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Fabrication of Large Diameter Alumino-Silicate K+ Sources*

D. Baca, LBNL, E. Chacon-Golcher, J. W. Kwan, J. K. Wu, LBNL

I. Introduction

Alumino-silicate K^+ sources have been used in HIF experiments for many years. For example the Neutralized Transport Expt. (NTX) and the High Current Transport Expt. (HCX) are now using this type of ion source with diameters of 2.54 cm and 10 cm respectively. These sources have demonstrated ion currents of 80 mA (Fig. 1) and 700 mA (Fig. 2), for typical HIF pulse lengths of 5 - 10 μs. The corresponding current density is ~ 10 - 15 mA/cm^2, but much higher current density has been observed using smaller size sources (Fig. 3). Recently we have improved our fabrication techniques and, therefore, are able to reliably produce large diameter ion sources with high quality emitter surface without defects. This note provides a detailed description of the procedures employed in the fabrication process. The variables in the processing steps affecting surface quality, such as substrate porosity, powder size distribution, coating technique on large area concave surfaces, drying, and heat firing temperature have been investigated.

II. Substrate Material and Preparation

Alumino-silicate ion sources are made by "melting" a layer of synthetic alumino-silicate onto a tungsten substrate. In many ways, the process is similar to working with ceramics. Since the ion source will be operated at temperature above 1050 degree C and the melting process is around 1550 degree C, the substrate is usually made of a refractory metal such as tungsten. One way to economically produce tungsten substrates of various sizes and shapes, and minimize the weight is to utilize sintered porous tungsten. The material can be easily machined before sintering or grinded to tolerance after sintering.

Our experience has shown that 80% dense porous tungsten is suitable for ion source application, the lighter density ones are usually too fragile.

There is another very important reason to use porous tungsten substrate. In comparison to a solid substrate, the porous tungsten has a "rough" surface that is more suitable for alumino-silicate to stick on during the melting stage. Previous experience has encountered many failures in trying to melt alumino-silicate to a smooth surface.

It turns out that a bare (without a layer of alumino-silicate coating) porous tungsten substrate is also suitable for being used as a contact ionizer because tungsten has a high work function. The porous structure is ideal for absorbing and storing a large amount of liquid alkaline metal carbonate, sufficient to provide nearly 40 hours of experimental run time at ~500mA current. This can be compared to alumino-silicate coated ionizers that have a considerably much longer experimental run time on the order of months and lower neutral emission levels (Fig. 4). Iridium and platinum have the highest work functions, and they are considerably more expensive. The work functions of several elements, together with the ionization potentials of several alkali metals, are given (Table 1).

In fabricating sintered tungsten substrates, the particle size distribution and geometry of the tungsten powder, spherical or irregularly shaped, can vary by vendor. The tungsten substrates we have chosen are irregular shaped particles and average 4.5μm in diameter and distribution between 8μm and 2.5μm. Dimensions for the HCX 10cm diameter source consist of a concave surface with 20.32cm (8.00") spherical radius for optical focusing considerations, a height of 3.81cm, and a weight of 1.90kg. The emitting concave surface area, in the shape of a spherical cap, has an area equal to $2\pi(1-\cos\theta)R^2$, or 82.38 cm^2. The machining tolerance for the emitting surface is +/-1mil. Expectedly, porous tungsten substrates supplied by the vendor are machined to dimension specifications with its pores closed by the machining processes. Our first step is to reopen the emitting surface pores by electrolysis etching.

