Serial Number 99021201

INSTRUCTION MANUAL

Model 672

Single Tilt Heating Straining Holder

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PACKING LIST

672 Heating Straining Holder

This shipment should contain the following items:

Packer's Initials [signature]

Item	Qty	Part Number	Description
☑	1	672._ _ _ _	Straining Holder Model 672.P2303T0 S/N 03122201
☑	1	672.40000	Instruction Manual
☑	2	654.00032	Hexlok™ (Tantalum)
☑	1	654.53000	Hexlok™ Tool
☐	2	654.00042	Specimen Clamping Plate (Tantalum)
☑	1	594.13000	Rod Stand
☐	1	654.50_ _	Strain Controller 100V___ 120V___ 240V___ S/N____________
☐	__	04333	Power Cord 120V (Japan,US)
☐	__	04983	Power Cord 240V (Continental European)
☐	__	04339	Power Cord 240V (United Kingdom)
☐	__	06306	Power Cord 240V (Australian)
☐	__	05071	Power Cord 240V (Swiss)
☐	__	03605	Plug Adaptor (Japan)
☐	1	654.52000	Holder Cable
☐	1	09163	Line Transformer
☐	1	654.51000	Hand Control Assembly
☐	1	672.00046	Service Kit (Tantalum)
☐	1	901.60_ _	SmartSet Control 100V___ 120V___ 240V___ S/N____________
☐	1	628.09J	Water Recirculator with 2 Hoses
☐	1	628.05070	Power Supply Cable
☐	1	901.40000	SmartSet Instruction Manual
☐	1	672.00046	Service Kit (Tantalum)
☐	1	902._ 6	Accutroller S/N____________
☐	1	902.40000	Accutroller Instruction Manual
☐	1	902.00011	Cable, Tensile Drive
☐	1		Accutroller Transformer
☑	2	654.00049	Washer (Macor)

For your own protection you should inspect the shipment upon arrival and inform the freight company immediately of any damage and GATAN Inc. if there is any discrepancy in the order.

1. Introduction

The Model 672 is a side entry, single tilt, heating, straining holder used for elongating miniature tensile specimens at controlled rates and temperatures in the transmission electron microscope (TEM). The elongation rate can be set to any level in the range 0.01µm/second to 1.0µm/second using the Model 654 6000 strain control unit. Elongation can be stopped and restarted at will with a push button hand control. The hand control also has a X10 range switch to display crosshead displacements with a resolution of 1µm or 0.1µm and an auto zero toggle switch to return the crosshead to its original zero position. A chart recorder output of 1µm/mV is provided at the rear of the strain control unit for recording the elongation history of the specimen.

The Model 672 also incorporates a temperature control unit (Model 628-0500) used to heat a furnace which is in direct physical contact with the tensile specimen. The furnace which is availiable in either a tantalum or inconel version to operate in vacuum or controlled atmosphere environments respectively, is mounted on three insulating pins and is spring loaded against the specimen tip in order to accommodate expansion and contraction occurring during heating and cooling. The furnace contains a miniature encapsulated 10 watt heater which is spot welded to two terminal posts in the specimen tip. Heat which leaks from the furnace to the specimen tip is removed by the Water Recirculating Unit (Model 628-0900) which cools a tubular conductor which runs from the tip to a manifold at the opposite end of the specimen rod. The heater and thermocouple leads from the specimen tip are brought to the outside of the TEM along the axis of the conductor and then to a 5 pin vacuum feed-through mounted in the side of the cooling manifold.

2. Specimen holder (see figure 1)

The specimen is elongated via a micrometer screw driven by a DC motor. The large gear reduction needed to provide the small displacement rate required in TEM studies is achieved using a 2190:1 low-backlash, spur gear train followed by a 40:1 reduction precision worm and wheel drive attached to the female body of the screw thread. The male part of the screw is connected to the specimen at one end and to a slotted key at the other end. The slotted key rides between two fixed pins that allow free in and out motion while preventing the screw from rotating. The screw position is sensed by a 10 turn potentiometer that is turned by a spur gear mounted on top of the worm wheel. The spur gear ratio is such that the potentiometer turns twice for each revolution of the worm wheel. The tensile drive shaft coupling the male screw drive to the specimen rides on an O-ring which forms a sliding vacuum seal within the specimen rod barrel. The total drive range available is about 2.0mm.

3. Specimen loading (see figure 2)

A drawing of the specimen tip showing the tools needed for specimen loading is shown in figure 2. The figure also shows the recommended design and dimensions of the tensile specimen for *in situ* experiments. Normally, tensile specimens of the type shown in figure 2 are made using some form of photo-resist etching technique. They can also be made by milling a thin foil that has been glued to a supporting substrate. The central web of the specimen shown in figure 2 must be thinned to electron transparency by electrolytic or ion polishing. The two outer arms of the specimen are left unthinned in order to provide the mechanical support for the thin central web. Having prepared the specimen the specimen loading steps are as follows:

Step 1. Place the specimen rod on the specimen rod stand and position the tip under a stereomicroscope.

Step 2. Connect the strain control unit to the specimen holder and to the hand control. Operate the AUTO ZERO switch on the hand control to drive the moveable crosshead to the zero position. After turning off the auto zero switch, the RANGE button should be actuated to display a 0.1µm resolution.

and the EXTEND button depressed until the display just starts to move above 0.0μm. This operation takes up the backlash from the drive mechanism so that when the EXTEND button is later depressed the crosshead will immediately start to move in the +direction.

Step 3. Unscrew and remove the Hexlok™ screws using the Hexlok™ tool.

Step 4. Using fine pointed tweezers place the specimen so that it straddles the crossheads and the furnace. Then gently nudge the specimen so that the holes on the crossheads are aligned with the holes in the specimen. The spacing of the holes in the crosshead can be changed by switching on the auto zero and adjusting the ZERO OFFSET knob on the control unit. If the zero offset is changed then the auto zero must be switched off and the procedure described above for removing the backlash must be repeated.

Step 5. Place the two clamping plates on the specimen so that the holes are aligned.

Step 6. Using fine pointed tweezers place the two Hexlok™ screws into the holes so that they are resting against the start of the threads in the crossheads. Then tighten the screws carefully using the Hexlok™ tool. Be careful not to buckle the specimen. The cross heads on which the specimen is mounted are slightly below the level of the furnace so that when the specimen is elongated it will remain in good physical contact with the furnace. Good physical contact is necessary in order to obtain efficient heat transfer between the furnace and the specimen.

4. Strain control unit and hand control (see figures 3 and 4)
The Model 654-5000 strain control is designed for use with the Gatan Model 654, 671 and 672 single tilt straining, straining cooling and straining heating holders. It provides the power for the motorized micrometer drive which elongates the specimen. It also monitors the position of the moveable crosshead with respect to a reference position set by the ZERO OFFSET control on the front panel of the control unit. Extension start and stop commands are conveniently made from a separate hand control which can be positioned next to the TEM stage drive so that the strain control can be operated without losing control of the specimen position. The hand control can also be used to automatically return the crosshead to the reference position. The LED becomes illuminated to warn the operator when the auto zero switch has been selected. This switch is located on the side of the hand control so that it cannot be accidentally actuated during an experiment.

