

# Operator's Manual

# **APS***pro*

Advanced Plasma Sources System for Ion-Enhanced Coating Processes

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#### Manufacturer and publisher

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English Translation of German original

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Bühler Alzenau

# 1 Application and Data

### 1.1 Product information

# 1.1.1 Information about this Operator's Manual

The Operator's Manual for the «Plasma Source Technology Advanced Plasma Source» (hereinafter referred to as «APSpro») contains extensive information about the construction, function, operation, maintenance and service of the APSpro and its system components. This Operator's Manual contains the information required in order to use the APSpro properly.

- Always keep this operating instructions available for reference.
- Bühler Alzenau would like to point out that the product illustrations and photos used in this
  user manual may differ from the actual machine configuration

#### **Product life phases**

The Operator's Manual describes all the product life phases of the APS*pro*. This consists of all the phases after manufacturing: transport, installation, initial start-up, operation, maintenance, service, storage and disposal.

#### User qualifications and safety

This Operator's Manual is directed exclusively to technically qualified personnel, who have been trained by Bühler Alzenau or have completed an instruction course for the APS*pro*. This instruction course must have been carried out under the authorization of Bühler Alzenau. Only technically qualified personnel are capable of correctly interpreting the safety regulations contained in this Operator's Manual and applying them in practice.

#### NOTE!

Persons who have not been trained on the APSpro by Bühler Alzenau or other authorized persons not permitted to work on the APSpro. Bühler Alzenau will not be held responsible for any claims resulting from failure to observe these conditions.

It is imperative that you read Chapter 2 Safety, 
21 before working with the APSpro! It contains important information concerning your personal safety. This chapter must be read and fully understood by all persons working on the system at any time during the product service life of the APSpro.

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# 1.1.2 Explanation of the safety symbols and danger levels

The safety precautions used in the Operator's Manual indicate different levels of danger.

Colour and signal word	Meaning
<b>▲</b> DANGER	High risk. Failure to follow these instructions will lead to serious physical injuries or death.
	Moderate risk. Failure to follow these instructions can lead to serious physical injuries.
ACAUTION-	Low risk. Failure to follow these instructions can lead to minor physical injuries.
NOTICE	Low risk. Failure to follow these instructions can lead to minor physical injuries, material damage or faulty products.



This draws attention to information contained in another document section.

#### NOTE!

Represents technical requirements which must receive special attention.

# 1.1.3 Standards and guidelines

#### Technical documentation

The APSpro Operator's Manual was written in compliance with the following standards and guidelines:

- EU Machine Guideline 2006/42/EG
- DIN/EN 12100
- DIN/EN 82079
- VDI 4500 (Page 1)

# 1.2 Product description

Plasma technology is predominately used for the ion-enhanced vapor deposition (coating process) of surface-substrates to manufacture thin layers. The plasma source (A) supports the vapor deposition process of electron beam evaporators (B) or thermal evaporators by bombarding (C) a substrate with highly energized ions. The use of plasma technology increases and sustains the quality of coated surfaces with regard to adhesion, hardness and density of the deposited layers.

The plasma source system APSpro was designed by Bühler Alzenau especially for the plasma-enhanced coating process and is optionally available as a single component within the coating installation.

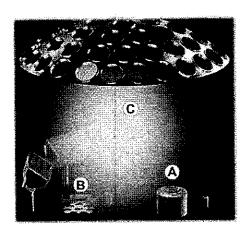


Fig. 1-1 Plasma-enhanced coating process

- A Plasma source
- B Evaporation source
- C Plasma cloud

The entire APSpro plasma source system is a combination of various mechanical and electrical system components (Fig. 1-2, 🖺 9) with the following main assemblies:

- Plasma source
- Plasma current power supply, GX 120/200
- Heater current power transformer, pe 4606
- Coil current power supply unit
- Gas, cooling water and compressed air supply
- Control, integration of the APS functions into the system's operating software

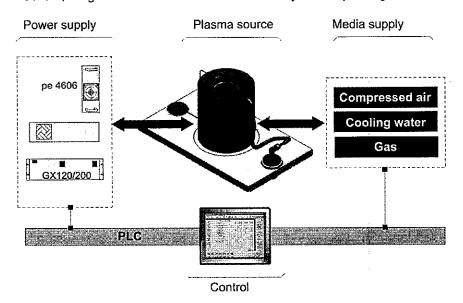


Fig. 1-2 Overview of the plasma source system components

#### Location of the system components within the installation

The APSpro plasma source system is designed for use in vacuum installations. The illustration Fig. 1-3, 

10 shows where the system components APSpro are installed in a SYRUSpro system.

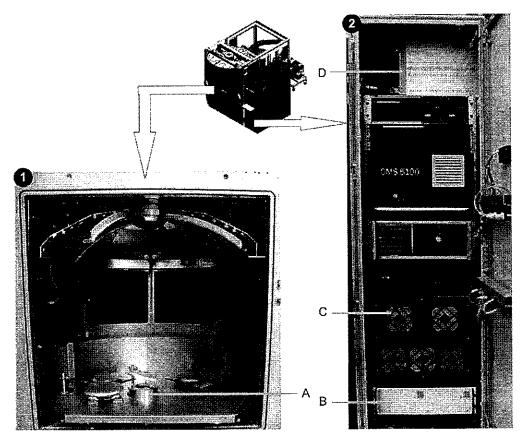


Fig. 1-3 Location of the APSpro system components (SyrusPro shown here as an example)

- 1 Process chamber
- A Plasma source

- 2 Electrical cabinet
- B Plasma current power supply GX 120/200
- C Coil current power supply
- D Heater current power supply pe 4606

#### 1.2.1 Functioning principle

As illustrated in the block diagram Fig. 1-4, 🖹 11, the plasma required for ion-enhanced vapor deposition is created from a plasma source located within the vacuum chamber. An ionization process is initiated within the plasma source through the electrical connections (DPS 500, NDDR, TSQ 400) using a heater cathode unit and by supplying argon gas. This process then ignites the energized plasma. The brightly illuminated plasma is emitted in a cone shape through the plasma opening where it then strikes the substrate as highly energized electrons and ions.

The APS III system is integrated into a master system controller. It allows the entire data communication (set point input and actual value display) between the individual system parts to be controlled on the screen. The anode current, coil current, heating current, the gas flow and the cooling water supply are regulated in a closed control cycle so that a plasma beam can be generated with the desired energy density.

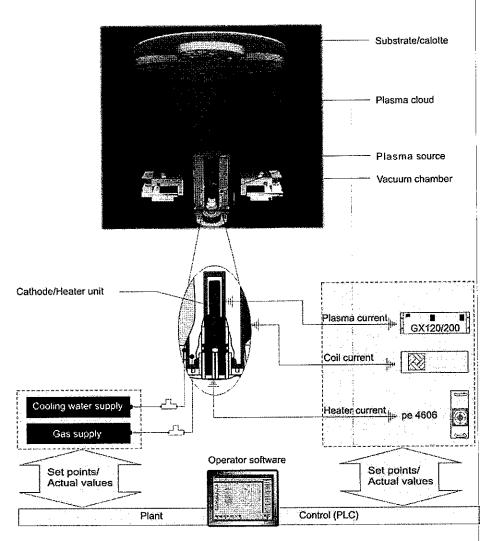


Fig. 1-4

Block diagram of the APSpro system functions

For a detailed description of the how the APS control functions, please see: Chapter 5.2.2 Operating phases and control sequence, 

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#### 1.2.2 Uses and features of the APSpro

The APSpro is used for the plasma-enhanced chemical vapor deposition of thin layers onto a substrate. By using the APSpro, the quality (property, structure) of the coating is consistently improved. In particular, the plasma-enhanced coating procedure with the APSpro offers advantages in reference to the following coating properties:

- Higher density, adhesion, hardness and scratch resistance of the layer
- Lower operating temperatures and thus ideal conditions for coating heat-sensitive substrates
- High deposition rates and thus shorter process times
- Temperature-resistant coatings over large surfaces (up to 1 m )
- Flat boundary layers and amorphous layer structures that lead to very low dispersion or absorption losses

A plasma-enhanced coating process with an APSpro is primarily used to produce the following coated products:

- Anti-reflective coatings
- Narrow-band filters
- Broadband filters
- Cut-off filters
- Dielectric mirrors
- Transparent, electrically conducting layers
- · Abrasion-proof, transparent layers
- Gain flattening filters
- Rugate-similar filters

# 1.2.2.1 Fields of application

In addition to improving the layer properties, the APSpro plasma source system is also used to clean substrate surfaces (pre-treatment) or manufacture oxide layers. Typical fields of application are:

#### Substrate pre-treatment (etching)

Substrate pre-treatment by means of ion bombardment is a method for cleaning and activating substrate surfaces (also known as etching). The substrate is bombarded with the gas ions created in the plasma source. This cleans and conditions the substrate surface before it is actually coated. Microscopic particles on the surface of the substrate are removed. The removed particles are pumped away together with the working gas. Argon is often used as a working gas.

#### Oxide coating (reactive coating)

When coating with oxides, a reduction in the coating material often occurs during the process. The resulting loss in the quality of the deposited layers can be counteracted by supplying additional oxygen. However, the additional oxygen can only support the coating process when it is present in a disassociated form. The oxygen molecules can be disassociated very easily and stored in the layer being formed with the aid of the plasma source. This is done by adding oxygen to the plasma from a connected gas shower. The ions emitted from the combustion

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The APSpro is used to create oxide layers for a wide range of coating materials. Typical examples are:

Material	Properties	
SiO <sub>2</sub>	Low refraction index, low absorption in the ultraviolet, visible and infrared spectral range	
TiO <sub>2</sub>	High refraction index, very low absorption in the visible and infrared spectral range	
Ta <sub>2</sub> O <sub>5</sub>	High refraction index, very low adsorption in the visible spectral range and transmission up to a wavelength of 400 nm	
HfO <sub>2</sub>	High refraction index, suitable in particular for UV filters	
ITO	Transparent, electrically conducting oxide	

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# 1.3 Using the system

#### 1.3.1 Proper use

Proper use and operation means:

- The APSpro plasma source system is intended exclusively for use in vacuum installations for the plasma-enhanced surface coating of substrates. The equipment components of the APSpro are fixed system parts that are exclusively designed for creating and using plasma.
- - Plasma source
  - Coil current supply unit
  - Plasma current power supply
  - Heater current power supply

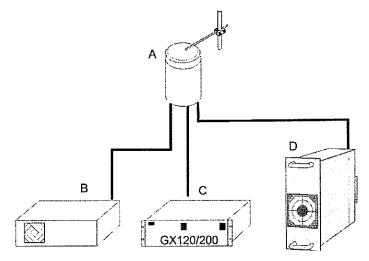


Fig. 1-5 Equipment components of the APSpro

- A Plasma source
- B Coil current power supply unit
- C GX 120/200 plasma current power supply
- D PE 4606 heater current power supply

Any application exceeding the bounds of these specifications is considered improper use, and can lead to serious personal injury or material damage. Bühler Alzenau disclaims responsibility for any consequential damages.

To ensure proper use, it is also required that:

- this Operator's Manual is read and observed.
- the technical data is observed. See Chapter 1.4 Technical data, 

  17.
- maintenance work is completed on schedule. See Chapter 6 Maintenance and Service,
   57.
- the safety precautions described in Chapter 2 Safety, 

  21 are observed for all processes carried out with the APSpro.

# 1.3.2 Unauthorized and improper use

All other uses of the APS*pro* are prohibited without the written approval from Bühler Alzenau GmbH (Bühler Alzenau). Any application that does not meet the above specifications is considered improper use. Bühler Alzenau disclaims responsibility for any personal injury or material damage resulting from improper use.

#### Dangers

The APS*pro* employs state-of-the-art technology and was built in line with the recognized safety regulations. It has undergone a comprehensive safety test and approval process. However, it is not possible to completely rule out all possible dangers when using the system. These are dangers pertaining to

- The life and well-being of the user
- The APSpro and other material possessions of the operator

and detrimental effects which could interfere with the ability to operate the APSpro efficiently.

For this reason, all activities involving the APS*pro* must be carried out by trained personnel in accordance with the guidelines in this Operator's Manual. The technical specifications must be strictly observed at all times.

#### NOTE!

The APS*pro* can be potentially dangerous if the safety precautions are not observed. Therefore, we strongly recommend reading Chapter 2 Safety, № 21.

# 1.3.3 Sealing the system and warranty terms

The plasma source APSpro is protected against unauthorized use of the assembly parts by a lead seal.

#### NOTE!

The four fastening screws are secured against unauthorized opening by means of a lead-sealed wire. Only Bühler Alzenau service personnel are permitted to open this lead seal.

All warranty claims will be voided if the lead seal or the wire are opened by unauthorized persons.

#### Termination of the warranty claims

The internal components of the plasma source may only be serviced by Bühler Alzenau service personnel.

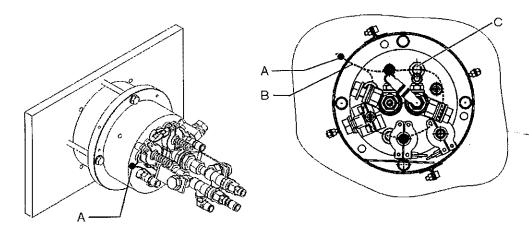


Fig. 1-6

Wire seal of the plasma source.

- A Lead seal
- B Lead-seal wire
- C Lead-sealed fastening screws

# 1.4 Technical data

# 1.4.1 Plasma source

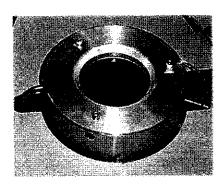


Fig. 1-7 APSpro plasma source

Dimensions and weight		
Weight	20 kg	
Overall height	approx. 640 mm	
External diameter	190 mm	

Operating characteristics	
Average cooling water flow rate	8 l/min
Discharge power in normal operating mode	12 kW (max. 15 kW)
Discharge current	60 A (max. 100 A)
Discharge voltage	up to 200 V
BiAS voltage	55 - 200 V
lon density with argon	1300 μA/ cm <sup>2</sup>
Ion energy	20 eV - 250 eV
Heater:	
Operating temperature	max. 1500 °C
Heating capacity	1.8 kW
Cathode:	
Heater	indirect
Material	lanthanum hexaboride (LaB6)

#### 1.4.1.1 Device identification

The plasma source is identified by a unique ID located on a type plate. The type plate is attached to the flange of the APS*pro* plasma source and lists the following information:

- APS source type
- Anode tube
- Heater consumption

When contacting Bühler Alzenau, be sure to provide all the information on the type plate. This information will enable the Bühler Alzenau customer service department to assist you as quickly as possible.

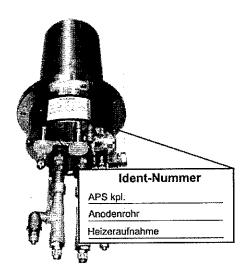


Fig. 1-8 Type plate APSpro

# 1.4.2 APS Energy supply

The APS is supplied with energy by the following three devices. They are installed in the electrical cabinet:

- Plasma current power supply GX 120/200 for generating the plasma (pos. A)
- Power supply for coil current (pos. B)
- Heater current power supply pe 4606 for cathode heating (pos. C)

The three devices are accessory equipment (purchased items), that have not been produced by Bühler Alzenau. Any technical data provided here comes from the suppliers' documentation

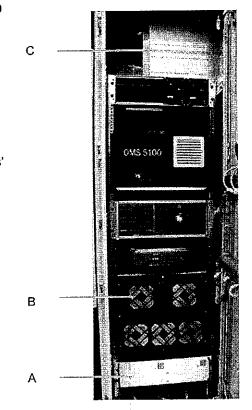


Fig. 1-9 Energy supply in electrical cabinet



For further information refer to the corresponding suppliers' documentation.

