low temperature treatment with an adsorbent for HF, and, after the contact of the fluorine with the adsorbent for HF, at least one subsequent step of passing the fluorine through a particle filter to provide highly pure fluorine.

7. The process of claim 5, wherein the particle filter to provide highly pure fluorine comprises pores having a diameter less than 1 μm.

8. The process of claim 7 wherein the particle filter comprises pores having a diameter less than 10 nm.

9. The process of claim 5, wherein the adsorbent for HF is sodium fluoride.

10. The process of claim 1, wherein continuously or intermittently, at least a part of the liquid HF is withdrawn from the solid-removing treatment, and wherein withdrawn HF is introduced into an electrolytic cell or into electrolytic cells for F_2 production.

11. The process of claim 1, wherein the fluorine is electrolytically produced in one or more electrolytic cells from an electrolyte containing KF dissolved in HF.

12. The process of claim 11, wherein the fluorine electrolytically produced is passed through a particle filter and is subsequently subjected to the solid removing treatment and the purification treatment.

13. The process of claim 11, wherein the fluorine electrolytically produced is passed through a particle filter and subsequently subjected to the solid-removing treatment comprising at least one step of contacting the fluorine with liquid hydrogen fluoride in a jet gas scrubber or by bubbling the fluorine through liquid HF in a vessel, and wherein the process further comprises subjecting the fluorine after the treatment with liquid HF to a low temperature treatment, subsequently contacting the fluorine with NaF as adsorbent for HF, and subsequently passing the fluorine through at least one particle filter having pores with a diameter of less than 1 μm.

14. A method for the manufacture of a semiconductor, a micro-electromechanical system, TFT (flat panel display) or a solar cell comprising a step of etching an item with fluorine purified according to the method of claim 5.

15. The process of claim 4, wherein the pressure is equal to or greater than 1 and equal to or lower than 20 bar (abs).

16. The process of claim 5, wherein continuously or intermittently, at least a part of the liquid HF is withdrawn from the solid-removing treatment, and wherein withdrawn HF is introduced into an electrolytic cell or into electrolytic cells for F_2 production.

17. A method for the manufacture of a semiconductor, a micro-electromechanical system, TFT (flat panel display) or a solar cell, said method comprising a step of depositing layers on items in a deposition chamber, wherein deposits form at least on a part of the interior of the deposition chamber, and further comprising contacting said deposition chamber with fluorine purified according to the process of claim 5 to remove at least a part of the deposits in the interior of the deposition chamber.