The crosshead displacement is displayed by an LED meter and shows ± movements relative to the reference position. At the lower sensitivity, which is the default mode at instrument power up, the maximum reading is ±1999 microns. Pressing and releasing the RANGE push button on the hand control switches between the lower and higher sensitivity ranges. The higher sensitivity range is ±199.9 microns. The position of the moveable crosshead is monitored by a 10 turn potentiometer. Circuitry in the control unit applies a reference voltage of +12Volts to this potentiometer. The wiper voltage is fed to the control circuitry where it is processed to control the drive power to a motor in the specimen holder, in response to the switch commands from the hand control.

The control unit includes two servo loops. A control loop to move the crosshead until the signal from the position sensing potentiometer matches the signal from the front panel zero control; and a second control loop to move the specimen at a rate such that the time differential of the signal from the sensing potentiometer matches the signal from the front panel STRAIN RATE control. The selection of the relevant control loop is performed by electronic analog switches driven by the hand control.

The range of the ZERO OFFSET control is limited to move the position sensing potentiometer from 1/2 turn to 9 turns in the clockwise direction. The EXTEND button on the hand control is also limited at the

9 turn position thus ensuring that the potentiometer cannot be damaged by being driven into the end stop. The control unit is powered via a double fused line power connector with a built-in international voltage selector switch. Observe that the voltage specified on the serial number label on the rear panel of the control is correct before plugging into the AC power source.

5. Heat Control Unit (see figures 5 and 7)

A drawing of the heat control unit is shown in Figure 5. Observe the voltage specified on the serial number label on the rear panel of the control unit before plugging into the AC power source. (The correct operating voltage is also marked on the card plugged under the fuse inside the fuse holder). When using a holder with a tantalum furnace which oxidizes rapidly when heated in air **do not turn on the heater power unless the furnace is under high vacuum** (<10^{-5} Torr). The terminal identifications to the specimen rod are:

A = Heater +15 Volt maximum
B = Heater common to microscope ground
D = Platinum (negative) thermocouple wire
E = Platinum 13% Rhodium (positive) thermocouple wire

Note that chart recorder terminals are provided at the rear of the power supply to facilitate recording the thermal history of the furnace. The circuit diagram of the control unit is shown in figure 7.

The digital current meter on the right of the front panel indicates the current (in milliamps) flowing through the heater. This current is controlled by the potentiometer mounted under the meter. No current will flow through the heater unless the heater push button is depressed and all connections to the heater are made. Always turn the heater control fully counterclockwise at the end of an experiment otherwise, when the heater switch is next depressed, the thermal shock due to the sudden power surge could crack the heater insulation.

Specimen temperature is measured using an Omega Model 650 digital thermometer and a platinum/platinum 13% rhodium thermocouple. The thermocouple is spot welded to the furnace body and so the temperature indicated is really that of the furnace and not of the specimen. In practice, the actual specimen temperature will always be lower than that of the furnace because of the heat drain to the crosshead and because of poor thermal contact between the specimen and the furnace. It is recommended that specimens be made as thin as possible in order to minimize the heat drain and maximize the degree of thermal contact with the furnace.

6. Water Recirculating Unit

If the holder is to be operated above 500°C the water recirculator must be connected. The recirculator plugs into the Model 628-0500 Heating Control Unit and operates from the HEATER switch on the front panel of the control unit. The pump is mounted directly to a stainless steel tank filled with 2.5 liters of distilled water at ambient room temperature. There are two main advantages to using the recirculator 1.) the elimination of a source of vibration due to fluctuations in pressure of water lines; 2.) the elimination of a source of drift due to sudden changes in water temperature.

The recirculator is fully self contained and measures 17.5cm high x 17.5cm wide x 46cm long. It is supplied with two lengths of polyurethane tubing, each one 260cm long. The length and diameter of the tubing should not be changed or the performance will be greatly affected. The tubing is fitted with nylon compression fittings that attach to the specimen holder fittings, flow direction is not important. These nylon compression fittings need only be tightened by hand (DO NOT USE A WRENCH), check for water leaks after turning on the recirculator. The pump and the tubing runs were optimized to permit sufficient cooling of the holder to a maximum height of 200cm above the pump. The flow rate will remain fairly constant (75ml/min) over this range. A detachable cover with gasket is fitted to the tank to prevent spillage should the recirculator be accidentally tipped over. The cover thumbscrews need only be lightly tightened.

7. Operation

After loading the specimen, the specimen holder should be inserted into the microscope according to the manufacturer's instructions. The heater and strain control cables are then connected to the specimen holder as shown in figure 6. **Warning:** Prior to inserting the holder into the microscope verify that the pole piece will accept the specimen tip. Make sure the goniometer is set so that the specimen tip cannot be tilted beyond the limit accepted by the TEM. This is especially important in laboratories using the same specimen holder on two or more TEMs. For optimum performance the water lines and heater and strain control cables close to the specimen rod should be secured to the microscope goniometer.

Select the area of interest in the specimen and depress the MAINS switch on the strain and heat control units. The specimen temperature and crosshead position will then be indicated on the digital meters. Depress the HEATER switch to display the heater current. Turn the heater current control clockwise to raise the specimen temperature. The specimen will start to move because of thermal expansion even though the furnace suspension system and the conductor of the Model 672 have been designed to minimize this movement. Typically, the specimen drift will decrease to manageable levels after about 10 to 15 minutes at temperature. If specimen vibration is observed, the cables connected to the holder should be secured to the microscope goniometer housing with adhesive tape or other means. When the specimen temperature increases the vacuum in the specimen chamber may deteriorate slightly. This is normal and is due mainly to water vapor being driven off the specimen holder.

Tantalum Furnace

The furnace is rated at 1000°C but has been shown in factory tests to survive 48 hours continuous operation at 1100°C. However, the operator must be aware of two serious problems which can limit the high temperature use of the holder:

1. The specimen can melt and weld to the furnace.
2. The specimen can form an alloy by solid state diffusion with the furnace material and this alloy can then melt and weld the specimen to the furnace or even cause the whole furnace to melt at an unexpectedly low temperature.

All high temperature metal parts in the furnace are made of the refractory metal tantalum. Before starting a heating experiment it is advisable to check (a) the melting point of the specimen (b) the phase diagrams of tantalum and the specimen. A safe operating temperature is about 50°C less than the melting or liquidus temperature. If this information is not available the compatibility between the furnace and the specimen should be tested by raising the temperature in increments and checking for any signs of melting or welding.

Inconel Furnace

The 672 holder with an inconel furnace is designed to be used in the controlled atmospheres of an environmental cell. Inconel furnaces are rated for operation at a maximum temperature of 900°C. However, the operator must be aware of two serious problems which can limit the high temperature use of the holder:

1. The specimen can melt and weld to the furnace.
2. The specimen can form an alloy by solid state diffusion with the furnace material and this alloy can then melt and weld the specimen to the furnace or even cause the whole furnace to melt at an

unexpectedly low temperature.