# Plasma current power supply «GX 120/200»

Electrical connection data and c	dimensions
Weight	23 kg
Nominal power	12 kW
Output voltage	200 V
Output current	60 A
Setpoint settings	from 0-100% for nominal power, voltage and current
Cooling	water-cooled

#### Coil current power supply

Electrical connection data and dimensions		
Weight	8.5 Kg	
Power supply	170265 VAC, 4763 Hz	
Rated voltage	0120 VDC	
Rated current	07 ADC	
Degree of protection	IP 20	
Operating temperature	5°C - 50°C	

#### Heater current power supply «pe 4606 250A/12V»

Electrical connection data and dimensions	
DC current	0 250A, infinitely adjustable
DC voltage	0 12V, infinitely adjustable
Weight	app 12 kg
Cooling	Air-cooled via fans

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# Safety

All persons who work with the APSpro must be technically qualified and properly trained. They must be familiar with all the dangers and risks associated with this device.

The system must only be used for its originally intended purpose. See Chapter 1.3.1 Proper use, 14. Any work instruction or working method which could infringe on the safety of the personnel or the APSpro is prohibited. The accident prevention guidelines specific to the industry and all local regulations must be observed at all times.

# 2.1 Read this!

# NOTE!

We strongly recommend reading this chapter before carrying out any work with the APSpro as it contains important information concerning personal safety!

This chapter must be read completely and fully understood by all persons who work with the APSpro and the system components during

- Maintenance and service
- Storage and disposal.

The safety precautions, instructions and warnings in Chapter 2 Safety, 21 must be strictly observed by all of these persons. Operator's responsibility

It is the responsibility of the operator to ensure that

- Persons involved in the installation, operation and maintenance of the APSpro have
- Persons who work with the APSpro have been duly informed about the potential dangers
- The device is only operated when it is in proper working order. All maintenance and service

# 2.2 General safety precautions for the operator

#### Personnel

All persons working with the APS*pro* must be technically qualified and properly trained. They must be familiar with all the dangers and risks associated with this device.

#### Operation

The system must only be used for its originally intended purpose. See Chapter 1.3.1 Proper use, 11. Any work instruction or working method which could infringe on the safety of the personnel or the APS*pro* is prohibited. The accident prevention guidelines specific to the industry and all local regulations must be observed at all times.

#### Transport, installation and initial start-up

The APS*pro* may only be installed and put into operation by authorized and technically qualified personnel. The APS*pro* components should be shipped in the original packaging whenever possible.

#### Maintenance and service

The operator is obliged to operate the APS*pro* only in a proper working condition. All maintenance and service work must be carried out in accordance with Chapter 6 Maintenance and Service, § 57.

# 2.3 Safety and protection locks

To ensure the safety of the operating personnel and reduce the possibility of damage to the device, the APS*pro* must be adequately integrated into the safety circuit of the vapor deposition system. The safety and protection locks can be divided into two categories:

- Hardware locks
- Software locks

#### 2.3.1 Hardware locks

Hardware locks are protection mechanisms in the form of limit switches and button functions with direct contact activation. Their purpose is the protection and active safety of the operating personnel.

When one of the locking components is triggered, the security system is interrupted. All system functions or system component functions within a locking system are switched off and locked to prevent them from restarting.

#### NOTE!

Only switch on and operate the APS*pro* when all locking components of the vapor deposition system are in proper function order. The operator is obligated to check the functioning of the hardware locking components, i.e.

- EMERGENCY-OFF switch
- Door limit switch

at regular intervals and especially when any new components are added to the system.



Using the «Operating Manual», please take time to familiarize yourself with the type, position and function of the hardware locks present on the vapor deposition system.

#### 2.3.2 Software locks

Software locks are protection mechanisms in the form of PLC safety circuits, limit switches and error monitoring programs. They protect the system against damage and incorrect operation, as well as the safety of the operating personnel.

#### Locking situations

In reference to the APS*pro*, the vapor deposition system is equipped with locks for the following situations:

- The coil current of the APSpro can only be switched on when the water cooling is switched on.
- The cathode heating of the APSpro can be switched on if
  - The chamber pressure is less than 10<sup>-4</sup> mbar
  - The water cooling is switched on.
- The plasma current power supply of the APSpro can only be switched on when the heating is on

All software locks are displayed as warning and error messages on the installation's visualization device.



For more information on error messages and troubleshooting, see the respective system documentation under Operating Manual.

#### Flood locking system

The function of the flood locking system is to provide the components an added level of protection. The plasma current power supply must be disconnected from the mains before the chamber is flooded.

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# 2.4 Plasma source protective cover

The electrical connections and high-voltage conducting parts of the plasma source are guarded against direct manipulation by means of a protective cover, see Fig. 2-1, 

24 /Pos. A. The plasma source must never be operated without the protective cover properly mounted.

#### NOTE!

The protective cover (casing parts) may only be removed for maintenance and repair work. Note the following:

- The protective cover may only by removed by authorized and qualified personnel.
- The protective cover may only be removed after it has been determined that the plasma source has been disconnected from the power supply. The works steps listed under Chapter 2.5 Disconnecting the energy supply, 

  25 must be strictly observed.
- The safety precautions listed under Chapter 2.6.1 Danger areas for operating personnel, 

  26 must be strictly observed.

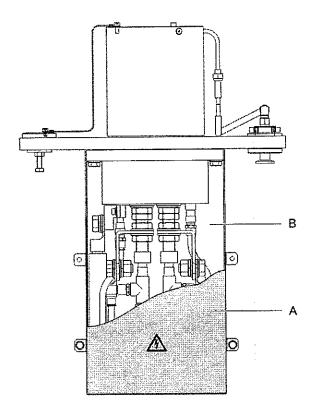


Fig. 2-1 Plasma source with electronic protective cover

- A Removable protective cover (casing parts)
- B Installed supply of media and electrical connections

# 2.5 Disconnecting the energy supply

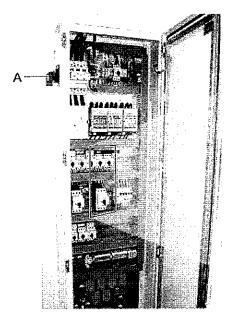
All danger areas at the APS*pro* must be freely accessible for maintenance and repair work. This is why it is necessary to disconnect the APS*pro* from the power supply prior to carrying out all such work. The following options are available (Fig. 2-2, 

25):

- Disconnecting the entire vapor deposition system with the main power switch (Pos.A) on the distribution cabinet.
- Disconnecting the power supply devices:
  - Switching off the plasma current power supply using the device switch and actuating the related safety switch (Pos.D) in the distribution cabinet.
  - Switching off the heater current power supply using the device switch and actuating the related safety switch (Pos.B) in the distribution cabinet.
  - Switching off the coil current power supply unit using the device switch on the rear side and actuating the related safety switch (Pos.C) in the distribution cabinet.



The layout and designation of the devices and circuit breakers may vary depending on the plant design. The location and identification of the switches can be seen in the relevant «circuit diagram».



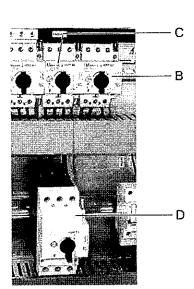


Fig. 2-2

Example: Safety switch for APSpro components in the SyrusPro distribution cabinet

- A Main power switch of the vapor deposition system
- B Safety switch for the coil current power supply unit
- C Safety switch for the pe 4606 heater current power supply
- D Safety switch for the GX 120/200 plasma current power supply

#### NOTE!

The main power switch and the safety switch have been designed so that they can be secured against unauthorized or inadvertent start-up by attaching a padlock to them.

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# 2.6 Dangers

# 2.6.1 Danger areas for operating personnel

When used in a Bühler Alzenau vapor deposition system, the APS*pro* plasma source system is fully integrated into the installation safety chain. This prevents a direct contact with the dangerous parts. It is possible to access the system components of the APS*pro* when the process chamber is open. For this reason, the following instructions must be strictly observed.

# AWARNING

The cathode material «Lanthanum hexaboride (LaB<sub>6</sub>)» is highly toxic.

The cathode used in the plasma source is composed of the highly toxic material «Lanthanum hexaboride». If the cathode is touched with the bare hands, this can lead to skin irritation or inflammation. Lanthanum hexaboride dust is highly toxic if inhaled and can cause damage to the lungs or health impairment.



The following protective measures and regulations must be adhered to:

- Do not touch the APS cathode (LaB<sub>6</sub>) with the bare hands. Always wear protective gloves!
- Always wash hands after coming into contact with the cathode and before eating.
- As far as possible prevent production of fine dust during mechanical processing of the cathode (grinding, scratches) and do not inhale. Always wear protective equipment!
- Always follow the information given in the manufacturer's safety data sheets, see: Chapter 8.1 Material Safety Data Sheet «Lanthanum boride»,
   103

# **ACAUTION**



Danger, the APSpro plasma source is hot

The plasma source becomes very hot during the process. Immediately after the end of the process, the plasma source is still hot which can cause burns.

Wait until the plasma source has cooled to room temperature before charging or unloading the process chamber. Wear protective gloves and work clothes with long sleeves.

# **ACAUTION**



Intensive light emission may cause blindness

Attempting to observe the plasma can cause eye damage due to the high intensity of the light.

Only view the plasma through a window equipped with a suitable light filter or wear suitable safety goggles.

# 2.6.2 Danger areas for maintenance and service personnel

In addition to the dangers for operating personnel outlined in Chapter 2.6.1 Danger areas for operating personnel, 

26, maintenance and service personnel must also observe the following safety precautions.

# **ADANGER**

High voltage and mains voltage.

The voltage for the magnetic coil, the plasma current power supply and the heater current power supply are all connected to the mains. The heater current power supply supplies the heater with high voltage. There is a danger of electrocution.



Please observe the following rules when working on the device connections:

- Switch off the devices and disconnect them from the electrical circuit
- Secure the devices against restarting (attach padlocks on the main power switch and the safety switch)
- Before restarting the device, ensure that there are no other persons in the danger area.

See Chapter 2.5 Disconnecting the energy supply, 25.

#### NOTE!

Only personnel with electrical training and with experience in dealing with high-voltage systems are permitted to carry out work on the electrical equipment.

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# AWARNING



plasma source, plasma current power supply, heater current power supply.



Improper handling during transportation can lead to serious or even fatal injuries.

Always maintain a safe distance from suspended loads. Never stand under a suspended load!

Only authorized personnel are permitted to operate forklifts, crane trucks and lifting devices.

# 



Water supply, gas supply and compressed air systems.

It is dangerous to open or damage a pressurized system part. It can lead to serious eye or skin damage.

Shut off the supply to the system and release the pressure before carrying out any work on these parts.

# AWARNING





Methanol is poisonous. If methanol enters the body (oral intake, through the skin, or through inhalation of the vapors) it can cause blindness and otherwise damage health.

Always wear protective gloves and work clothes with long sleeves when working with methanol. Wash methanol splashes off the skin immediately with water. Make sure that there is sufficient ventilation and do not inhale methanol vapors.

# **ACAUTION**



Cleaning materials.

Cleaning materials can contain substances hazardous to health.

Follow the safety instructions on the containers. When in doubt, always wear protective gloves and work clothes with long sleeves. Do not inhale the vapors.

# 



Dust and contamination.



Depending on the evaporating process, the plasma source could be contaminated with metal and oxide particles. The inhalation of dust can damage lungs and cause other health problems.

- Always wear a suitable breathing protection mask (P3) when carrying out cleaning work.
- Wear disposable protective clothing with long sleeves and gloves.
- Dispose of the collected dust as special waste.

# 3 Technology

The APSpro plasma source system is an assembly of various system components which together create plasma. Fig. 3-1, 🖹 31 provides an overview of the construction and location of the individual components.

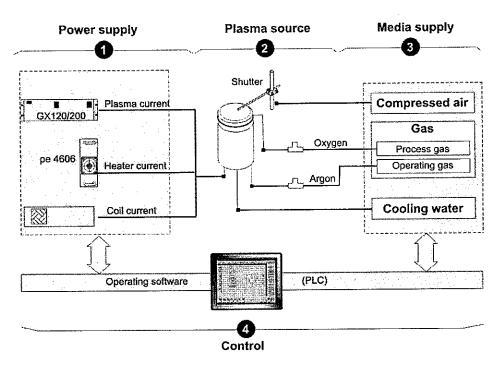


Fig. 3-1 APSpro -System overview

The individual components are described in the following chapters:

- Power supply (pos. 1) see: Chapter 3.2 Electrical connection, 

  38
  The devices GX 120/200, pe 4606 and the coil current power supply unit regulate the entire current and voltage for operating the plasma source.
- Plasma source (pos. 2) see: Chapter 3.1 The plasma source, 

  32 Is the main assembly and forms the plasma source for creating the plasma.
- Control unit (pos. 4) see: Chapter 3.4 Control, 

  45
  All APS system components are integrated into the operating concept of the respective system control (PLC).

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# 3.1 The plasma source

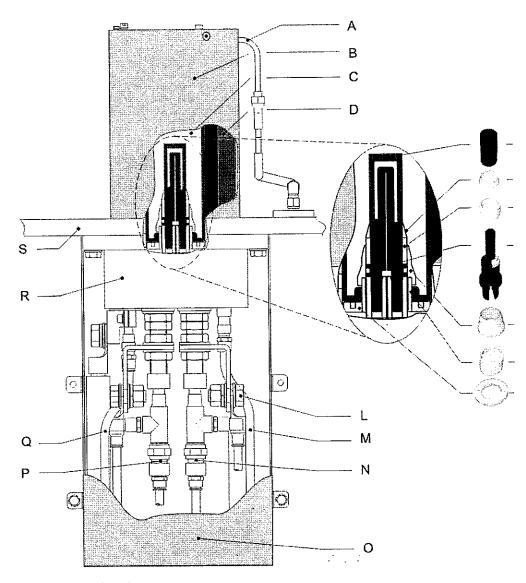


Fig. 3-2 Structure of the APSpro plasma source

- A Gas shower (oxygen inlet)
- B Shielded hood
- C Anode protective tube (insert)
- D Deflection coil
- E Cathode
- F Bornitiride ring (conical insulator)
- G Bornitiride ring (cylindrical insulator)
- H Graphite heater
- I Bornitiride bushing (insulator)
- J Bomitiride ring (insulator)

- K Ceramic ring (insulator)
- L Heater current connection
- M Cooling water return line anode protective tube
- N Cooling water supply line graphite heater
- O Protective cover/Casing
- P Cooling water return line graphite heater
- Q Cooling water supply line anode protective tube
- R Hub flange
- S Chamber base/Receiver
- T Metal ring
- U Gas shower (oxygen supply)

#### 3,1.1 The plasma source

The fig. below shows the important functional components in the plasma source.

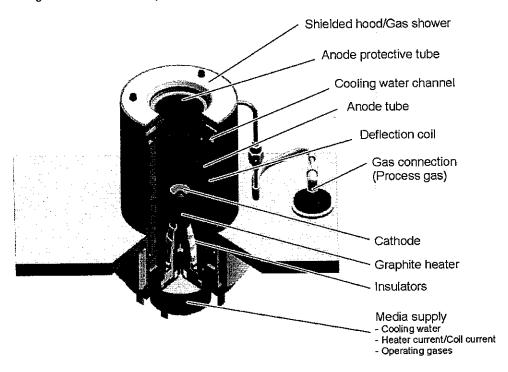


Fig. 3-3 APSpro cross section

#### Anode tube

The plasma is created in the electrical field of two electrodes. The anode tube forms the positive electrode and is made of copper. It is provided with an integrated water cooling unit. The anode tube is protected from damage and wear by the anode protective tube. A separate plasma current power supply provides the «discharge current» which is required to create the plasma. For more information concerning the «Anode tube» see:

Chapter 3.3.1 Cooling water connection and cooling circuit, @ 42

Chapter 3.2 Electrical connection, @ 38

#### Anode protective tube

The anode protective tube is a replaceable insert that is fitted in the anode tube. The anode tube and the anode protective tube are connected electrically via a multifunction strip. Ringlike drill holes allow process gas to enter into the combustion chamber. The anode protective tube must be serviced on a regular basis. For more information about the «Anode protective tube» see:

Chapter 6.3.2 Anode protective tube, @ 67

#### **Deflection coil**

A current-carrying coil generates a magnetic field that is used to align the plasma in the anode tube. The magnetic field is located parallel to the anode axis and forces the electrons emitted from the cathode onto spiral trajectories along the magnetic field lines. The coil current is supplied via a separate coil current power supply unit. For more information about the «deflection coil» see:

Chapter 3.2 Electrical connection, 38



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Process gas shower

The so-called process gases (oxygen) is fed to the plasma source via the gas shower. The gas shower is an integral component of the removable shielded hood. Gas is supplied via a separate gas opening mounted separately onto the base of the process chamber and a flow controller mounted on the outside of the process chamber. For more information about the «Process gas shower» see:

Chapter 3.3.2 Gas supply, @ 44

#### Ceramic insulators

The plasma source is equipped with various ceramic insulators that electrically isolate the components from each other.