The high temperature parts of the holder are made of either inconel or platinum and hence it is important not to perform heating experiments in atmospheres that attack these materials. Inconel was chosen because it can be heated in a wide variety of gases, however Inconel is highly susceptible to attack by sulphur and environments containing this element should be avoided. Before starting a heating experiment it is advisable to check (a) the melting point of the specimen (b) the phase diagrams of inconel and the specimen. A safe operating temperature is about 50°C less than the melting or liquidus temperature. If this information is not available the compatibility between the furnace and the specimen should be tested by raising the temperature in increments and checking for any signs of melting or welding.

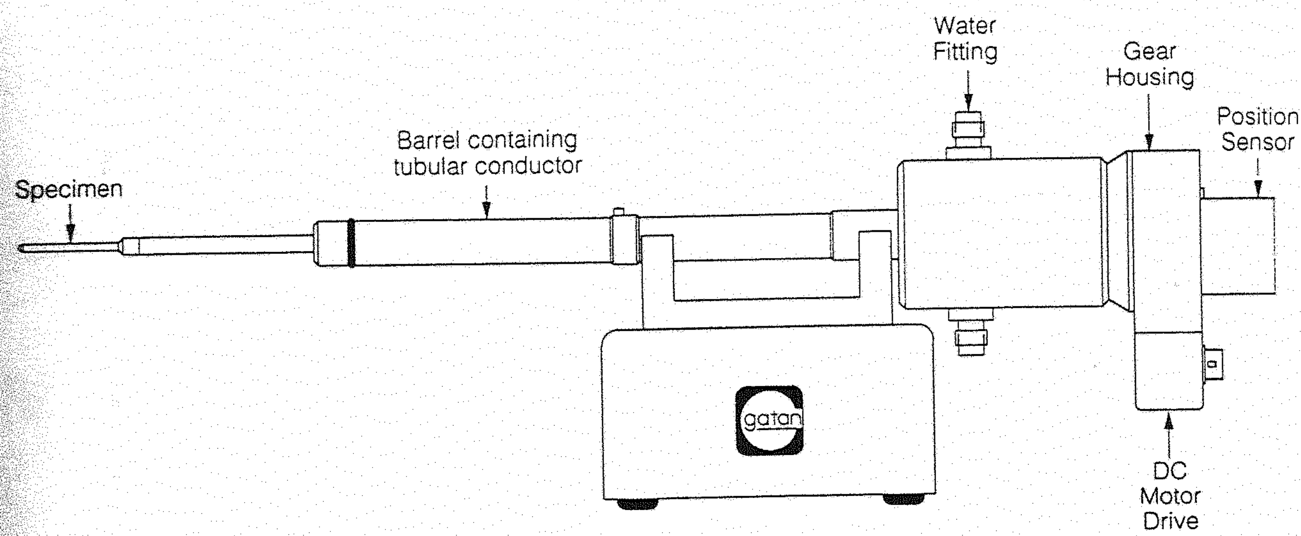
The power required to achieve a given temperature is sensitive to the pressure and composition (and hence thermal conductivity) of the environment surrounding the furnace. Normally, this does not cause a problem since the furnace temperature is measured directly by a thermocouple attached to the furnace and can be adjusted accordingly. However, if the cell pressure is suddenly changed during an experiment the specimen temperature may vary at a rate which is hard to compensate. In extreme cases, it is possible to melt the specimen and burn out the furnace if the environmental cell is suddenly evacuated. The effect is especially pronounced if the controlled environment prior to evacuation consisted of a light gas such as helium or hydrogen.

Any questions concerning the use of the holder or experimental conditions should be addressed to Gatan before experiments are attempted.

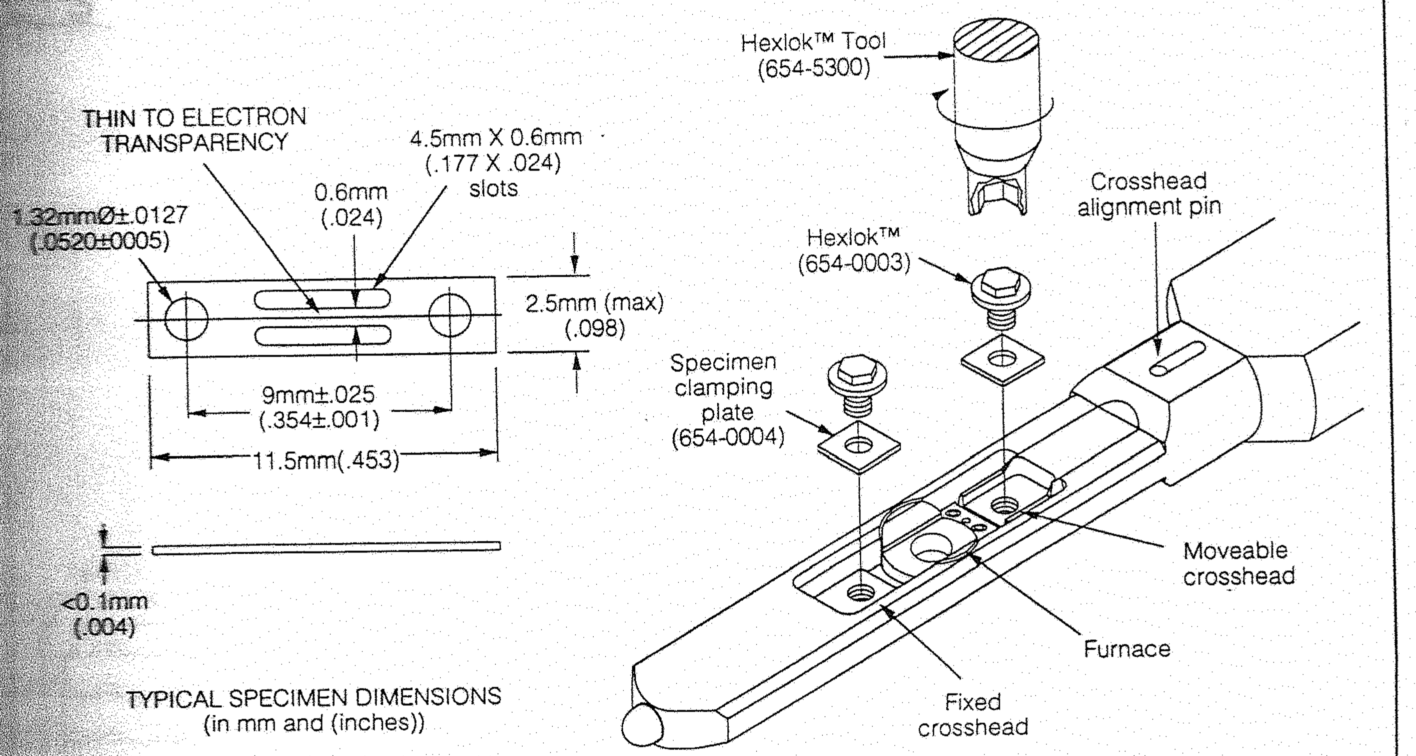
8. Servicing

Failures resulting from poor workmanship will be repaired free of charge up to one year from the date of shipment. Other types of failure will be repaired at the fixed rates specified in the GATAN product service price list. Failures not covered by the warranty include:

a. Burnt out heater coil
b. Broken ceramic furnace mounts
c. Jammed Hexring™
d. Furnace and furnace lead failures caused by melting or solid state welding
e. Damaged or broken furnace thermocouple leads



gatan Figure 1. Model 672 Single tilt heating straining holder 11/92



gatan Figure 2. Recommended specimen dimensions and specimen loading tools 11/92

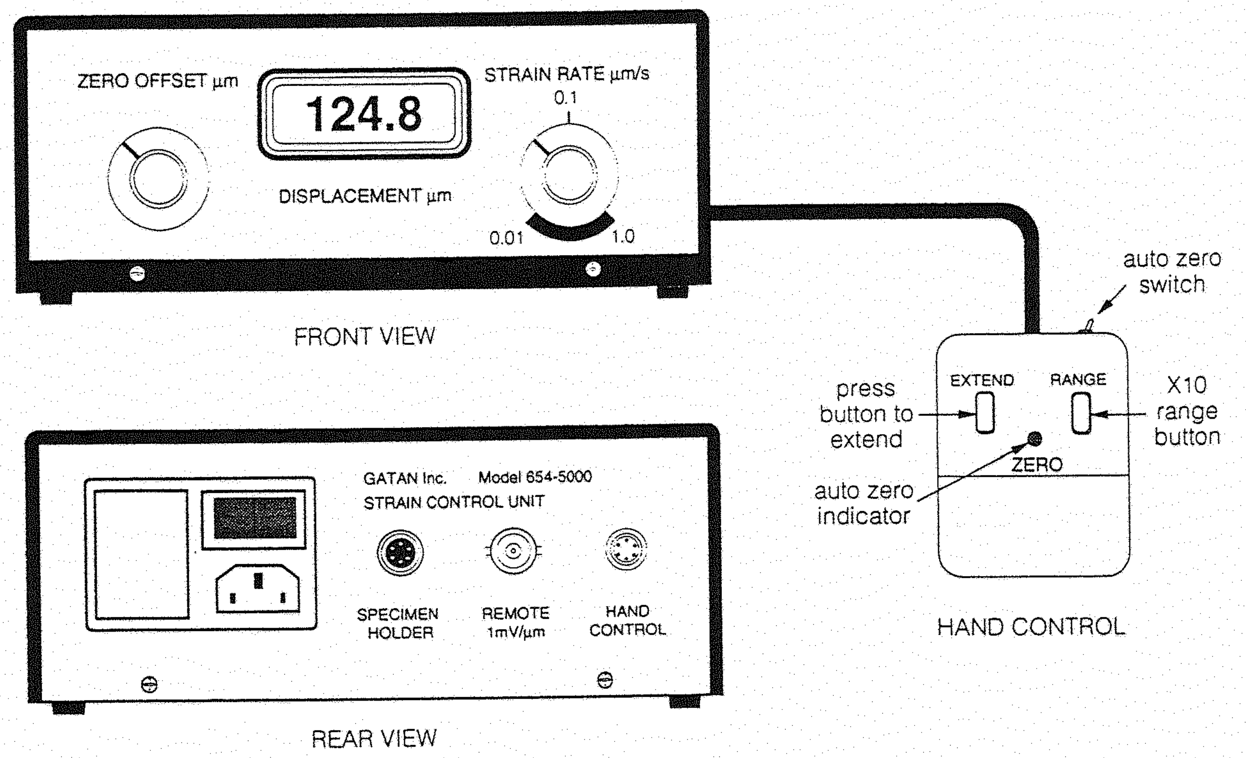


Figure 3. Strain control unit with hand control

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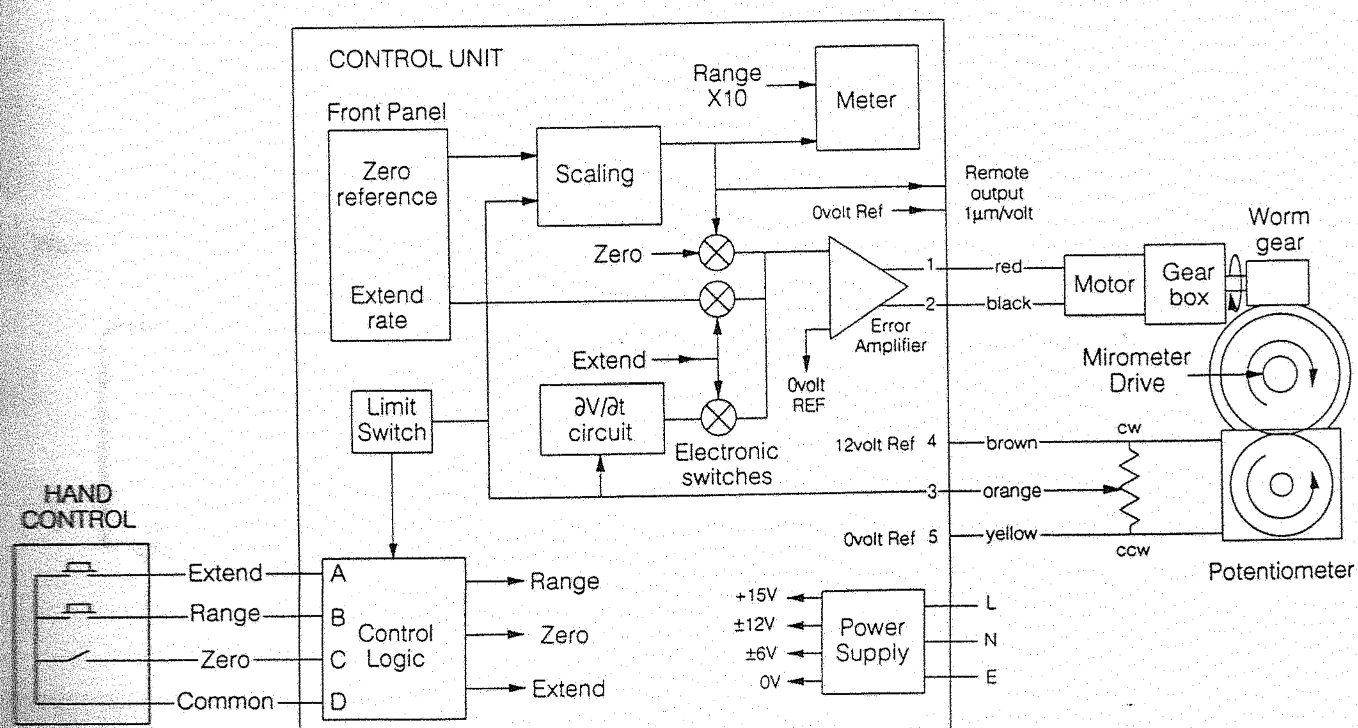