Fig. 3-4, \$\exists 34\$ shows the insulators relevant for the maintenance and service work and their position within the plasma source. The ceramic insulators must be cleaned and maintained on a regular basis, see:

Chapter 6.2 Maintenance plan, \$\exists 61\$

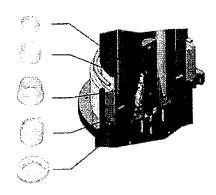


Fig. 3-4 Position of the insulators

#### 3.1.2 Cathode

The cylindrical cathode is manufactured from lanthanum hexaboride ( $LaB_6$ ). The cathode supplies the electrons for the ionization process for creating plasma

#### Principle structure

The cathode (A) is plugged onto a heater (B). Through the indirect heating of the cathodes by the connected heater, the electrons required for the ionization process are released from the cathode material (emission). As a result, material is removed from the cathode surface. The diminishing wall thickness limits the service life of the cathode

#### **Electrical connection**

The cathode is electrically connected to one leg of the heater (B) on the bottom side. The second leg of the heater is electrically isolated from the cathode by means of an insulator (C). The cathode has a separate power supply which controls the discharge voltage in the electrical field, see: Chapter 3.2.2 Connection diagram of the voltage and electrical circuits, § 40

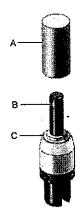


Fig. 3-5 Cathode with heater

- A Cathode
- B Heater
- C Insulator

During the process, cathodes are subjected to strong thermal and process-specific loads as well as to a loss of material. Cathodes can exhibit various degrees of wear which is indicated by their color and shapes depending on the loads they are subject to and how they are used. A new cathode (A) has a violet color. If it is exposed to oxygen either too long or at excessive temperatures, it will gradually turn orange in color (D). This is referred to as a «poisoned» cathode. The plasma source can no longer be operated in this condition. By conditioning a cathode, it can be restored to a state in which it can be reused, see:Chapter 7.4.2 Cathode <Regeneration>, 

92

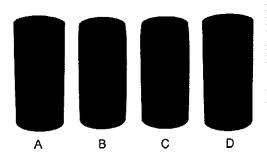


Fig. 3-6 Cathodes subject to different loads

- A New cathode
- B Used cathode (less loss of material)
- C Slightly discolored cathode («poisoned»)
- D Very discolored cathode («poisoned»)

#### Safety note for cathode materials

The cathode is composed of a sintered form of «Lanthanum hexaboride (LaB<sub>6</sub>)». This material is highly toxic. Health can be seriously damaged if dust particles are inhaled or by coming into direct contact with this material.

# AWARNING

The cathode material «Lanthanum hexaboride (LaB<sub>6</sub>)» is highly toxic.

The cathode used in the plasma source is composed of the highly toxic material «Lanthanum hexaboride». If the cathode is touched with the bare hands, this can lead to skin irritation or inflammation. Lanthanum hexaboride dust is highly toxic if inhaled and can cause damage to the lungs or health impairment.



The following protective measures and regulations must be adhered to:

- Do not touch the APS cathode (LaB<sub>6</sub>) with the bare hands. Always wear protective gloves!
- Always wash hands after coming into contact with the cathode and before eating.
- As far as possible prevent production of fine dust during mechanical processing of the cathode (grinding, scratches) and do not inhale. Always wear protective equipment!
- Always follow the information given in the manufacturer's safety data sheets, see: Chapter 8.1 Material Safety Data Sheet «Lanthanum boride»,
   103

#### **3.1.3** Heater

#### Principle structure

The heater consists of graphite and its function is to indirectly heat the attached cathode through radiated heat. The heater is heated to a temperature of about 1500 ° via a power supply. The connecting legs (B) for the heater are plugged into a water-cooled heater receptacle. The cooling water is supplied via connections from the bottom. See: Chapter 3.3.1 Cooling water connection and cooling circuit, § 42

#### Electrical connection

One leg of the heater is electrically connected to the cathode, whereby the other leg is electrically isolated from the cathode by means of an insulator (A). The heater is connected to an AC power supply. See: Chapter 3.2.2 Connection diagram of the voltage and electrical circuits. 

40

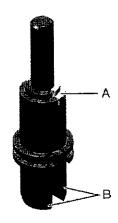


Fig. 3-7 Graphite heater

#### NOTE!

The heater is made of porous, pressure-sensitive graphite. Please note

- Do not touch the heater with your bare hands (wear cotton gloves)
- Do not use undue pressure when pressing the heater legs together. They may break.

#### Markings and features on the heater

The notch (pos. C) on the heater leg marks the position of the insulator (pos. A). The marking is intended as an orientation to enable the heater to be positioned correctly in a mounting tool when servicing the cathode. See: Chapter 7.3.4 Installing the heater-cathode unit, § 87.

The rating for the cold resistance (pos. B) is established by the manufacturer and engraved on the surface (manufacturer specification). The value indicated is used to calculate the theoretical heating voltage. This adjusts the heat flow when new heaters are put into operation

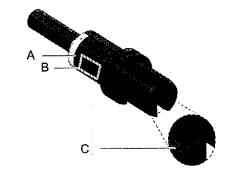


Fig. 3-8 Markings on the heater

- A Ceramic insulator
- B Heater cold resistor (manufacturer specification)
- C Marking for the installation position of the ceramic insulator

### Insulators on the graphite heater

The graphite heater is equipped with various insulators.

The insulators (A) and (B) protect the graphite heater from coming into direct contact with the plasma in the plasma source.

The ceramic shell (C) electrically isolates the attached cathode from the graphite heater

The ceramic shell is fastened to the graphite heater by a pin (D).

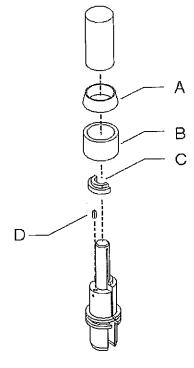


Fig. 3-9 Insulators on the graphite heater

# 3.2 Electrical connection

### 3.2.1 Power supply devices

As shown in figure Fig. 3-10, § 38, the following plasma source assemblies (pos. A) are connected to the electrical power supply:

- Anode and cathode: See Chapter 3.2.1.2 Plasma current power supply (GX 120/200),
   39.

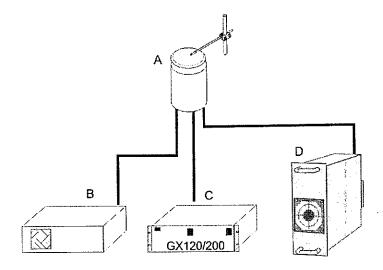


Fig. 3-10 Overview of the APSpro power supply devices

- A Plasma source
- B Power supply for the coil current
- C GX 120/200 plasma current power supply
- D pe 4606 heater current power supply

# 3.2.1.1 Coil current power supply unit

The power supply unit provides direct current to the deflection coil of the plasma source. Normally, the power supply unit is installed in the control cabinet of the vapor deposition system. The output current level is regulated by external control signals from the PLC electronic controller.



For detailed information refer to the respective supplier documentation.

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## 3.2.1.2 Plasma current power supply (GX 120/200)

The plasma current power supply is a fully digitized and micro process controlled current generator, that guarantees a constant plasma current supply and that is equipped with two state-of-the art arc-management-systems for discharge voltage and bias voltage. The set point for the discharge current is provided via the PLC.



For detailed information see the respective supplier documentation.

# 3.2.1.3 Heater current power supply (pe 4606)

The Heater current power supply supplies the heater with direct current. Because of the fluctuations in potential that can occur after the plasma is ignited, the output voltage of the heating generator must be potential-free. The output power is regulated via the PLC.



For detailed information see the respective supplier documentation.

# 3.2.2 Connection diagram of the voltage and electrical circuits

The illustration Fig. 3-11, 

40 shows the electrical connection diagram of the current and voltage circuits on the plasma source which are required to create the plasma.

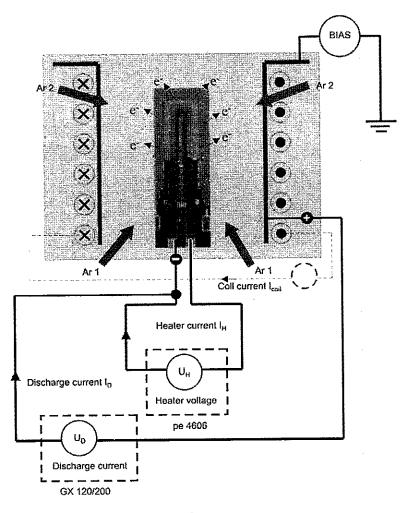


Fig. 3-11 Overview of the electrical circuits of the APSpro

### **Heating current**

A current-carrying graphite heater indirectly heats the cathode located above it. The supply of thermal energy causes electrons to be emitted from the cathode material. The thermal energy must be so high that there are enough electrons available to trigger the avalanche-type build-up of gas ions in the plasma source. If the heating current is too high, the service life of the heater and the cathode unit will be reduced considerably.

#### Discharge voltage

In the electrical field between the cathode and the anode, the free electrons emitted from the cathode are accelerated. If the kinetic energy of the free electrons is as large as the bond energy of the electrons of the gas atoms, ions can be created through the collisions between the electrons and the gas atoms.

The positive pole of the electrical current is connected to the anode tube and the negative pole is connected to the cathode via a heater connection. Because of the potential shifts that can occur after the plasma is ignited, the output voltage of the plasma current power supply must be potential-free.

#### Coil current

The coil current creates the magnetic field within the plasma source. Thus depending on the energy they release, the electrons can be accelerated so strongly that they ionize further gas atoms. Furthermore, the magnetic field strength determines the shape of the plasma cone. The more the coil current increases, the more field lines contract along the magnetic field axis. During this process, the plasma cone becomes narrower.

#### Bias voltage

The bias voltage is a parameter value that is determined by the following settings:

- Coil current
- Heating current
- Discharge voltage

The bias voltage level is critical for generating the field strength between the plasma source and the substrate. The gas ions of the plasma are accelerated towards the substrates through the bias voltage (electrical field). The bias voltage controls the kinetic energy of the gas ions where the substrate is located and thus plays an important role in determining the property of the layer to be deposited.

# 3.3 Media supply

# 3.3.1 Cooling water connection and cooling circuit

Due to the thermal loads that occur, the anode tube and the heater receptacle are protected against overheating by cooling circuits. The components are cooled by two cooling circuits that are fed separately to the plasma source. Both cooling circuits are integrated into the cooling water system of the system



For detailed information about the cooling system, see the respective «Operating Manual» of the vapor deposition system.

### 

The cooling water flows through the inlet (A) in the direction of the anode tube and then through the cooling water channels (B) of the anode tube. The neighbouring anode protective tube is indirectly cooled in the process.

The cooling water is then returned to the cooling circuit via the outlet (C). The cooling water flow (volume flow) is monitored by a flow monitor in the return line.

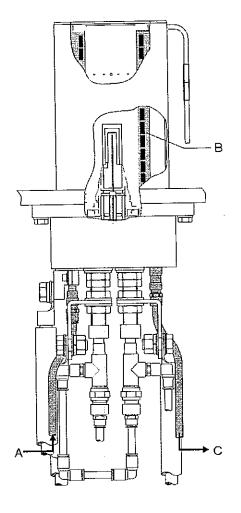


Fig. 3-12 Cooling water circuit < Anode tube>

### Cooling circuit of the heater receptacle Fig. 3-13, 2 43

The cooling water first flows through the inlet (A) directly to the heater socket in the area of both heating connections (B). Afterwards the cooling water is returned to a connecting branch and the water is then re-supplied to the heater socket. The cooling water is then returned to the circulation system through the outlet (C). The cooling water flow (volume flow) is monitored by a flow monitor in the return line.

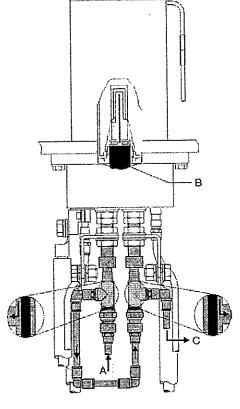


Abb. 3-13 Cooling water circuit < heater receptacle>

### Cooling water connection requirements

To prevent condensation from forming on the cooled assemblies prior to the start of the process, the cooling water must be preheated to a temperature above the current dew point.

Criteria	Data:
Flow rate – inlet (min)	8 liter/minute
Flow rate – outlet (min)	2-3 liter/minute
Water temperature	15 -25 °C

Tab. 3-1

Cooling water data

### 3.3.2 Gas supply

To operate the plasma source, so-called operating and process gases are required. The APS*pro* is equipped with a total of three gas connections with which the gases are supplied.

A gas supply unit outside the process chamber helps regulate the amount of gas. The unit basically consists of a flow regulator (D) for metering the gas and a solenoid valve for shutting off the gas. Both are driven by the PLC electronic controller. The illustration in Fig. 3-14, \( \begin{align\*} \begin{align\*} \text{44} \\ \text{displays an overview of the locations of the gas connections.} \end{align\*}

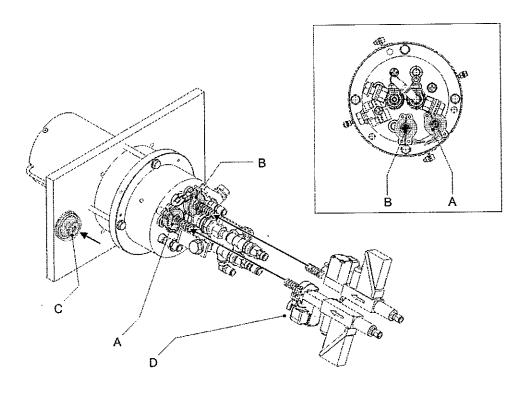


Fig. 3-14 Gas connections on the APSpro .

- A Operating gas Argon (Gas flow 1)
- B Operating gas Argon (Gas flow 2)
- C Process gas (for the oxygen shower)
- D Gas flow regulator

#### Gas connection for the operating gas «Argon»

Two gas connections are located on the flange of the plasma source through which the operating gas argon is introduced into the plasma source. The operating gas is used to create the plasma. One connection (A) conducts gas from the bottom directly into the plasma source near the cathode. A second connection (B) conducts the gas directly through the anode tube into the plasma source.

### Gas connection for the process gas «oxygen»

A separate gas connection (C) on the bottom of the process chamber supplies the gas shower with oxygen.

## 3.4 Control

The APSpro is not a stand-alone system, but an constituent part of the system controller. The APSpro and their components are integrated into the hardware and software configuration of the system control and are controlled and regulated via a PLC connection. The PLC installation component is represented by an independent Profibus station in the control cabinet of the system. Data is transferred between the system controller (Vis PC) and the Profibus station via a communication PC (IPC) and an Ethernet connection to which other system components may also be connected. The user operates the APSpro from the operating terminal of the system controller using the respective screen displays to the display and enter the parameters (set points/actual values).

The illustration Fig. 3-15, § 45 shows an overview of the APSpro device components integrated into the system controller (the SYRUSpro system controller is shown here).

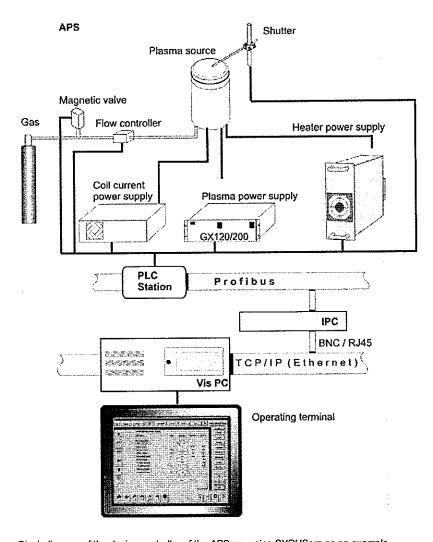


Fig. 3-15 Block diagram of the device controller of the APSpro, using SYRUSpro as an example.

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Operator's Manual § 45

Bühler Alzenau

# 4 Installation, Initial Start-Up

# 4.1 Notes on installation and initial start-up

The APSpro system is always mounted, installed and put into operation as a part of the overall vapor deposition system. The following section refers exclusively to a plasma source that has already been fully installed in the vapor deposition system. The initial start-up of the plasma source is always a part of the system start-up procedure.

### NOTE!

Only specially trained Bühler Alzenau service technicians are permitted to put the APSpro into operation.

# 4.1.1 Transferring responsibility of the APSpro to the operator

After the APSpro has been successfully installed and started, responsibility for the system is transferred to the operator.

# 4.1.1.1 Demonstration of the safety locks

When the APSpro is transferred to the operator, the function of the safety and locking circuits must be demonstrated to the operator. The vapor deposition installation must have been fully tested and be in a ready-for-operation condition.

The error-free functioning of the safety provisions must be confirmed in writing on the acceptance certificate by both the operator and the Bühler Alzenau service technician.

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# 4.1.2 Repeating the tests

In accordance with the DIN IEC 703/VDE 0721 regulation, section 6, as well as the relevant national safety regulations, a repeated test of the electrical equipment (safety circuits and protective conductor system) must be carried out every 6 months. The test is also required when technical changes are carried out on the safety circuits or safety switches.

#### Safety circuits and protective conductor system

After changes have been made to the system, or following maintenance work, whereby safety-related components are affected, the function of the safety circuit must be tested.

- 1 Test the safety circuit. Activate the EMERGENCY OFF switch and the safety locks several times (at least five times).
- Test the protective conductor system and the protective conductor connections of the APSpro, especially the connection points, by conducting a manual and visual inspection. We also recommend measuring the protective conductor's resistance between the main connection and different conductive system parts.
- 3 Check that all cables carrying a large current have good contact.

#### Safety information for cooling water circuit

Carry out the following tests:

- 4 Check that the flow monitors for the cooling water activate when the flow rate falls below the specified minimum value. Repeat this test several times (at least five times).
- 5 Check the entire cooling water installation for leaks and damaged or cracked hoses.

### Mechanical tests

Carry out the following mechanical tests:

- 6 Carry out a visual inspection of all the wearing parts. For further details, please consult the relevant instruction manuals.
- 7 Check that all the mechanical safety equipment (e.g. hoods, covers, locks, etc.) has been correctly installed.
- If mechanical or static changes are carried out on the system (e.g. holes are drilled in the reinforcing ribs), check the mechanical stability of the system. Reinforce the relevant parts as needed.

The repeat acceptance test is to be confirmed in writing on an acceptance certificate by a person appointed by the operator.

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#### Adjust voltage value

The voltage value can be adjusted via the machine constants using a calibration factor.

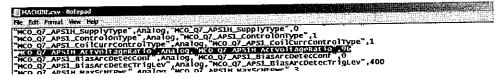


Fig. 4-1 Machine constants

- 9 Call up machine constants file
  - 9.1 Change the factor «MCO\_Q7\_APS1\_MActVoltageRatio» (Default = 100) (e.g. adjust to = 96).
    The value displayed in the visualization is changed according to the factor entered here.
  - 9.2 Repeat the procedure until the value displayed in the visualization matched the actually measured value Fig. 4-2, 149.

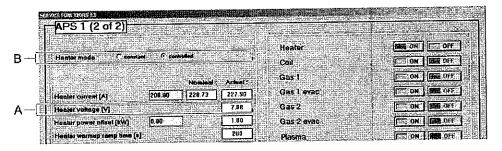


Fig. 4-2 Screen Service Functions APS (OptiControl)

Check heater for wear and wrong positioning

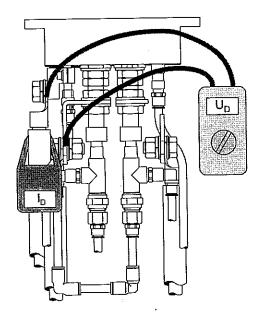


Fig. 4-3

# 5 System Operation

## 5.1 Introduction

# 5.1.1 Personnel qualifications

The APSpro may only be switched on and operated by personnel with the required authorization. It is the responsibility of the operator to ensure that the APSpro is only operated by appropriately trained personnel.

# 5.1.2 Operation and safety

The APSpro is not an independent system. It can only be operated from a higher-level system (i.e. SYRUSpro).

Before operating the APS*pro*, Chapter 2 Safety, \$\mathbb{B}\$ 21 must be read and fully understood. Pay particular attention to the information concerning potential dangers. For further information, see Chapter 2.6 Dangers, \$\mathbb{B}\$ 26

### NOTE!

Bühler Alzenau assumes no responsibility for damages caused by unauthorized personnel operating the device. The safety precautions in Chapter 2 Safety, 

21 and the permission must be strictly observed.

# 5.2 Operating the APSpro

The APSpro is operated from the operator terminal of the conjunction system. A user-friendly visualization program supports users in manually controlling the individual system components and in creating automatic processes.

A vapor deposition system is comprised of a number of components. As the control commands of the individual components in process operation must be adapted to each other, there is no point in describing operation of the APS*pro* here as an isolated process.



For details on operating the APSpro and integrating it into an automated process, see the «software manual» of the visualization of your system.

# 5.2.1 APSpro screen display

All parameters required for operating the plasma source can be seen and set on the corresponding APS*pro* screens of the visualization system. As an example Fig. 5-1, § 52 shows a screen of the visualization system «OptiControl».



For details on the display screen of the APSpro, see the «software manual» of the visualisation of your plant system.

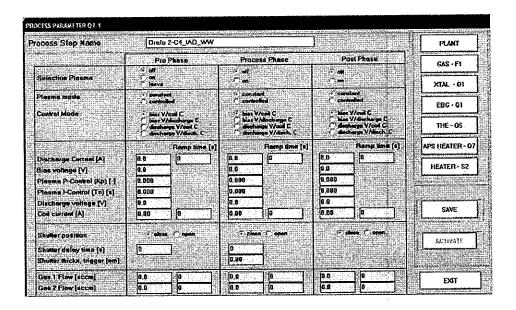


Fig. 5-1 Example: Monitor screen for APS control for «SyrusPro-Plant»

# 5.2.2 Operating phases and control sequence

The following Fig. 5-2, \$\bigsim 53\$ shows exemplarily the typical manual operating sequences of the APS*pro* system during the plant operating phases. The entire process steps from «switch on» until «switch off» will be controlled via the plant control system.

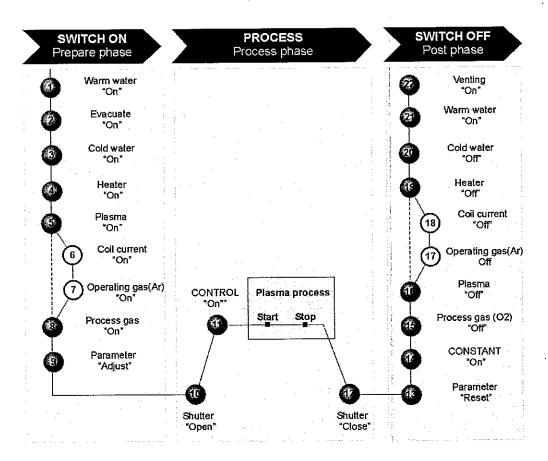


Fig. 5-2 APSpro manual control sequences during operating phases for «SYRUSpro» plant

#### 1 Warm water on:

To prevent water condensing on the cooled components, warm water flows through the pipes.

### 2 Evacuate on:

The process chamber is closed and evacuated.

#### 3 Cold water on:

Cold water supply activated for the anode tube and heating receptacle. The interlocks to switch on the APS (heater on, coil current, etc.) are only lifted when the cooling water circuit is switched on.

#### 4 Heater on:

The heater is heated by a alternating current, which is steadily increased to approx. 1500 °C, thereby heating the cathode above it. The heating power remains constant during the entire process time.

#### 5 Plasma on:

When the «plasma on» function is switched on, at the same time the function **coil current on (6)** and **operating gas on (7)** are automatically activated. The plasma current power supply ignites the plasma and controls the set point of the discharge current.

The plasma source is charged to a positive self-bias voltage whose potential depends on the following factors:

- Gas flow of the operating gas
- Gas flow of the process gas
- Discharge current
- Strength of the magnetic field of the deflection coil

#### 6 Coil current on:

The deflection coil round the anode tube is supplied with direct current, which generates a constant magnetic field to control the plasma. The function switches on automatically when the «plasma on» function is activated.

#### 7 Operating gas on:

Argon gas flows into the combustion chamber of the plasma source. The function switches on automatically when the "plasma on" function is activated.

#### 8 Process gas on:(option)

The reactive gas (oxygen) is fed in via the gas shower.

#### 9 Set parameter:

The parameter values for the discharge current, voltage and gas flow are adjusted to the typical process conditions and activated.

#### 10 Shutter open:

The shutter over the plasma source pivots to the side, thus releasing the opening to the combustion chamber. The plasma treatment process starts.

### 11 «CONTROL» mode on:

Plasma source operating type is switched from CONSTANT to CONTROL. The heater power controlled automatically via the heating current.

#### 12 Shutter closed:

The shutter closes over the plasma source. The plasma treatment process is finished.

#### 13 Parameter reset:

The parameter values of the plasma source for discharge current, voltage and gas flow are reset.

#### 14 «CONSTANT» mode on:

The plasma source operating mode is switched from CONTROL to CONSTANT.

#### 15 Process gas off::

The gas inlet (oxygen shower) is turned off.

#### 16 Plasma off:

The current supply to the deflection coil round the anode tube is interrupted. The plasma is extinguished, as soon as the discharge current generated by the plasma current power supply is interrupted. At the same time, the **operating gas (17)** and the **coil current (18)** are switched off.

Operating gas off: 17

The supply of argon gas in the combustion chamber is closed. The function switches on automatically when the «plasma off» function is activated.

#### 18 Coil current off

Direct current supply of the deflection coil is interrupted. The function switches on automatically when the «plasma off» function is activated.

#### 19 Heater off:

The heater current is slowly reduced to zero. The plasma source is deactivated.

The plasma source can be switched on again with the «Heater on» (4) function. 20

# 6 Maintenance and Service

# 6.1 Introduction

This chapter provides an general overview of all the measures necessary for the maintenance and servicing of the APS*pro*.

The APSpro provides virtually trouble-free operation when the measures for preventative maintenance and the working conditions and regulations specified are observed.

## 6.1.1 Safety information

### NOTE!

Before carrying out maintenance and service work on the APS*pro*, the appointed personnel must have read and fully understood the Chapter 2 Safety, 

21.

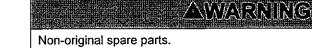
# 6.1.2 Personnel qualifications

#### NOTE!

It is the responsibility of the operating firm to ensure that:

- All work is carried out in accordance with the system maintenance schedule
- The maintenance personnel have completed the training and instruction necessary for this work

# 6.1.3 Spare parts and modifications





The use of third-party parts can lead to malfunctions. This in turn can cause serious or fatal injuries or considerable material damage.

When carrying out maintenance and service work, only use original Bühler Alzenau spare parts.

#### Ordering

Send all orders, including the order numbers and the exact name of the spare parts, to Bühler Alzenau.

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#### **Contamination statement**

When sending vacuum components for repair or replacement to Bühler Alzenau, you must enclose a completed and signed contamination statement. See: Chapter 7.5.2.1 Contamination declaration, 102

#### **Modifications**

Unauthorized modifications and alterations which affect the safety of the APSpro are not permitted.

### 6.1.4 Use of operating supplies

The term "operating supplies" covers all consumables and cleaning materials required for the maintenance and service of the APS*pro*. Essentially, these are the cleaning agents and lubricants used.

Cleaning materials used are:

- 3M Scotch Brite
- Alcohol (methanol, ethanol, isopropyl)
- Solvents for grease (e.g. acetone)

#### NOTE!

The use of conventional greases and lubricants is not permitted. In addition, the amount of vacuum grease used on vacuum seals and bushings must be kept to a minimum.

All the objects and parts that enter the process chamber and are exposed to the vacuum must be free of grease. The maintenance time required depends largely on how clean objects, process chamber and parts are.

## 6.1.5 Tools and Devices

To carry out the maintenance and service work on the plasma source, special tools and devices are required that remove and mount the individual components. The required tools and devices have been combined in a tool kit and are included in the delivery scope of the APS plasma source.

#### NOTE!

The tool kit is included in the spare part packages of the APS plasma source. Carry out the maintenance and service work only with the tools that have been furnished for the plasma source.



For detailed information on ordering the tools, see: CD2, electronic spare parts catalogue for the respective system documentation.

Fig. 6-1

Fig. 6-2

### Overview of the required tools and devices

### Tweezers (long)

Are required to install and remove the isolating insulator on the heater receptacle. See: Fig. 7-13, 

84



#### Retaining clamp

Is required to install and remove the bornitride ring on the heating receptacle.

See: Fig. 7-13, 

84



Tweezers

Retaining clamp

### Carrying handle

Is required for installation and for transporting the plasma source.



#### Change device

Is required for removing and installing the heater and cathode.

See: Fig. 7-18, 

87



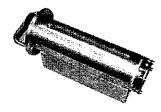


Fig. 6-4 Change device

### 6.1.6 Final checks

After maintenance or service work, check that all the safety equipment is installed and functioning properly. Check the safety equipment especially after working on the electrical system.

#### Obligation to notify

The operator is obliged to notify Bühler Alzenau immediately about any changes or irregularities that have been observed in the APSpro.

## 6.1.7 Using the maintenance documents

The maintenance documents for the APSpro consist primarily of:

- A maintenance schedule
- Maintenance instructions

#### Maintenance schedule

The maintenance schedule provides an overview of the maintenance work to be carried out on the APSpro system. Each maintenance schedule is in tabular form and contains the components to be maintained, the work to be carried out, and the frequency of the maintenance.

#### **Maintenance instructions**

Comprehensive maintenance work is indicated by a cross-reference in the «Remarks/instructions» column of the maintenance schedule. It is described in greater detail in the «Maintenance instructions» section. See Chapter 6.3 Maintenance instructions, 

64.

#### Maintenance frequency

The test and maintenance frequencies indicated are the compulsory minimum requirements for normal system operation. Normal system operation amounts to three-shift operation 6 days a week or 7500 operating hours per calendar year.

#### Maintenance verification

Bühler Alzenau recommends that all maintenance work be recorded in a logbook. Especially when several people are responsible for the maintenance of the system, this provides a reliable form of verification of what maintenance work was carried out, and at what time.

#### Use of supplier documentation

The maintenance and service of individual components of the system is described in detail in the supplier documentation from the manufacturer.

Maintenance work referred to by the supplier documentation must be carried out precisely according to the information in the relevant operating instructions. See Chapter 1.1 Product information, 3.7.

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# 6.2 Maintenance plan

Fig. 6-5, 🗎 61 shows the position of the plasma source components, which must be serviced. The work that should be undertaken is given in the maintenance list.