Figure 4. Block diagram of strain control circuit

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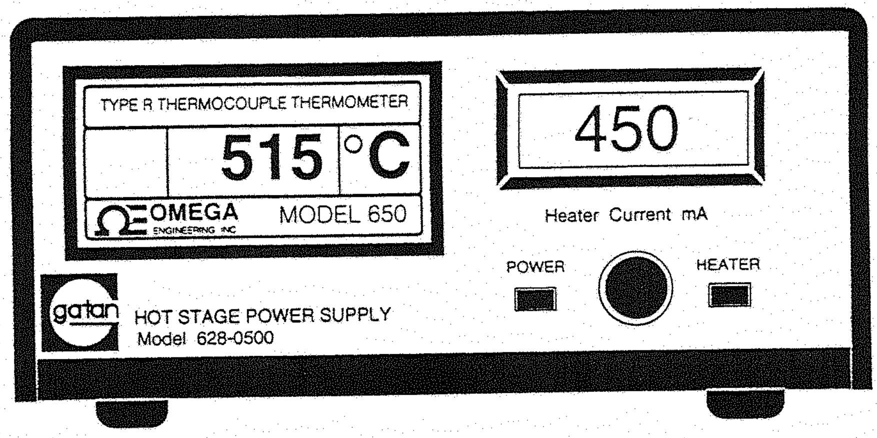
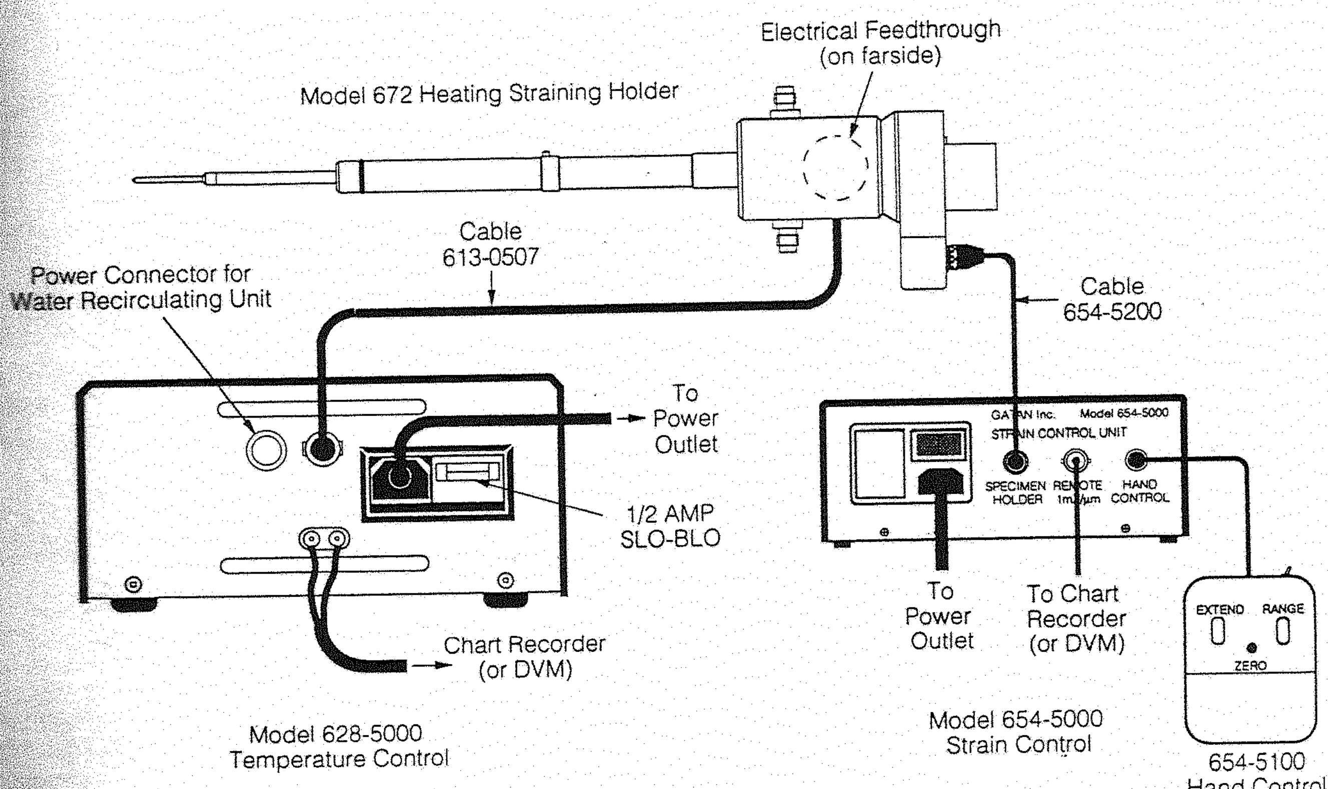
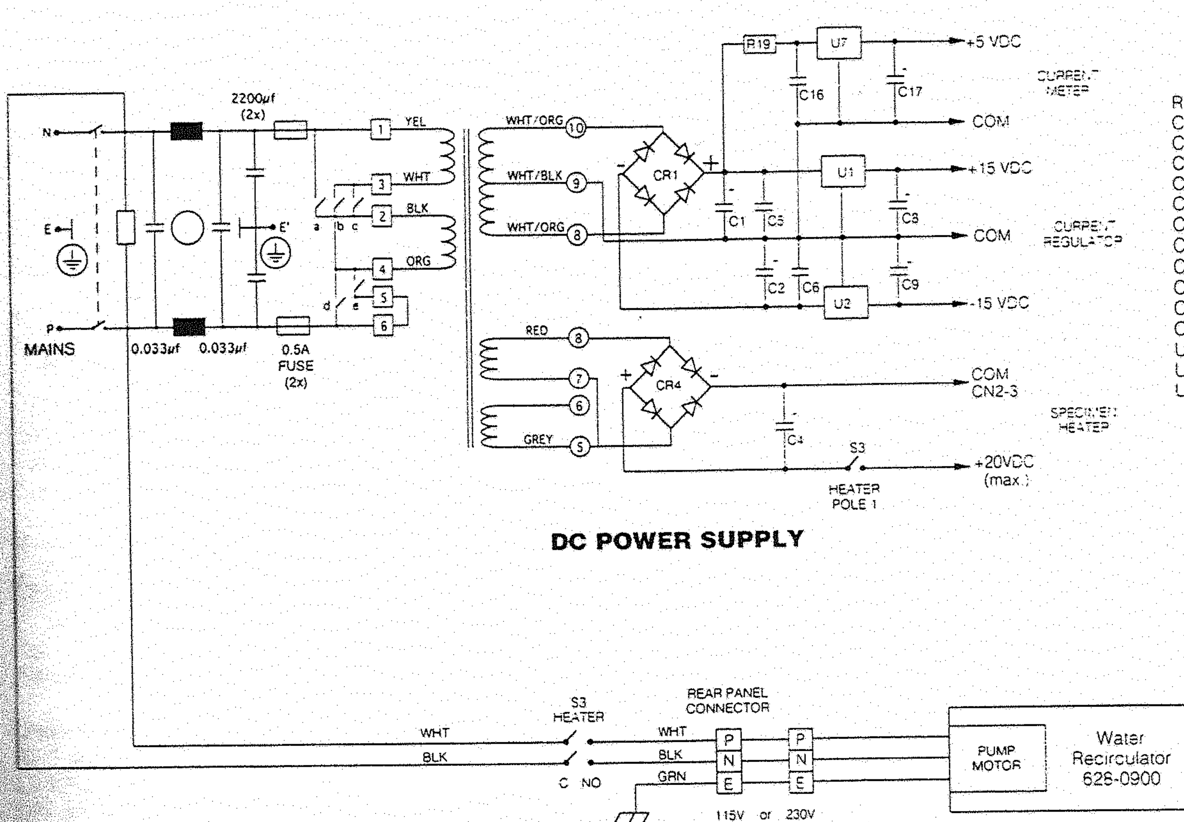


gatan Figure 5. Model 628-0500 Temperature Controller 4/92



gatan Figure 6. Electrical Interconnections 4/92



R19 = 50Ω
C1 = 2200µF 35V
C2 = 2200µF 35V
C4 = 3300µF 35V
C5 = 1µF 50V
C6 = 1µF 50V
C8 = 4.7µF 35V
C9 = 4.7µF 35V
C16 = 1µF 50V
C17 = 4.7µF 35V
CR1 = SK3648
CR4 = SK3648
U1 = MC7815
U2 = MC7915
U7 = MC7805