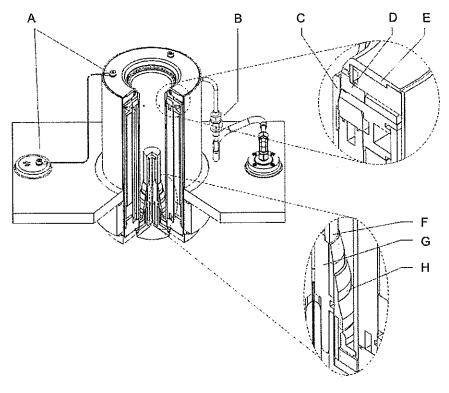


Fig. 6-5 Maintenance points on the plasma source

- A Earth connection
- B Gas connection (oxygen shower)
- C Anode protective tube
- D Ceramic ring (oxygen shower)
- E Aluminium ring (oxygen shower)
- F Cathode
- G Heater
- H Ceramic insulators

Component activity	Interval	Remark/instruction
Earth connection Check contact and connection	monthly	Check that the cable connections are firm and undamaged. Replace damaged cable connection.
Gas connection (oxygen shower) Check O-ring sealing	monthly	Check screw connections and sealing surface. Check O-ring seal.
Anode protective tube Clean, replace if necessary	*) after each batch	*) Clean depending on level of contamination. See: Chapter 6.3.2.2,   69

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Component activity	Interval	Remark/instruction
Ceramic ring (Gas shower) Clean	*)	*) Depending on level of contamination. Wipe with a rag or Scott Brite. See:Chapter 6.3.3,   71
Aluminium ring (gas shower) Clean and check for roasting residue (arc traces)	*)	*) Depending on level of contamination. Wipe with a rag or Scott Brite. Replace ring if contamination is excessive. See: Chapter 6.3.3,   ** 71
Heater Check, if necessary replace	After reaching specified number of operating hours	Check amount of material/damage and replace if necessary. See: Chapter 7.3.2,   81
Cathode Check, if necessary replace	After reaching specified number of operating hours	Check amount of material/damage and replace if necessary See:Chapter 7.2.3,  76 Reset the operating hours counter after the maintenance work
Ceramic insulators Check for amount of material/damage and replace if necessary	After reaching specified number of operating hours	See: Chapter 7.3.2.3,



Carry out the maintenance work only in accordance with the «Operating Manual» and with the existing «supplier documentation» for the APS*pro* parts and units.

#### NOTE!

Never touch parts taken from the process chamber with bare hands. Always wear clean room gloves before touching any process chamber part.

### NOTE!

Before starting any cleaning and repair work in the process chamber, close off the combustion chamber with a stopper. This prevents any particles from falling into the combustion chamber. Upon completion of the cleaning, the stopper must be removed.

# 6.2.1 Power supply



Carry out the maintenance work only in accordance with the «supplier documentation» for the APS*pro* parts and units.

Component activity	Interv.	Pers.	Remark/instruction		
Heater current power supply pe 4606					
<ul> <li>Clean fan and fan channel</li> <li>Check function of fan</li> <li>Check quality of cooling air</li> <li>Visual check of housing and connections</li> <li>Visual check electrical connections</li> </ul>	P*	MP	*) Depending on ambient condi- tions and operating hours		
Coil current power supply					
<ul> <li>Clean the filter mats</li> <li>Check that the filter fan is functioning</li> </ul>	P*	MP	*) Depending on how dirty it is. Clean the filter mat with compressed air, or replace it if necessary.		
			Use the supplier documentation!		
Plasma current power supply GX 120/200					
Visual check	P*	MP	*) Depending on ambient conditions and operating hours		

## 6.3 Maintenance instructions

This section contains guidelines and work instructions for carrying out maintenance work on the APS*pro*.

## 6.3.1 Notes on the glass bead blasting

# AWARNING



Dust and blasting material.

The inhalation of dust can damage the lungs and cause other health problems.

Always wear a suitable breathing protection mask (P2) when carrying out work with a glass bead blasting device. Wear disposable protective clothing with long sleeves and gloves. Dispose of the collected dust as special waste.

#### NOTE!

Seal off all sealing surfaces, threaded holes and other sensitive surfaces of the part being treated before beginning glass bead blasting.

#### NOTE!

Observe the following requirements for glass bead blasting. This is the only way to ensure that the components are properly cleaned.

Designation	Requirements
Surface of the parts to be cleaned	Clean, dry and free of oil
Compressed air	Filtered, dry and free of oil
Working pressure	Approx. 1.5 bar
Angle of incidence	< 45° for thin components to a minimum 0.5 mm thickness. Parts with a thickness of < 0.5 mm should not be glass bead blasted.

#### Tab. 6-1 Requirements for glass bead blasting

#### Blasting procedures:

- Blast cleaning: This blasting procedure is used to remove characteristic or foreign layers as well as impurities from the surface of the objects.
- Shot peening: This blasting procedure is used to create tensile strain in the layers near the surface of the object in order to improve certain component properties (such as surface adsorption). Shot-peening is carried out with rounded pellet-like material (glass beads).

 Rough blasting: This blasting procedure is used to alter the surface shape in order to achieve better adhesive properties for the coating. Rough blasting is carried out with edgedshaped blasting material (special fused alumina).

### Blasting material and recommended surface roughness values

The degree of surface roughness to be attained depends on the material, surface and stability of the object to be blasted as well as on the blasting material and blasting procedure. The recommended surface roughness values are listed in relation to these parameters in the following table.

Blasting procedure:					
	Clean blasting	lean blasting Shot peening		Rough blasting	
Recommended blasting material:					
	Chilled cast iron GH-K 0.3-0.6	Glass beads MGL 0.071-0.16	Special fused alumina MKE 1-2		
Blasting material:	Surface roughness 50μm:		Dimen- sionally stable	Dimen- sionally instable	
Aluminium and alloys		≤ 40	60120	4060	
Copper and alloys		≤ 40	60120	4060	
CrNi steel		≤ 35	50 80	3050	
Steel, ferritic	30100				

Tab. 6-2 Blasting material and standard surface roughness values for various objects

#### General procedure

- 1 Prior to glass bead blasting, seal off all sealing surfaces, threads, threaded holes and bore holes that have tolerance specifications.
- 2 Use mechanical tools to carefully remove thick layers that have poor adhesive qualities.
- 3 Clean all parts with clean water and degrease as needed with alcohol. Dry all parts in the dry flow of compressed air.
- Blast all parts under the conditions listed in Tab. 6-1, 

  6-1, 6-4 to a final roughness listed in Tab. 6-2, 6-5.
- 5 Clean all parts in the ultrasonic bath. Be sure to use distilled water.
- 6 Rinse off and dry all the cleaned components in a clean environment.

### Dispose of the blasting material after you are finished

The blasting material must be renewed under the following circumstances:

 After blasting hard layers (e.g. W, Mo) to prevent damage to softer parts caused by hard layer particles.

- After blasting non-stainless steel parts to prevent contaminating other materials.
- After blasting toxic layers. In this case, please observe the following warning.

# 



Poisonous coating materials.

Depending on the process type, the APS*pro* may be contaminated with a poisonous coating material.

Carefully read the safety information sheets of the process materials used and strictly observe the precautionary measures specified therein.

# 6.3.2 Anode protective tube

The inner wall of the anode tube of the plasma source becomes increasingly covered with roasting residues and scales (material layer) as the process time progresses. This layer changes the electrical conductivity of the surface of the anode, and thus also the plasma properties. The anode tube must therefore be changed and cleaned at regular intervals.

## 6.3.2.1 Replace anode protective tube

### Dismounting <anode protective tube>

- Hold the anode protective tube as shown in Fig. 6-6, 6 67 and pull upwards out of the plasma source.
  - The bornitride bushing will also be pulled out in the process.
  - Set the anode tube down onto a suitable surface



Fig. 6-6 Dismounting anode protecting tube

- 2 Using long tweezers remove the bornitride bushing from the anode protective tube
  - Check the bornitride bushing for signs of cracking or wear. Clean the bornitride bushing or replace it as needed.
  - Check the anode protective tube for dirt. Clean the inside of the anode protective tube only by blasting it with glass beads.
     For cleaning instructions, see: Chapter 6.3.2.2 Cleaning instruction for <anode protective tube>,
     69



Fig. 6-7 Remove bornitride bushing

#### Mounting the <Anode protective tube>

- 3 Insert the bornitride bushing into the anode protective tube (see Fig. 6-7, 

  67)
  - Ensure that the bornitride bushing is positioned concentrically into the anode protective tube.
- 4 Clean the plasma source before inserting the anode protective tube.
  - Check the inside of the anode tube for dirt by using a flashlight. Remove any loose particles of dirt and vacuum the anode tube. The vacuum cleaner should be equipped with a narrow, plastic nozzle attachment.

- 5 Insert the anode protective tube into the plasma source (see Fig. 6-6, 🖺 67)
  - Lower the anode protective tube insert as far as it will go into the plasma source.
  - Press the anode protective tube down with both thumbs until it comes to a stop

#### NOTE!

The anode protective tube is fitted into the anode tube in such a way that there is a good electrical and thermal contact between both components. This is why you will feel a mechanical resistance when you install the anode protective tube. If the anode tube is deformed, this resistance can be so strong that the anode protective tube can only be inserted into the anode tube with great difficulty or not at all.

- At any rate, do not apply mechanical force.
- Keep in mind that it may not be possible to pull out an anode protective tube that has been forced into the anode tube.
- Do not use deformed or damaged anode protective tubes
- 6 Check whether the bornitride ring is in the proper position by using a flashlight. If necessary, slide the bornitride bushing into the proper position using long tweezers.

# 6.3.2.2 Cleaning instruction for <anode protective tube>

The inside of the anode protective tube must be regularly glass bead blasted. To attain as identical conditions as possible for the plasma-supported vapor deposition, we recommend cleaning the anode tube after every charge. Observe the conditions specified in Chapter 6.3.1 Notes on the glass bead blasting, § 64 when cleaning the anode tube.

#### NOTE!

To achieve uniform process characteristics, we recommend installing a clean (or new) anode tube insert for each new production batch. To avoid downtimes or keep them as brief as possible, we recommend that working with at least two anode tube inserts.

#### NOTE!

When cleaning the anode protective tube, you must observe the following:

- Only the inside of the tube may be blasted with glass beads. To prevent the rough surface of the tube interior being removed after repeated cleaning, only a glass bead beam with a final roughness of approx. 25 µm may be used.
- The surface must not be blasted with glass beads. The multi-contact strips (B) and the gas inlet nozzles (A) must be protected against scratches and mechanical influences.

Fig. 6-8, 

69 shows the anode protective tube after cleaning.

The rough surface may only be cleaned with Scotch Brite or a fine grease.

The inside of the tube is blasted with glass beads.

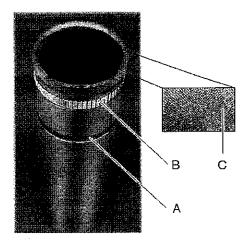


Fig. 6-8 Cleaned anode tube

- A Gas inlet nozzles
- B Multi-contact strips (electrical contact)
- C Groove on inside of tube

### Cleaning procedure (cleaning instruction)

#### 1 General cleaning

Carefully remove the coating residue on the inside of the tube with mechanical tools.
 Please note that the mechanical tools must be made of a softer material that the anode tube itself (copper) to prevent any damage to the inside of the tube.

### 2 Wet cleaning

Clean the anode tube with clean water and degrease, if necessary, with alcohol.
 Dry the anode tube with a dry, oil-free flow of compressed air.

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#### 3 Glass beads

#### 4 Ultrasonic bath

 Clean the anode tube for at least 15 minutes in the ultrasonic bath. Use distilled water at a temperature of 80 °C.

#### 5 Dry kiln

Heat the anode tube at least one hour in a dry kiln at 80 °C and store it there until you
plan to use it.

### 6 Nitrogen treatment

 Using a gas pistol, blow out the anode tube with nitrogen from the gas tank before you insert it in the APSpro

# **ACAUTION**



Fingerprints.

Fingerprints (and their deposits such as perspiration, oil etc.) on components within the process unit can impair the process.

Protect the process unit from contamination. Put on clean-room gloves before you touch cleaned components and install them in the APS*pro*.

### 6.3.3 «Gas shower» maintenance

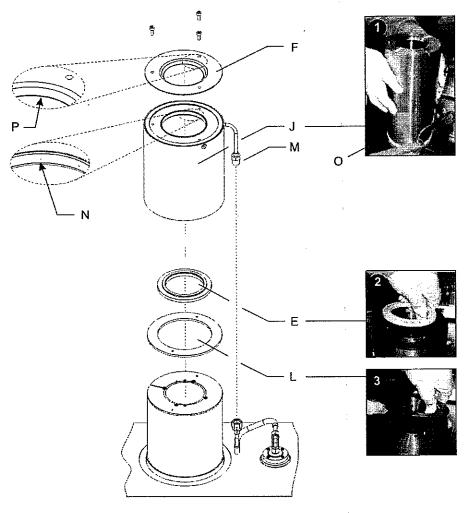


Fig. 6-9 Dismounting the APSpro gas shower

### Dismounting the gas shower and cleaning the components (see Fig. 6-9, 🗎 71)

- 1 Loosen gas connection (M) and pull the entire shielded hood (J) upwards to remove.
  - 1.1 Remove the Teflon strip (O) from the gap between the bottom of the process chamber and shielded hood. Clean the Teflon strip and check for damage and burnt spots. Replace Teflon strip if damaged!
  - 1.2 Clean the shielded hood (J) with «Scott Brite». Ensure that the gas inlet nozzles (N) are free of deposits. Check in particular that there are no burns on the edge (P) of the aluminum ring (F). If there is excessive wear and burning, replace aluminum ring.
- 2 Remove ceramic ring (E) and clean with «Scott Brite»
- 3 Remove steel ring (L) and clean with «Scott Brite»

### Mounting the gas shower

The gas shower is mounted in the reverse sequence (see Fig. 6-9, 🖹 71). Note the following procedure and instructions:

### 1 Ensure the gas shower rings are positioned correctly

■ Mount the rings (E = ceramic ring, L= steel ring) as shown in Fig. 6-10, 

72.

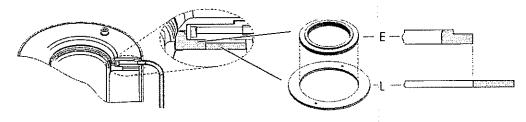


Fig. 6-10 Position of the APS rings

### 2 Mount teflon strip and shielded hood (see, Fig. 6-11, 🖺 72)

- 2.1 Clean the gap between the anode tube and process chamber.
  - Remove all particles and vacuum out the gap. Use a thin plastic attachment on the vacuum hose.
- 2.2 Insert the Teflon strip (O) into the gap between the anode tube and process chamber floor.
  - The bottom edge of the Teflon strip must be lie on the flange of the plasma source. Teflonband immer bis zum Anschlag einschieben!

Check the installation dimension (x) of the Teflon strip as follows: Measure the distance (x) between the top edge of the Teflon strip and the chamber floor. The dimensions should be as those shown in the following table, depending on which APS source is installed (with/without connection flange).

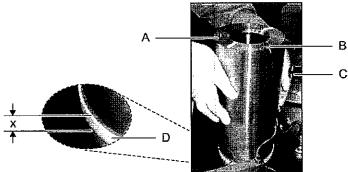


Fig. 6-11 Installation of shield hood

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- 2.3 Connect the gas shower to the gas inlet (M)
- 2.4 Connect the grounding cable (B) on the shielded hood

## 3 Make a visual check of the gas shower

Fig. 6-12, 🖺 73 shows the correctly mounted APS gas shower. When mounting, ensure that:

- the APS rings are mounted centrally as shown and not tilted.
  - F = Aluminium ring
  - E = Ceramic ring
- an even gap (X) must be kept for the gas inlet.