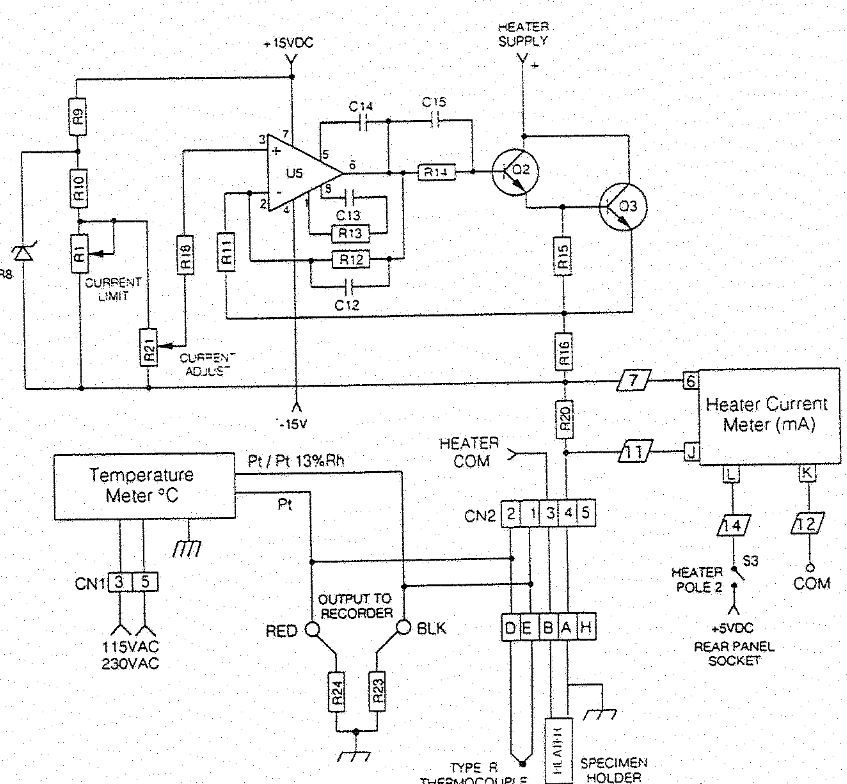
R1 = 500Ω
R9 = 2.2KΩ
R10 = 4.7KΩ
R11 = 1KΩ
R12 = 1MΩ
R13 = 2.7KΩ
R14 = 2KΩ
R15 = 100Ω
R16 = .15Ω
R18 = 1KΩ
R20 = .1Ω
R21 = 1KΩ
R23 = 68KΩ
R24 = 100KΩ

C12 = .0047µF
C13 = .0047µF
C14 = .001µF
C15 = .470pF

CR8= IN823

Q2 = MJE3055
Q3 = MJE3055

U5 = UA709



gatan Figure 7. Circuit Diagram of Model 628-0500 Hot Stage Temperature Control Unit | 4/97

Serial Number 03122201

INSTRUCTION MANUAL

Model 672

Single Tilt Heating Straining Holder

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ITEM NO. 672.40000

1. Introduction

The Model 672 is a side entry, single tilt, heating, straining holder used for elongating miniature tensile specimens at controlled rates and temperatures in the transmission electron microscope (TEM). One end of the specimen is attached to a fixed mounting point and the other end is attached to a moveable crosshead. The specimen attachment method uses a Hexlok™ and clamping plate at each end of the sample. The Hexlok™ is a threaded fastener with a washer head that has a hexagon protrusion for tightening and removal. The Hexloks™ pass through holes in the clamping plates and each end of the specimen and mate with threaded holes in both the specimen tip and crosshead. An Accutroller control unit is provided to apply a constant elongation rate in the range of 1.0μm/second. Elongation can be stopped and restarted at will with a push button. The Accutroller displays crosshead displacements with a resolution of 1μm and has an auto zero button to return the crosshead to either its original zero position or an offset zero position.

The Model 672 also incorporates a temperature control unit (Model 901 SmartSet) used to heat the furnace which is in direct physical contact with the tensile specimen. The furnace which is availiable in either a tantalum or inconel version to operate in vacuum or controlled atmosphere environments respectively, is mounted on three insulating pins and is spring loaded against the specimen tip in order to accommodate expansion and contraction occurring during heating and cooling. The furnace contains a miniature encapsulated 10 watt heater which is spot welded to two terminal posts in the specimen tip. Heat which leaks from the furnace to the specimen tip is removed by the Water Recirculating Unit (Model 628-09J) which cools a tubular conductor that runs from the tip to a manifold at the opposite end of the specimen rod. The heater and thermocouple leads from the specimen tip are brought to the outside of the TEM along the axis of the conductor and then to a 5 pin vacuum feed-through mounted in the side of the cooling manifold.

The 672 furnace temperature is measured using a platinum/platinum 13% rhodium thermocouple. The thermocouple is spot welded to the furnace body and so the temperature indicated is really that of the furnace and not of the specimen. In practice, the actual specimen temperature will always be lower than that of the furnace because of the heat drain to the crosshead and because of poor thermal contact between the specimen and the furnace. It is recommended that specimens be made as thin as possible in order to minimize the heat drain and maximize the degree of thermal contact with the furnace.

2. Specimen holder (see figure 1)

The specimen is elongated via a micrometer screw driven by a DC motor. The large gear reduction needed to provide the small displacement rate required in TEM studies is achieved using a 2190:1 low-backlash, spur gear train followed by a 40:1 reduction precision worm and wheel drive attached to the female body of the screw thread. The male part of the screw is connected to the specimen at one end and to a slotted key at the other end. The slotted key rides between two fixed pins that allow free in and out motion while preventing the screw from rotating. The screw position is sensed by a 10 turn potentiometer that is turned by a spur gear mounted on top of the worm wheel. The spur gear ratio is such that the potentiometer turns twice for each revolution of the worm wheel. The tensile drive shaft coupling the male screw drive to the specimen rides on an O-ring which forms a sliding vacuum seal within the specimen rod barrel. The total drive range available is about 2.0mm.

3. Accutroller (see separate Accutroller Instruction Manual)

The straining version of the Accutroller is designed for use with the Gatan Model 654, 671 and 672 single tilt ambient straining, cooling straining and heating straining holders. It provides the power for the motorized micrometer tensile drive which elongates the specimen. It monitors the position of the moveable crosshead using the signals from a 10 turn position sensing potentiometer. The crosshead can also be positioned with respect to a reference location set by the SET OFFSET menu. The range of the SET OFFSET function is limited to the movement of the position sensing potentiometer from 1/4 turn to 9 3/4 turns in the clockwise direction. The COMPRESS button travel is limited at the 1/4 turn position. The EXTEND button travel is limited at the 9 3/4 turn position thus ensuring that the potentiometer cannot be damaged by being driven into either of the end stops.

4. Model 901 SmartSet Hot Stage Controller (see separate Instruction Manual)

The Model 901 SmartSet Hot Stage Controller is used for temperature control of any Gatan heating holder this includes models 628, 652 or 672. The unit can be set to work with holders which have either inconel or tantalum furnaces. The Model 901 has multiple functionality in that it can be used in either a totally "MANUAL" mode, a

"SMART" heating mode, or a "RAMP" heating mode. In the manual mode, the furnace temperature is changed by adjusting the heater current until the desired temperature is reached. This mode operates the unit in exactly the same manner as the previous design 628.05000 Hot Stage Control Unit. The smart mode allows the operator to set a desired temperature and the unit will automatically raise the furnace temperature to the set point and maintain that temperature within a range controlled by the built in microprocessor. The temperature ramping mode allows the operator to set temperature and time intervals to raise the furnace temperature incrementally. All of these modes can be controlled by using the knob and pushbutton switches on the front panel. The "SMART" and "RAMP" modes as well as other functions are also accessible through the RS232 serial port on the rear panel. Please see separate Model 901 Instruction Manual.

5. Specimen loading (see figure 2)
A drawing of the specimen tip showing the tools needed for specimen loading is shown in figure 2. The figure also shows the recommended design and dimensions of the tensile specimen for *in situ* experiments. Normally, tensile specimens of the type shown in figure 2 are made using some form of photo-resist etching technique. They can also be made by milling a thin foil that has been glued to a supporting substrate. The central web of the specimen shown in figure 2 must be thinned to electron transparency by electrolytic or ion polishing. The two outer arms of the specimen are left unthinned in order to provide the mechanical support for the thin central web. Having prepared the specimen the specimen loading steps are as follows:

Step 1. Place the specimen rod on it's stand and position the tip under a stereo microscope. When handling the specimen holder avoid touching any parts of the holder which are on the vacuum side of the main O-ring. Finger prints contain grease and salt which corrode metal parts, degrade the specimen chamber vacuum and may cause the specimen to become contaminated. Instructions for cleaning the specimen holder are given in section 8.
Step 2. Unscrew and remove the Hexlok™ screws and clamping plates using the Hexlok™ tool.
Step 3. Connect the Accutroller unit to the specimen holder.
Step 4. Operate the AUTO ZERO switch to drive the moveable crosshead to the absolute zero position of the holder. This position places the mounting holes at a distance of 9 millimeters apart.
Step 5. Using fine pointed tweezers place the specimen so that it rests on the furnace and straddles the mounting surfaces in the holder. Then gently nudge the specimen so that the clamping holes are aligned.

If it is necessary to adjust the distance of the mounting holes farther apart to allow for variations in samples, the zero position can be altered using the SET OFFSET menu selection. Use the ELONGATE button to move the crosshead to a position that will align the threaded holes in the holder with the holes in the sample. Scroll through the menus until the SET OFFSET menu appears. Activate the ENTER button. The Accutroller will now recognize this position as zero. The AUTO ZERO function will now return the crosshead to this point instead of the absolute zero position. Please note that the position of the mounting holes will not be less than 9 millimeters. This protects the tensile drive from damage.
Step 6. Place the two clamping plates on the specimen so that the holes are aligned at each end of the sample.
Step 7. Using fine pointed tweezers, place the two Hexlok™ screws into the holes so that they are resting against the start of the threads. Then tighten the screws carefully using the Hexlok™ tool.

6. Water Recirculating Unit
If the holder is to be operated above 500°C the water recirculator must be connected. The recirculator plugs into the Model 901 SmartSet controller and operates automatically when the holder temperature reaches 500°C. See the separate Model 901 instruction manual for operating the pump at temperatures lower than 500°C. The pump is connected to a stainless steel tank filled with 2.5 liters of distilled water at ambient room temperature. There are two main advantages to using the recirculator 1.) the elimination of a source of vibration due to fluctuations in pressure of water lines; 2.) the elimination of a source of drift due to sudden changes in water temperature. The recirculator is fully self contained and measures 17.5cm high x 17.5cm wide x 46cm long. It is supplied with two lengths of polyurethane tubing, each one 260cm long. The length and diameter of the tubing should not be changed or the performance will be greatly affected. The tubing is fitted with nylon compression fittings that attach to the specimen holder fittings. These nylon compression fittings need only be tightened by hand (DO NOT USE A WRENCH). FLOW DIRECTION IS IMPORTANT. Please see figure 3 for the proper connection of the water lines to the holder. The pump and the tubing runs were optimized to permit sufficient cooling of the holder to a maximum height of 200cm above the pump. The flow rate will remain fairly constant (75ml/min) over this range. A detachable cover with gasket is fitted to the tank to prevent spillage should the recirculator be accidentally tipped over. The cover thumbscrews

need only be lightly tightened.

7. Operation

After loading the specimen, the specimen holder should be inserted into the microscope according to the manufacturer's instructions. **Warning:** Prior to inserting the holder into the microscope verify that the pole piece will accept the specimen tip. Make sure the goniometer is set so that the specimen tip cannot be tilted beyond the limit accepted by the TEM. This is especially important in laboratories using the same specimen holder on two or more TEMs. For optimum performance the water lines, SmartSet and Accutroller cables close to the specimen rod should be secured to the microscope goniometer.

Since the specimen is grounded through the control unit, the heater cable and control unit should be connected at all times when imaging the specimen to prevent charging. When the water recirculator is operating, allow approximately 20 minutes for the specimen rod temperature to stabilize to the water temperature and microscope temperature. At any temperature increase the specimen will start to move because of thermal expansion. The furnace suspension system and the conductor of the Model 672 have been designed to minimize this movement. Typically, the specimen drift will decrease to manageable levels after about 10 to 15 minutes at temperature. If specimen vibration is observed, the cables connected to the holder should be secured to the microscope goniometer housing with adhesive tape or other means. When the specimen temperature increases the vacuum in the specimen chamber may deteriorate slightly. This is normal and is due mainly to water vapor being driven off the specimen holder. This effect can be reduced by baking out the specimen holder in the Gatan model 655 Dry Pumping Station or microscope prior to loading the specimen. If the microscope is used, the airlock must be vented with nitrogen or argon and the specimen loaded immediately.

Tantalum Furnace

The furnace is rated at 1000°C but has been shown in factory tests to survive 48 hours continuous operation at 1100°C. However, the operator must be aware of two serious problems which can limit the high temperature use of the holder:

1. The specimen can melt and weld to the furnace.
2. The specimen can form an alloy by solid state diffusion with the furnace material and this alloy can then melt and weld the specimen to the furnace or even cause the whole furnace to melt at an unexpectedly low temperature.