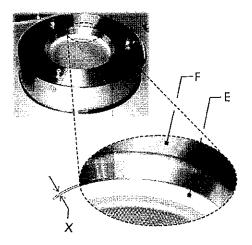


Fig. 6-12 Correct installion position

## 7 Service

## 7.1 Introduction

This chapter describes the service work that the operator can carry out on the APSpro.

## Termination of the warranty claims

### NOTE!

The internal components of the plasma source may only be serviced by Bühler Alzenau service personnel. Access to the internal components of the APS plasma source is protected by a lead seal.

See: Chapter 1.3.3 Sealing the system and warranty terms, 🖹 16.

## Safety information

## NOTE!

Before attempting to service the APSpro, the designated personnel must have read and fully understood Chapter 2 Safety, 100 21.

## **ADANGER**

High voltage and mains voltage.



The voltage for the magnetic coil, the plasma current power supply and the heater current power supply are all connected to the mains. The heater current power supply supplies the heater with high voltage. There is a danger of electrocution.

The system must be shut off and electrically discharged before any work is carried out on the APS*pro*. Ground the equipment and secure the system against restarting. Ensure that nobody is left standing in the danger area before switching the installation on again.

### Final checks

After completing the work, check that all the safety equipment has been installed and is functioning properly. Check the safety equipment especially after working on the electrical system.

# 7.2 Checking and cleaning the seal surfaces and sealing rings

## 7.2.1 Requirements

Please note the following instructions when cleaning seal surfaces and sealing rings:

- For all cleaning work, observe the Technical Rule for Hazardous Substances (TRGS) No. 900 - «Threshold limit values for air pollution at the workplace» (MAC values) dust masks, vacuuming, etc.
- When cleaning seal surfaces and sealing rings, always wear clean, lint-free gloves
- In clean rooms, always wear clean-room gloves and avoid the build-up of particles

## 7.2.2 Approved cleaning materials

For cleaning, use:

- Sand paper with a grain size of 400 or finer
- Scotch Brite
- ISO (isopropyl, propanol 2)
- Ethanol (ethyl alcohol) and acetone (propanone)
- Lint-free cloths, such as Dastex 400 series cloths

## NOTE!

Ethanol (ethyl alcohol) and acetone (propanone) must not be used for O-rings, because these substances chemically attack the rings!

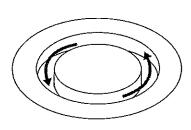
## 7.2.3 Checking and cleaning the seal surfaces and sealing grooves

Seal surfaces made of steel, stainless steel, or aluminum must be without scratches or grooves. The permissible surface roughness, regardless of the vacuum area, is set at  $R2 = 6.3 \ \mu m$ .

Scratches and grooves can be removed by sanding with sandpaper (grain size > 400) and Scotch Brite. Always sand in the direction of the metal's grinding pattern. The depth of the sealing surface must not be altered. Finally, clean the surface with ISO (isopropyl) and a lint-free cloth.

The sealing surface of the groove must also be clean. Clean the sealing groove as follows:

Remove the O-ring. Take care not to damage (scratch) the seal surface of the groove or the O-ring itself. Do not use any sharp or pointed tools (e.g. a screwdriver)!



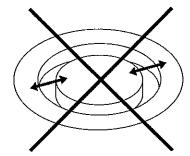


Fig. 7-1 Wiping direction in the sealing groove

## 7.2.4 Checking and cleaning the sealing rings

When in perfect condition, the rings have the following characteristics (Fig. 7-2, 12 77):

- A clean surface
- No damage (cracks, cuts, ridges or porous sections)
- A uniform diameter
- No sticky spots

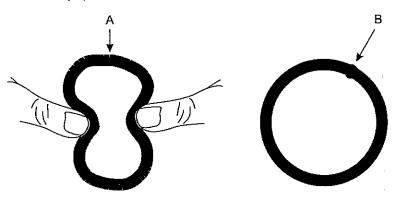


Fig. 7-2

Checking an O-ring

- A Cracks, cuts, porous sections
- B Swelling, bulge

Oil, grease, dust, talcum, etc. are removed from the rings in the following steps:

- 1 Pull the ring through a dry cloth to remove the larger particles of dirt
- 2 Pull the ring through a cloth soaked in ISO to remove the finer dirt

## NOTE!

The following two steps must not be carried out if the O-ring can become hotter than 100 °C. The materials must not exceed the following temperatures:

NBR O-ring (Perbunan): 100 °C FPM O-ring (Viton): 200 °C

Leybold LITHELEN vacuum grease: 150 °C

Grease the ring lightly (very thin film) with vacuum grease (Leybold LITHELEN)

4 Pull the ring through a dry cloth, so that the surface and pores of the ring remain only lightly moistened by the vacuum grease

### NOTE!

Dust particles stick to lightly greased rings very easily. Therefore the ring should be installed immediately and not first put down for any period of time.

The ring must protrude above the groove (relative to the seal surface) at a constant distance around the entire circumference. See Fig. 7-3, 12 78. Depending on the thickness of the ring, the values are as follows:

Ring thickness	Protrusion (x)
5 mm	0.8 mm
6 mm	1.1 mm
8 mm	1.9 mm

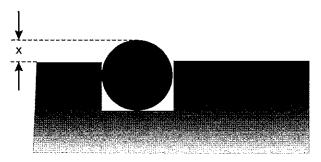


Fig. 7-3 Protrusion (x) of a sealing ring above the sealing surface

## 7.3 Replacing the heater and cathode

## 7.3.1 Removing the heater-cathode unit

The heater and cathode can only be replaced as a single unit. A special change device is available for this purpose. It is not possible to remove and install the heater and cathode without this special tool (see: Fig. 7-4, 1979). For removal, further tools/devices are required, please have these on hand:

- Change device
- Tweezers (long)
- Retaining clamp

### NOTE!

- The heater is made of graphite which breaks very easily. Therefore be very careful when handling the heater.
- 1 Turn the rotary knob on the change device to the «UNLOCK» position (A).
  - Only in the «UNLOCK» position can the heater-cathode unit be placed into the APS.

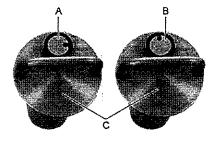


Fig. 7-4 Preparing the change device

- A Rotary knob position <UNLOCK>
- B Rotary knob position <LOCK>
- C Marking <DOOR>
- 2 Insert the change device carefully into the plasma source as far as it goes.
  - The arrow marked «DOOR» (C) must point in the direction of the process chamber door. The heatercathode cannot be fitted unless the plasma source is in this position.
  - Carefully use weight (1-2 kg) for inserting the change device. Insert the device completely. Do not let the device fall down by its own weight.

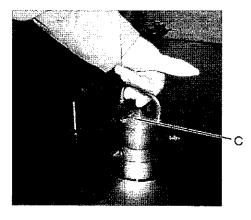


Fig. 7-5 Inserting the change device

- 3 Turn the rotary knob on the special tool to the «LOCK» position (B).
  - The heater-cathode is gripped by the tool and held in place.
- 4 Pull the change device out of the plasma source. Place the entire unit on its U-profile section (A).
- 5 Turn the rotary knob to the «UNLOCK» position
  - The change device opens (B) The secured heater-cathode unit is unlocked (D)
- 6 Pull the heater-cathode unit (D) out of the change device in a forward direction.

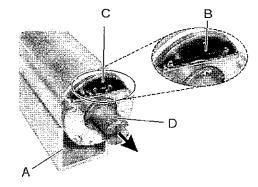


Fig. 7-6 Change device

- A U-profile section
- B Lock open, rotary knob position <UNLOCK>
- C Lock closed, rotary knob position <LOCK>
- D Heater-cathode unit
- Disassembling the heater-cathode unit
   Gently press the connecting legs of the heater (D) together with the fingers and, at the same time, pull the cathode (A) off of the heater (see illustration).
- 8 Pull both bornitride rings (B, C) off the heater.

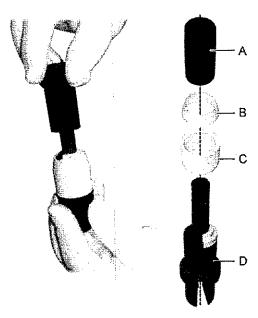


Fig. 7-7 Disassembling the heater-cathode unit

## 7.3.2 Servicing the heater-cathode unit

Due to the thermal and process-specific load, the heater-cathode unit is subject to varying degrees of strain.

To ensure the proper functioning of the plasma source, the individual components of the heater-cathode unit must be checked on a regular basis. The check procedures are:

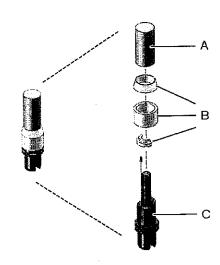


Fig. 7-8 Heater-cathode unit

## 7.3.2.1 Servicing the cathode

- 1 Check the cathode for loss of material
  - The cathode wears particularly strong because of the material it continuously loses in the plasma process.
  - The service life of the cathode is directly dependent on the operating conditions (plasma output) and the wear caused by them. The average downtime is about 50-80 operating hours.

### Characteristics

A severely worn cathode has the following characteristics:

- Cathode edges (A) are rounded off in a conical shape
- There are recesses and grooves (caused by erosion) on the cathode face (B). The cathode may become gray in colour.

## Procedure:

Replace cathodes that exhibit a considerable loss of material.

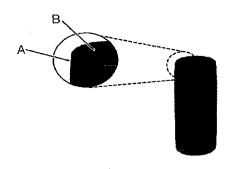


Fig. 7-9 Typical signs of cathode wear

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- 2 Check the cathode for discolouration (cathode is «poisoned»)
  - A new or usable cathode (A) has a violet colour. If it is exposed to oxygen or high temperatures for prolonged periods, it will turn orange in colour (B). This is referred to as a «poisoned» cathode. The plasma source cannot be operated with a «poisoned» cathode.

## Characteristics

A "poisoned" cathode has the following typical characteristics:

- The ability of the cathode to emit electrons is greatly reduced. The parameter values for the coil current and the discharge current increase.
- The cathode becomes increasingly orange in colour.
- Bad or no plasma ignition is possible.

### Procedure:

Regenerate the cathode through a condition procedure so that is can be reused, see: Chapter 7.4.2 Cathode <Regeneration>,

1 92

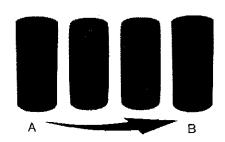


Fig. 7-10 Cathode status

- A Process-capable
- B Poisoned

## 7.3.2.2 Servicing the graphite heater

- 1 Check the heater for damage, loss of material and deposits.
  - 1.1 Check the graphite for cracks and loss of material. A loss of material can be especially detected if the area (A) begins to taper off. Furthermore a loss of material becomes noticeable if there is an increase in the heater resistance.

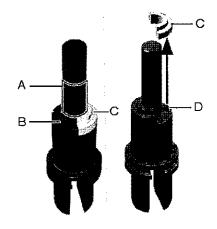


Fig. 7-11 Heater

- 1.2 Check the electrical insulation (C) for signs of cracking. A defective insulation must be replaced.
  - The insulation is attached onto a graphite pin (D) and can be pulled off in an upward direction. Be very careful, as the graphite pin can easily break.
- 1.3 Check the heater for deposits. The area (B) in which the heater makes an electrical contact with the cathode in particular must be free of deposits. Clean the dirty areas with a lint-free cloth.

## 7.3.2.3 Servicing the «bornitride insulators» on the heater

- Check the bornitride rings for signs of wear, ruptures and cracking.
  - The conical bornitride ring on the upper edge (A) is particularly susceptible to rupturing. Such a bornitride ring must always be replaced, as otherwise the area between the heater and the cathode can produce dangerous electrical arcs.
  - Therefore make sure that the top bornitride ring in the installation position (B) is flush against the edge of the heater.

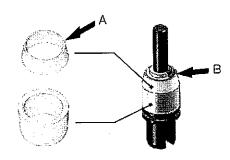


Fig. 7-12 Bornitride rings on the heater

2 Clean both bornitride rings with a lint-free cloth or with fine-grade sanding paper (>400).

### NOTE!

Bornitride rings are ceramic parts and must never be cleaned with a liquid solvent such as alcohol! Ceramic parts are porous and absorb solvents. There is a danger of the solvent emitting gas when in a vacuum.

## 7.3.3 Servicing the «ceramic insulators» on the heater socket

The ceramic insulators positioned on the heater socket must also undergo regular inspection. To do this, they must be removed from the plasma source.

## NOTE!

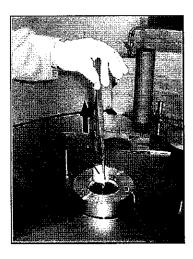
Before removing the ceramic insulators, first:

- Remove the anode protective tube. See Chapter 6.3.2 Anode protective tube, 🖹 67
- Remove the heater-cathode unit. See Chapter 7.3.1 Removing the heater-cathode unit, 

   79

Take the ceramic insulators out of the plasma source.

- First take the double-groove insulator (Pos. A) out of the plasma source using the long tweezers.
- Then take out the bornitride ring (Pos. B) out of the plasma source using the retaining clamp.
  - Guide both ends of the retaining clamp into the drill holes of the ceramic ring, gently squeeze the retaining clamp.
  - Gently press the retaining clamp together and pull out the ceramic ring from the combustion chamber.



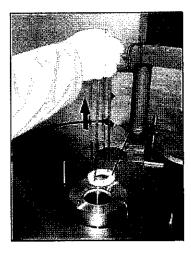


Fig. 7-13 Take the insulator out of the plasma source

- A Tweezers with double-groove insulator
- B Retaining clamp with bomitride ring

## Checking/Cleaning the ceramic insulators

- 3 Check the ring (A,B) and the small plate (C) of the double-groove insulator for ruptures and cracks.
  - · Replace an damaged parts found.
- 4 Clean all ceramic parts with a lint-free cloth or with fine-grain sanding paper (<400).

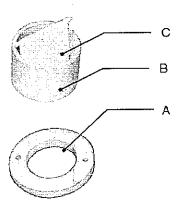


Fig. 7-14 Insulators of the heating receptacle

## NOTE!

Ceramic parts must never be cleaned with a liquid solvent such as alcohol! Ceramic parts are porous and absorb solvents. There is a danger of the solvent emitting gas when in a vacuum.

## Cleaning the heater receptacle

5 Carefully vacuum the inside of the plasma source (A) again. Make sure that the gap (B) between the receptacles of the heater connecting legs and the bearing surface (C) for the ceramic ring are completely free of dirt and dust. Use a thin plastic nozzle attachment on the vacuum cleaner.

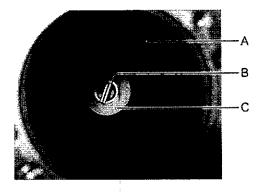


Fig. 7-15 View inside the plasma source, heater receptacle

Inserting the ceramic insulators

The ceramic insulators are installed in the reverse order of the removal procedure.

## NOTE!

Always use the retaining clamp when installing the bornitride ring to ensure that the ring is correctly inserted, see Fig. 7-13, 

84. Since the drill holes in the ring are only accessible from one side, it is very difficult to remove an incorrectly inserted ring.

Verify the correct position of each part after installation by using a flashlight.

Make especially sure that the parts of the bornitride double-groove insulator are in the proper position.

- The top edge of the ring (B) must be flush with the top side of the heater receptacle (C)
- The small plate (A) must be fully inserted in the gap of the heater receptacle. This is the case if the isolating plate is correctly seated in both grooves located in the ring.

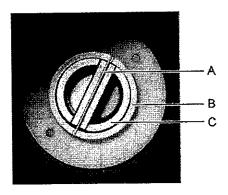


Fig. 7-16 Insulator on the heater receptacle

7.3.4

The heater and cathode can only be replaced or installed as a single unit. The unit is installed in the reverse order of the removal procedure, see Chapter 7.3.1 Removing the heatercathode unit, @ 79

### NOTE!

The heater is made of graphite which breaks very easily. Therefore be very careful when handling the heater.