All high temperature metal parts in the furnace are made of the refractory metal tantalum. Before starting a heating experiment it is advisable to check (a) the melting point of the specimen (b) the phase diagrams of tantalum and the specimen. A safe operating temperature is about 50°C less than the melting or liquidus temperature. If this information is not available the compatibility between the furnace and the specimen should be tested by raising the temperature in increments and checking for any signs of melting or welding.

Inconel Furnace

The 672 holder with an inconel furnace is designed to be used in the controlled atmospheres of an environmental cell. Inconel furnaces are rated for operation at a maximum temperature of 900°C. However, the operator must be aware of two serious problems which can limit the high temperature use of the holder:

1. The specimen can melt and weld to the furnace.
2. The specimen can form an alloy by solid state diffusion with the furnace material and this alloy can then melt and weld the specimen to the furnace or even cause the whole furnace to melt at an unexpectedly low temperature.

The high temperature parts of the holder are made of either inconel or platinum and hence it is important not to perform heating experiments in atmospheres that attack these materials. Inconel was chosen because it can be heated in a wide variety of gases, however Inconel is highly susceptible to attack by sulphur and environments containing this element should be avoided. Before starting a heating experiment it is advisable to check (a) the melting point of the specimen (b) the phase diagrams of inconel and the specimen. A safe operating temperature is about 50°C less than the melting or liquidus temperature. If this information is not available the compatibility between the furnace and the specimen should be tested by raising the temperature in increments and checking for any signs of melting or welding.

The power required to achieve a given temperature is sensitive to the pressure and composition (and hence thermal conductivity) of the environment surrounding the furnace. Normally, this does not cause a problem since the furnace temperature is measured directly by a thermocouple attached to the furnace and can be adjusted accordingly. However, if the cell pressure is suddenly changed during an experiment the specimen temperature may vary at a rate which is hard to compensate. In extreme cases, it is possible to melt the specimen and burn out the furnace if the environmental cell is suddenly evacuated. The effect is especially pronounced if the controlled environment prior to evacuation consisted of a light gas such as helium or hydrogen.

Any questions concerning the use of the holder or experimental conditions should be addressed to Gatan before experiments are attempted.

When the desired temperature is reached some backlash may be introduced due to thermal expansion of the tensile drive shaft. If necessary, backlash can be removed from the system before elongating the sample. Activate the ELONGATE button until the display just starts to read above zero. Reset the SET OFFSET position at this new point. When the ELONGATE button is activated again the elongation measurement will be accurate to this new position.

The offset zero position can be removed by selecting the CLEAR OFFSET menu and activating the ENTER button. Once the offset position is cleared, the AUTO ZERO function will again return the crosshead to the absolute zero position (9 mm between mounting holes).

8. Cleaning the Holder

Vacuum grease and finger prints accidentally smeared onto the specimen tip are most conveniently removed by ultrasonic cleaning in 100% Ethanol or a similar solvent. The recommended procedure is to half fill a small measuring cylinder with the 100% Ethanol and place it into an ultrasonic bath filled with water. The O-ring should then be removed from the specimen rod and any grease in the O-ring groove cleaned off with a piece of lint free tissue dipped in 100% Ethanol. The tip of the specimen rod is then lowered slowly into the solvent up to and including the O-ring groove. Do not immerse the entire specimen rod into the 100% Ethanol. After ultrasonically cleaning for about 30 seconds the rod should be removed from the solvent. The process should then be repeated with a fresh quantity of 100% Ethanol. The 100% Ethanol should be allowed to drain off the specimen rod and any remaining fluid must be given time to evaporate before installing a clean, lightly greased O-ring and placing the specimen holder in the TEM. Ideally, the holder should be dried for about one hour in a Gatan Model 655 Dry Pumping Station before using in the TEM. Please note that if the holder is used carefully it should not require cleaning. The above cleaning procedure should only be carried out if there is evidence that specimen contamination is coming from the specimen holder.

9. Warranty

Gatan specimen holders are warranted per the terms and conditions outlined in the product warranty. Customers should contact their local Gatan representative for instructions on where to send the holder for repair.

Items not covered by the warranty include:

(a) Damage caused by operating the holder beyond the tilt limit imposed by the TEM pole pieces.
(b) Replacement of a cracked jewel at the specimen tip.
(c) Repair of a bent specimen tip.
(d) Burnt out heater coil
(e) Broken ceramic furnace mounts
(f) Jammed Hexlok™
(g) Furnace and furnace lead failures caused by melting or solid state welding
(h) Damaged or broken furnace thermocouple leads
(i) Replacement of a lost Hexlok™

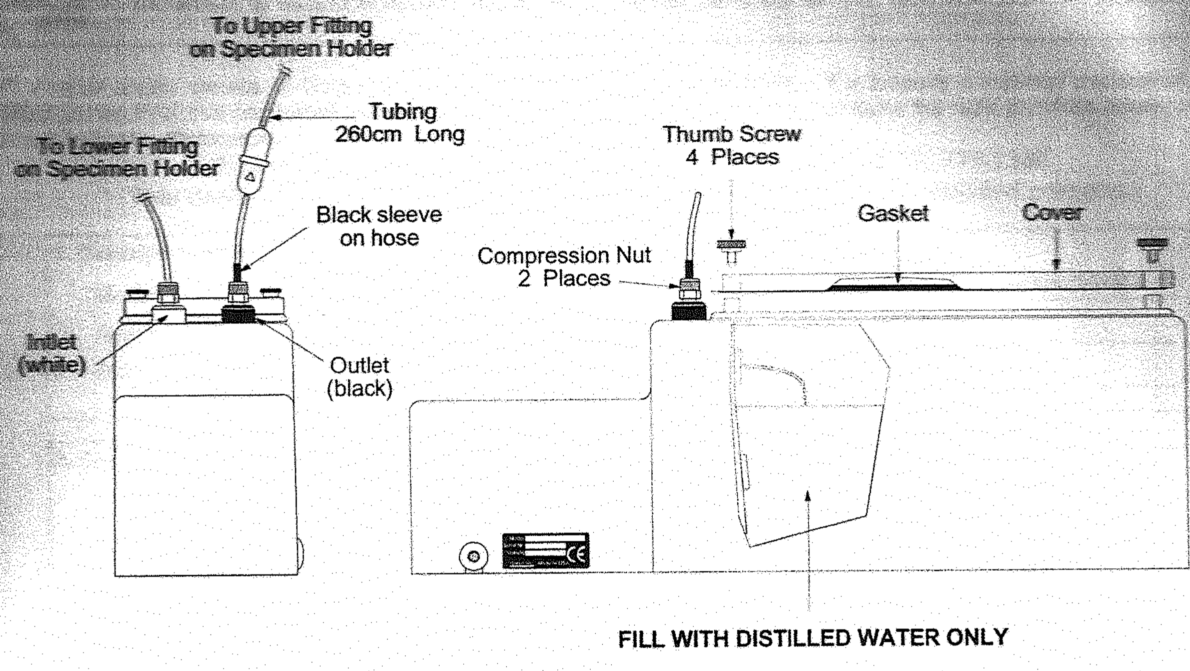

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Figure 3. Model 628.09J Water Recirculator

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