- 1 Assemble the heater-cathode unit as shown in Fig. 7-7, 8 80. When plugging in the cathode, gently press the connecting legs of the heater together with the fingers.
- 2 Gently pull the connecting legs of the heater apart so that the cathode is clamped.

Installing the heater-cathode unit

- 3 Insert the heater-cathode unit into the change device.
  - One of the connecting legs of the heater is marked by a notch (B). This notch must be located on the side of the change device marked by «IS» (A). This ensures that the installed insulator on the heater is on the correct side of the heating circuit.
  - Make sure the heater-cathode unit is in the correct installation position in the change device, see Fig. 7-17, **87**
- 4 Turn the rotary knob on the change device to the «LOCK» position.
- 5 Insert the change device into the plasma source. Make sure that the arrow labelled «DOOR» points to the process chamber door and that the handle is aligned parallel to the process chamber, see Fig. 7-18, 87.
- Lower the change device until there is a mechanical resistance.
  - When the heater receptacle is correctly adjusted, the inside edges of the heater connecting legs lie flat on the heater receptacle. The heater connecting legs can be slid into the heater receptacle by applying slight pressure on the special tool. This forms the electrical contact between the heater and the receptacle.

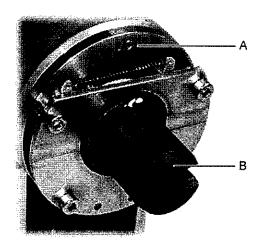


Fig. 7-17 Installation position of the heater-cathode unit in the change device



Fig. 7-18 (Inserting the heater-cathode unit with the change device

87

- If the heater-connecting legs can be inserted without any tangible resistance, the heater receptacle is incorrectly adjusted. This means that there is either no contact between the heater and the receptacle or the contact is poor. In this case, the heater receptacle must be set with a special tool.
- 7 Turn the rotary knob on the special tool to the «UNLOCK» position.
- 8 Carefully pull the special tool out of the plasma source.
- 9 Check with a flashlight whether the cathode and the bornitride rings are in the proper position.

89

## 7.4 Conditioning the cathode and the heater

Conditioning the heater and the cathode is intended to ensure that the graphite heater and/or poisonous cathodes are ideally prepared for process operation. The conditioning procedure consists of two different but related steps:

- Degasing procedure
- Regeneration procedure

## Why do the heater, cathode and ceramics have to be degased?

The heater, cathode and ceramics must be degased whenever their surfaces are strongly covered with gas atoms or molecules. This always happens when the heater and/or cathode are exposed to the atmosphere for prolonged periods, for example

- When inserting a new heater (original packaging)
- Inserting a new cathode
- Inserting new ceramics (bornitride)
- During a long operating time for the vapour deposition installation

## When is it necessary to regenerate a cathode?

The cathode must be regenerated whenever it is «poisoned». A cathode is considered «poisoned» when its surface undergoes a chemical change. This drastically reduces the ability of the cathode to emit electrons. Consequently, you will be unable to effectively operate the plasma source.

A new cathode is always «poisoned», since it usually has been exposed to the atmosphere for a long time. In addition, a cathode will always become «poisoned» whenever new parts are added to the plasma source and heated. When one of these components is heated to the target temperature after installation, gasses will be release from the component material or from its surface. The emitted gases react with the cathode material, and the cathode is then «poisoned». In the case of the plasma source, the components are the:

- Heater
- Bornitride bushing
- Bornitride ring

The cathode can also be «poisoned»

- if operating conditions for the plasma are unfavourable, such as when plasma output is low (< 2 kW) and, at the same time, the O<sub>2</sub> flow is high (> 50 sccm).
- if the system is vented too soon (APS is still hot).



## 7.4.1 «Degasing» heater and cathode

The degasing procedure is carried out by gradually increasing the heating current while the vacuum pressure increase is monitored at the same time. In the process, the heating current is incremented along a start-up ramp, from its initial value of 60 A to 200 A. At the end of the heating phase, the <heater output> control is switched on and the plasma source is operated with a set point of 1.8 kW. The schematic diagram Fig. 7-19, \$\mathbb{B}\$ 90 shows the typical curve of the <gas emission procedure>.

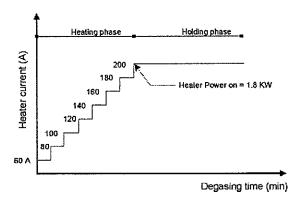


Fig. 7-19 Process diagram for the degasing procedure

## «Degasing»

For both visualization systems «SyrusPro» and «OptiControl» the degasing procedure is carried out in the same way. In the instruction the software screens are named for both visualization systems.

### 1 Evacuating the process chamber

- 1.1 Pump down the process chamber to a pressure less than 1.0 x 10<sup>-5</sup> mbar.
  - · If present, switch on the Meissner trap
- 1.2 Switch on the cooling circuit

## 2 Adjust/set the start parameters for the «heater»

SyrusPro visualization: Enter the following set point values on screen < Preparation/APS/Fleater>
Constants Machine
Constants

OptiControl visualization: Enter the following set point values on-screen <Service
Functions #3/APS>

- IS	
Parameter	Set point
Heater current	60 A ( WISCL 200)
Heater mode (Plasma mode)	constant long controlled
Heater warm-up ramp-time	10s Was 1063 (was not)
Heater warm-up wait time	200 s

Tab. 7-1

Starting parameters of the APSpro for the <degasing procedure>



## 3 Switching on the APS heating current

- 3.1 Actuate the [Heater ON] switch.
  - The heater is then heated along the set time ramp (10 s) to the set heating current set point (60 A).

### NOTE!

After switching on the heater, the vacuum pressure will first begin to rise in the process chamber. Wait until the peak value of the vacuum pressure has been reached before starting the degasing procedure.

## 4 Carrying out the degasing procedure

- 4.1 Increase the value «heater current» in steps of 20 A.
  - The heating current is entered incrementally until you reach 200 A
  - After each input step, the pressure in the process chamber should be observed before continuing.
  - If the chamber pressure does not rise, wait approx. 2 minutes and then increase the heater current by a further 20 A.
  - The rise in pressure (peak value) in the process chamber must not exceed 1.0∞10<sup>-4</sup> mbar, otherwise there is risk that the cathode will be «poisoned».

### NOTE!

Should the vacuum pressure exceed 1.0  $\infty$  10<sup>-4</sup> mbar in the process chamber during the degasing procedure, then:

- Immediately switch off the heater
- Wait about until the vacuum pressure decreases again to under 1.0 ∞ 10<sup>-4</sup> mbar before switching on the heater again without changing the parameter value settings
- Select smaller steps (e.g. 10 A) as needed when increasing the heating current

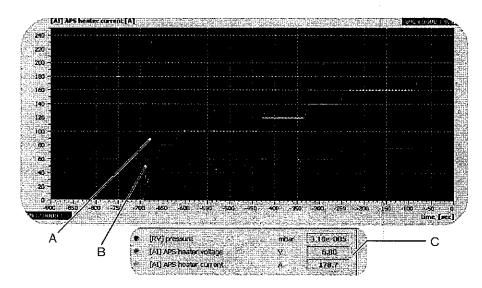


Fig. 7-20 procedure

Trend display (SyrusSpro Visualization): Progress of the heating current, voltage and pressure during the degasing

- A Vacuum-pressure progress during the degasing procedure
- B Progress of the incremental increase of the neating current
- C Current actual value during the degasing procedure

Once the value «actual heater current» has reached 200 A, the heater control is switched on:

Set the parameter «Heater Power» to 1.8 kW - Machine constants to, not 4.2

Switch the heater mode (Plasma mode) to «Controlled» 4.3

The heater current will be set automatically according to the default «Heater power» of 1.8 kW.

To attain optimum conditioning, the regeneration procedure for the cathode should be take place immediately afterwards, see: Chapter 7.4.2 Cathode <Regeneration>, 🗎 92

#### 7.4.2 Cathode < Regeneration >

The cathode is burned off as a part of the conditioning process of the heater-cathode unit. Its purpose is to regenerate the ability of new cathodes or ones already poisoned to emit electrons so that they can re-used. Section «When is it necessary to regenerate a cathode?», 1 89

## NOTE!

The regeneration procedure is always carried out with the default heater output and by supplying gas when the plasma is ignited. Particularly when installing new parts (cathode, heater, ceramic parts), it is crucial that the parts are thoroughly degased prior to the regeneration procedure, see Chapter 7.4.1 «Degasing» heater and cathode, 🖹 90.

## «Regeneration»

For both visualization systems «SyrusPro» and «OptiControl» the regeneration procedure is carried out in the same way. In the instruction the software screens are named for both visualization systems.

- Setting /Checking the start-parameter values for the <Heater>
  - SyrusPro visualization: Enter the following-set-point-values\_on\_screen <Prepara-</li> tion/APS/Heater>-
  - OptiControl visualization: Enter the following set point values on screen <Service Functions 13/APS>

Parameter	Value
Heater power	1.8 kW
Heater mode (Plasma mode)	controlled
Heater warm-up ramp-time	10 s
Heater warm-up wait-time	200 s

Tab. 7-2 Starting parameters of the APS heater for the <regeneration procedure>

## Switching on the APS heating current

- Actuate the [Heater ON] switch.
  - Once a chamber pressure of  $1.0 \times 10^{-5}$  mbar has been reached, the heater is then heated to the set heating current output of (1.8 kW).

- SyrusPro visualization: Enter the following set points values on screen <Preparation/APS/Param>
- OptiControl visualization: Enter the following set point values on screen <Service Functions 12/APS>

Value
Constant
Bias v/ coil c
0.8 A ( 1. % )
15 sccm ( , ~ )
0 sccm ( ( 6 )
8A (15)

Tab. 7-3

Starting parameters for plasma mode <regeneration>

E 7.5A

## 4 Switching on the plasma source / Igniting the plasma

- 4.1 Actuate the [Plasma ON] button.
  - The coil current and the gas supply are switched on and the plasma ignites.
  - When the plasma is initially ignited, a discharge current of less than 10 A may be present and the plasma may then flicker. This may trigger the undervoltage detector which switches off the source if arcing occurs. In this case, the system control displays an error message.

Acknowledge the error message and switch the source back on. Repeat the procedure until the discharge current of 10 A can be held constant.

## NOTE!

Before beginning with the regeneration procedure, ensure that the following operating parameters are safely attained:

- Actual value display «Discharge current» greater than 8 A
- Actual value display «Discharge voltage» smaller than 100 AV

5 Carry out the regeneration procedure

■ The <egeneration procedure is carried out by entering parameter values step by step and the respective stop phases. The order of the entry steps to be executed are listed in the table below Tab. 7-4, 

95. The chronological sequence of operations of the entry steps and the process parameters can be monitored - exemplarily for SYRUSpro- on the screen <Trend> (see Fig. 7-22, 

96).

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Step ⊫	Activity/Description
5.1	Increase the «coil current» to 1 A  Wait until an «actual discharge voltage» of less than 100 V is displayed. Once this value is attained, proceed to the next entry.
5.2	Minimise «gas flow #1» to 12 sccm (see Fig. 7-22,   96, pos. B)  ■ Wait until an «actual discharge voltage» of less than 100 V is displayed.  Once this value is attained, proceed to the next entry.
5.2	Increase the «discharge current» to 20 A (see Fig. 7-22,   96, pos. C)  Wait until an «actual discharge voltage» of more than 18 A is displayed.  Wait until an «actual discharge voltage» of less than 100 V is displayed.  Once this value is attained, proceed to the next entry step.
5.3	Increase the «discharge current» to 30 A  Wait until an «actual discharge current» of more than 28 A is displayed.  Wait until an «actual discharge voltage» of less than 100 V is displayed.  Once this value is attained, proceed to the next entry step.
5.4	Increase the «discharge current» to 40 A  Wait until an «actual discharge current» of more than 38 A is displayed.  Wait until an «actual discharge voltage» of less than 100 V is displayed.  Once this value is attained, proceed to the next entry step.
5.5	Increase the «discharge current» to 50 A  Wait until an «actual discharge voltage» of more than 48 A is displayed.  Wait until an «actual discharge voltage» of less than 100 V is displayed.  Once this value is attained, proceed to the next entry step.
5.6	<ul> <li>Increase the «coil curren» in steps of 0.2 A (Fig. 7-22, ₱ 96/Pos. D)</li> <li>■ Wait until an «actual discharge voltage» of 100 V is displayed.</li> <li>■ Once the «actual discharge voltage» of 100 V has been reached, then wait until the value «actual bias voltage» has a value 15 V larger (ca.115 V) than the value displayed for the «actual discharge voltage». Afterwards, wait approx. 15 minutes.</li> </ul>

## NOTE!

Once the discharge voltage has fallen well under 100 V, increase the coil current steps of 0.1 A, until the discharge current has reached about 100 V, and the bias voltage is again at least 15 V higher than the discharge voltage.

	increase the «coil curren» in steps of 0.1 A
5.7	<ul> <li>Wait until an «actual bias voltage» of 150 V is displayed. Once this value is</li> </ul>
	attained, proceed to the next entry.

Step	Activity/Description				
5.9	<ul> <li>«Set the bias voltage» to 150 V and switch on APS <controlled> mode see</controlled></li> <li>(Fig. 7-22, 9 96 /Pos. E)</li> <li>Enter the parameter value under «APS bias voltage» to 150 V and then switch over to the operating mode «controlled» under the function «APS plasma mode»</li> <li>The specified bias voltage is now controlled by the coil current.</li> <li>Afterwards, wait approx. 15 - 30 minutes.</li> </ul>				
5.11	Check the plasma parameter:  At the end of the regeneration Pos. B must have been reach fully run regeneration proced:  «Actual discharge current» is «Actual coil current» less that paramile continuity is paramiled to config.  Preparation  Preparation	on procedure the actual ched. Important target dure are:	al values displayed under parameters for a success-		
	APS bise voltage:  APS discharge to base  APS discharge power	V 150.0 V 0.0 KW	1500 1600 1 643		
	Fig. 7-21 Example screen «Preparat	tion Aps/Param> SyrusPro	visualization		

Tab. 7-4 Procedural steps for the <regeneration procedure>

## Chronological sequence diagram for the «regeneration procedure»

Fig. 7-22, ⓐ 96 shows exemplarily the monitor screen <Trend> of the SyrusPro plant control. The screen shows the visual progress (trend) of the parameter values during the regeneration procedure.

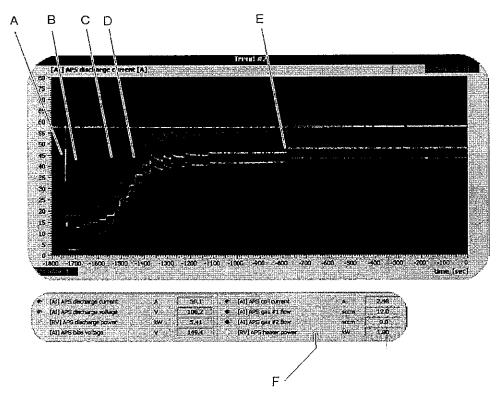


Fig. 7-22 Trend display: Chronological sequence of the entry parameters for the <regeneration procedure>

- A Plasma ignites
- B Reduction <gas flow#1> to 12 sccm
- C Increase of the <discharge current> to 50 A
- D Increase <coil current> until a <bias voltage> of 150 V is displayed
- E <Set bias voltage> to 150 V / Switch plasma source to <controlled> mode
- F Current actual value display (target parameters) at the end of the regeneration procedure

## 7.5 Returning the product

## 7.5.1 Removing the plasma source

The procedure for removing the plasma source can be broken down into the following steps in the order indicated:

- 1 Interrupt the supply of current and media, see: Chapter 7.5.1.1 Interrupting the supply of current and media, 97.
- Remove the anode tube insert and bornitride bushing, see: Chapter 6.3.2 Anode protective tube, 

  67
- Remove the heater and cathode, see:
  Chapter 7.3.1 Removing the heater-cathode unit, \$\exists 79\$
- 4 Remove the shielded hood, see: Chapter 6.3.3 «Gas shower» maintenance, 

  71
- Remove the plasma source, see:
  Chapter 7.5.1.3 Removing the plasma source from the process chamber, \$\mathbb{B}\$ 100

### NOTE:

You must follow the order of the steps precisely!

## 7.5.1.1 Interrupting the supply of current and media

When working with the APS*pro*, there are potential dangers present for the maintenance and service personnel that are described in Chapter 2.6.2 Danger areas for maintenance and service personnel, ⓐ 27. To safeguard yourself against such dangers, the supply of current and media must be cut off prior to servicing the APS*pro*.



The supply of current and media of the APS*pro* is regulated via the control of the vapour deposition installation. By referring to the documentation that came with your vapour deposition installation, familiarize yourself with the interfaces and related switching elements and how they are operated.

- 1 Shut off the supply of electrical power to the vapour deposition installation with the main power switch.
- Shut off the supply of water to the APSpro and blow out the water circuit to which the APSpro is connected.
  - Always shut off the inlet first and then the outlet of the water supply.
  - If your installation is not equipped with an integrated blow-out unit, pull off the water hoses and blow them out with compressed air.

Translation: English, Edition: V4.1\_06/2015\_en

- 3 Shut off the compressed air and release the pressure.
- 4 Shut off the supply of gas and release the pressure.

## 7.5.1.2 Disconnect the supply connections

## NOTE!

Before you disconnect the supply connections, check that the cables and hoses are adequately labelled so that you can re-connect them properly. Replace missing or illegible labels.

## **ADANGER**



High voltage and mains voltage.

The voltage for the magnetic coil, the plasma current power supply and the heater current power supply are all connected to the mains. The heater current power supply supplies the heater with high voltage. There is a danger of electrocution.

Switch off the devices and disconnect them from the power supply before you begin work on the device connections.

franslation: English, Edition: V4.1\_05/2015\_en

- 1 Release the clamping rings on both of the operating gas connections (A) and (B). Each gas line is connected to the connecting nozzle on the APS by means of an O-ring with centring ring and inserted plastic tube. Remove these components and
  - Check if they are in proper working order
  - Replace any damaged components with new ones
  - Clean the dirty components, see Chapter 7.2 Checking and cleaning the seal surfaces and sealing rings,
     76
  - Store the components at a suitable location for installation in the APSpro.
- Release the screws on the cooling water inlet (H) and outlet (C) of the anode tube and pull off the water hoses.
- 3 Release the screws on the cooling water inlet (J) and outlet (F) of the heater
- 4 Release the screws on the heater current connections (D), the discharge current connection (G) and the coil current connection (L).
- Remove the three screws, that fasten the base plate (M) to the three base plate holders (K) and put the base plate aside.

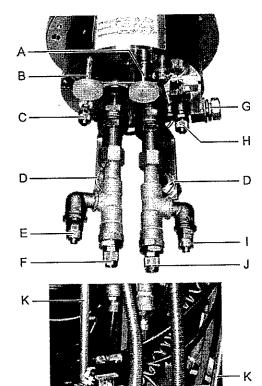


Fig. 7-23 APSpro connections

- 6 Unscrew the three base plate holders (K).
  - The base plate holders are screwed on to the flange of the APSpro. These connections can be released using a screwdriver that is inserted into the existing drill holes in the base plate holders.
- 7 Release the screws on the cooling water inlet (I) and outlet (E) of the heater and pull off the U-shaped water hose.

₽

## 7.5.1.3 Removing the plasma source from the process chamber

## 

Heavy APSpro.



The plasma source has a total weight of about 20 kg. When removing the plasma source, there is danger of injury.

At least two persons are required to remove the plasma source. Use only the tools and devices described in this chapter. Avoid the area underneath the plasma source as far as possible and always wear safety gloves.

### NOTE!

If using a crane, use only ropes to fasten the plasma source. Never use chains! Chains can damage the process chamber.

- 1 Tighten the carrying handle (B) on the top side of the anode tube (C).
- 2 Fasten a carrying strap to the carrying handle (A).
  - The plasma source can also be attached to a suitable crane.
- 3 Hold the plasma source securely using the carrying strap on the carrying handle while a second person removes the four fastening screws on the flange.
- Working together, carefully lower the plasma source and place it on a suitable support.
- 5 Remove the carrying strap and unscrew the carrying handle.

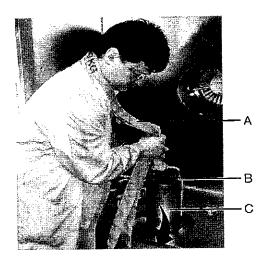


Fig. 7-24 Securing the plasma source

## 7.5.2 Packing the plasma source for transport

The plasma source must only be transported in its original packaging. See Fig. 7-25, 🖹 101.

### NOTE!

Improper transport will damage the plasma source.

- Pack the plasma source in the original packaging. Fill the empty spaces with suitable filling materials.
- Observe the transport symbols

If the plasma source is not properly packed for transport, all guarantee claims will be voided.

## **ACAUTION**



Shipping contaminated products

All products shipped should free of harmful substances (e.g. radioactive, toxic, caustic or microbiological substances). The operator is obligated to properly clean and declare the product prior to shipping.

Enclose the contamination statement with the product. See: Chapter 7.5.2.1 Contamination declaration, 

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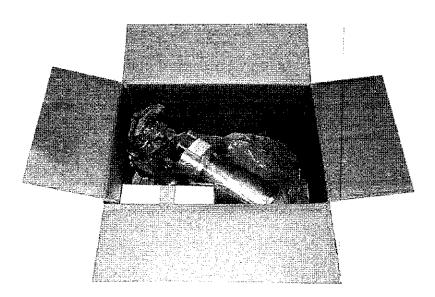


Fig. 7-25

Plasma source in the original packaging

## 7.5.2.1 Contamination declaration

Included with the product are one or more forms for the contamination declaration for parts to be sent to Bühler Alzenau for inspection or repair purposes.

### NOTE!

Bühler Alzenau will not accept any components that are not accompanied with a valid form. Leybold Optics reserves the right to handle all products that have not been clearly identified as «free of harmful substances» as follows:

- The components will be decontaminated, the costs of which will be charged to the sender.
- The product will be rejected and returned to the sender

Please enclose the «Contamination statement» form, completed in full and signed, with the product.

### NOTE!

The «Contamination statement» form for Bühler Alzenau products can be requested from Bühler Alzenau GmbH / Alzenau, Customer Support Department by phone or by fax.

Phone no.: +49 - 6023- 437 Fax no.: +49 - 6023- 453

## 8 Appendix

## 8.1 Material Safety Data Sheet «Lanthanum boride»

## MATERIAL SAFETY DATA SHEET Lanthanum boride, cathodes

Page: 1

CERAC, inc. 1316 W. St. Paul Avenue Milwaukee, WI 53233

Printed: 12/29/2005 Revision: 07/06/2001

Date Created: 05/09/1990

	l - Product Id	lentification	
Product Name:	Lanthanum baride, cathod	zs	
Product Cade:	L-MSDS0100		
Reference #:	12008-21-8		
Manufacturer Information			
Company Name:	CERAC, inc.		
Emergency Contact:	CHEMTREC	(800)424-9300	1
Alternate Emergency Contact:	CERAC, inc.	(414)289-9800	:
Chemical Family:	Metal boilde		

CAS Number: Formula: Molecular Weight:

12008-21-8 LaB6 203-77

Synonyms

Lantasnum coride, lanthanum hexaberies

II - Hazaro	lous Ingr	edients/Ide	entity Inform	ation	· · · · · · · · · · · · · · · · · · ·
Hazardous Components (Chemical Name)	CAS #	Percentage	OSHA PEL	ACGIH TLV	Other Limits
Lanthanum boride	12008-21-8	0.0 -160.0 %	NE	NE	NE
See SECTION IX-ADDITIONAL COMMENTS     FOR COSHH Regulations	NA	0.0 -100.0 %	No daţa.	No data.	No dala.

Hazardous Components (Chemical Name)	CAS#	Sec.302 (EH5)	Sec.304 RQ	Sec.313 (TRI)
Lanthanum borkle	12008-21-8	No	No	No
2. See SECTION IX-ADDITIONAL COMMENTS	NA	No	No	i No
FOR COSHH Regulations				· '

[X] Solid

III - Physical/Chemical Characteristics

Physical States: [ ] Gas [ ] Liquid
Melling Point: 2210.00 C (4010.0 F)

Boiling Point: N.A.

Specific Gravity (Water = 1): 4.76

 Vapor Pressure (vs. Air or mm Hg):
 No data.

 Vapor Density (vs. Air = 1):
 No data.

 Evaporation Rate (vs. Butyl Acetate=1):
 No data.

 Solubility in Water:
 insolubic

Other Solubility Notes: insoluble in hydrochloric acid

Percent Volatile: N.A.

Appearance and Odor

Purple powder, no edor.
Mikily air sensitive, will pick up water and hydrolyze

Translation: English, Edition: V4.1\_06/2015\_en

## MATERIAL SAFETY DATA SHEET Lanthanum boride, cathodes

UEL: NA

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## IV - Fire and Explosion Hazard Data

Flash Pt

N.A. Method Used: NON-FLAMMABLE

Explosive Limits:

LEE NA

NA

Autoign/Llon Pt:

No data.

Extinguishi ng Madia

USE: Not applicable. Use suitable extinguishing media for surrounding materials and type of fire.

Special Fire Fighting Procedures

FireFig.Ros must wear full face, self-contained breathing apparatus with full protective clothing to prevent contact with skin and eyes. Fumes from fire ore hezordos. Isolate runni? to prevent environmental pollution.

Unusual Fire and Explosion Hazards

Contact with acids may emit flummable hydrogen gas and volatile boranes.

## V - Reactivity Data

Stability:

Unsiable[ ] Stable[X]

Conditions To Avoid - Instability

Notic

Incompatibility-Materials To Avold

Strong soids and fluorine Hazardous Decomposition Or Byproducts

Hydrogengas, Lu2O3 and exides of boron. Hazardous Polymerization:

Will occur [ ]

Will not occur [X]

Conditions To Avoid - Hazardous Polymerization

None

## VI - Health Hazard Data

Health Hazards (Acute and Chronic)

To the best of our knowledge the chemical, physical and toxicological properties of Lamiltzourn baride have not been theroughly intrestigated and recorded.

Lambaroum at considered a rate earth metal. These metals are moderately to highly toxic. The symptoms of toxicity in animals of the rate earth elements include withing, atexia, inhered respiration, walking on the loss with arched back and sudation. Oral or intraperitorical doors of 5 to 10 g/kg of dysprosium oxide had an pathological effects in mice and delily doors of 2 g/kg were harmless. Rare earth oxides are much less toxic that chlorides or citrates. The rate earth elements exhibit few toxicity by ingestion exposure. However, the imagenitorical route is highly toxic while the substancous route is poison to moderately foxic. The production of skin and long groundoms after exposure to their requires extensive protection to prevent such exposure. (Sax, Dangerous Properties of Industrial Muterials, eighth edition)

Boron compounds are very toxic and disrefere considered an industrial poison. Beron is one of a group of elements, such as Ph. Mn. As which affects the actival nervous system. Hunon poisoning causes depression of the circulation, persiment varniting and diarrhea followed by profound shock and const. The temperature becomes subnatural and a scarlesing-form rush may cover the entire body. (Sax, Dangerous Properties of Industrial Materials, eighth edition)

### INHALATION:

Acute: May cause inflation to the apper respiratory system and nuccus menteranes.

Chronic: May cause writing, atoxia, lobored respiration, preumonocomiosis, walking on toes with suched book, sedanon, preumonocasiesis, hencefolimentia and lung grasulonu.

INGESTION:

## **MATERIAL SAFETY DATA SHEET** Lanthanum boride, cathodes

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Acute: May cause gastraintestinal disturbances.

Chean it: May cause burns paisoning and act as a blood anticoogulate.

SKIN: Auto: May cause initation.

Chromic: No cinonic health effects recorded

EYE:

Acaret May cause inflation.

Chronic No chronic health effects recorded.

TARGETORGANS: May affect the blood, lungs and central nervous system.

Carcinoperticity:

NTP? No IARC Menographs? No

Sec "Section II"

LD SO/LC 50:

No toxicity data recorded.

Signs and Symptoms Of Exposure

Recommended Exposure Limits:

INHALATION: May cause a red, dry throat, congestion, success, and enugling.

INGESTION: Boton poisoning may cause: a below normal temperature, poor circulation, vomiting and distribute. May affect the congulation time of the olood-

OSHA Regulated? No

SKIN: May cause redness, burning and deling.

EYE: May cause redness, burning, itching and watering. Medical Concilions Generally Aggravated By Exposure

Pre-existing respiratory disorders. Emergency and first Aid Procedures

INHALATION: Remove victim to fresh air; beep warm and quiet; give expgen if breathing is difficult and seek medical ancertion if symptoms persist.

INGESTION: Give 1-2 glasses of milk or water and budges remaining seek medical attention if symptoms persist. Never induce vomiting or give anything by month to an unconscious person.

SKIN: Remove communitated alcaling; brush moterial off ikin; wash affected area with mild scap and water; seek medical attention if symptoms perkist.

EYE: Flush eyes with lukewarm water, lifting upper and lower eyelids, for at least 15 winutes. Seek medical attention if symptoms posses.

## VII - Precautions for Safe Handling and Use

Steps To Be Taken in Case Material is Released Or Spilled

Wear appropriate respiratory and proceetive equipment specified in section. VIII-control measures, Isolate spill area and provide ventileties. Vacuum up spill using a high efficiency particulate absolute (HEPA) air filter and place in a closed container for proper disposal. Take care not to raise dust.

Wasie Disposal Method

Dispose of it accordance with local, state and federal regulations.

Hazard Label Information:

Store in airigin containers. Store in rightly scaled container. Wash thoroughly after handling

Procautions To Be Taken in Handling

## MATERIAL SAFETY DATA SHEET Lanthanum boride, cathodes

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### Precautions To Be Taken In Storing

Mildely is sensitive, will pick up water and hydrolyze. Store in ma simight container or under argan.

Other Precautions

None

### VIII- Control Measures

Protective Equipment Summary - Hazard Label Information:

Imperious glaves Safety glasses

Respiratory Equipment (Specify Type)

Not applicable

Eye Protection

Soldty glasses

Protective Gloves

Rubber gloves

Other Protective Clothing

Not applicable

Ventilation

Local Extract: If user operations generate dust, fune or mist, use ventilation to keep exposure to airborne contaminants to a minimum. Special: None Mechanical (Cen): Good general ventilation is recommended

Other: None

Work/Hygienic/Maintenance Practices

implement engineering and work practice controls to reduce end maintain concentration of exposure at low levels. Use good housekeeping and sanitation practices. Do not use tobacco or food in work area. Wash theroughly before eating and smoking. Do not blow dust off clothing prakin with compressed air.

## IX - Additional Comments

### Additional information About This Product

Control of Substances Hazardras to Health Regulations 6H40 Occupational Exposure Limits

Maximum Exposure Limit: NE Occupational Exposure Standard: NE

### Company Policy or Disclaimer

The above information is accurate to the best of our knowledge. However, since data, safety standards, and government regulations are subject to change, and the conditions of bendling and use or unstate are beyond our control, CERAC.MARES NO WARRANTY, EITHER EXPRESSED NOR IMPLIED. WITH RESPECT TO THE COMPLETENESS OR CONTINUING ACCURACY OF THE INFORMATION CONTAINED HEREIN, AND DISCLAIMS ALL LIABLITY FOR RELIANCE THEREON. Users should ratiefy themselves that they have all current data relevant to their particular use.

Abbreviations used: NA=Not Applicable NE: Not Established