Etching is done by a DC electro-chemical process. Throughout the entire process, all tools, utensils, and apparatus are thoroughly cleaned or dedicated to reduce and minimize any possible sources of chemical contamination. A 5 molar KOH solution is produced by mixing 2 liters of high purity (18.1Mohm-cm) deionized water and 500g of potassium hydroxide (KOH) in a 4-liter glass beaker. Because our goal is to make a potassium ion source, potassium hydroxide is preferred in order to minimize the chance of contamination with other alkaline ions. The preferred electrode materials are 316L stainless steel and tantalum because they are

less chemically reactive in an electrolytic solution of KOH. A tungsten substrate is attached to the positive electrode using a clamping ring around its outer diameter with the region to be etched exposed and facing the negative electrode. The negative electrode has a similar shape and contour of the sample etching area, a convex spherical radius of 19.05cm (7.5”), in this case, the shape of a convex spherical cap, so that the electric field lines are uniform across the entire etched surface. Separation between the tungsten and the electrode is set uniformly at ~1.2cm. During the etching process, the negative electrode surface is the recipient of ions removed from the electrolytic solution and the positive electrode loses tungsten ions. A reasonable guideline for electrolytic conduction is to increase the current until sufficient gas bubbles are being generated and the solution looks effervescent. In etching the 10cm diameter source, a 20A DC current with ~2.5V applied across the electrodes for 40 minutes will etch away ~8mil of Tungsten material and this is enough to re-open the pores and produce a uniform surface free of any remaining machining marks. Smaller diameter ion source will need less current, scaled according to the surface area.

After electro-chemical etching, the residual KOH electrolytic solution must be removed from the substrate. However, porous tungsten is spongy and permeable so it is difficult to accomplish this rapidly. Our normal practice is to soak the tungsten substrate in high purity deionized water for 4 days of dilution in a glass container, with the etched surface up, and keep changing the deionized water periodically once every few hours. Ultrasonic cleaning has been tried for short periods (~ two hours) with good success. This method should be taken with some caution with since there is the risk during ultrasonic cleaning of propagating any prior fractures in the porous tungsten. It should be noted that ultrasonic cleaning has been observed to re-close the pores when etched tungsten surfaces come in contact with the glass container (i.e. vibrating against a hard surface). Taking small tungsten substrates and lying the etched surface face down on nylon scouring pads during ultrasonic cleaning has given good results, but with large tungsten substrates, a fixture to hold it upside down during ultrasonic cleaning works better. An accumulation of tungsten particles is normally observed at the bottom of the container after ultrasonic cleaning as loose particles are dislodged from the etched surface. The dilution process is considered completed when the pH value reaches ~6.0 or higher. Afterward the sample is air dried (using clean filtered air), and then further slow dried in a vacuum oven at a pressure of $\sim 1(10)^{-3}$ Torr, and temperature of ~100 degree C to remove any remaining water vapor.

The last step in preparing a substrate is to "clean" fire it at 1750 degree C in a vacuum furnace at a pressure of ~ 1 $(10)^{-6}$ Torr. At this temperature, most low melting point materials, e.g., copper and aluminum, will be removed. SEM photos shows that the surface pore average size is typically ~15μm (Photo 1). For storage purposes, the substrate may be kept in a container filled with dry inert nitrogen gas until ready to coat.

III. Alumino-Silicate Powder Preparation

This procedure describes the preparation of synthesizing potassium-aluminosilicate, as it is heated to ~1450 degree C to produce the Leucite crystalline phase, $K_2OAl_2O_3*4SiO_2$ (Fig. 5). It begins with thoroughly mixing raw high purity chemicals followed by a two-step reaction process in an atmospheric air furnace. Mechanisms such as convection, shear, and dispersion are common blending processes. As with the etching process, all tools, utensils, and apparatus are thoroughly cleaned or dedicated to reduce and minimize any possible sources of chemical contamination.

The chemicals, all in powder form, are 99.995% Potassium Carbonate (K_2CO_3), 99.9% Silicon Oxide (SiO_2), and 99.8% Aluminum Oxide (Al_2O_3). The respective stoichiometric ratio by weight is 1.0:1.739:0.738. Since potassium carbonate is hygroscopic, the mixing process is done in a controlled low humidity environment by flowing dry nitrogen gas through a small glove box and keeping the humidity to below 5%. After mixing, the mixture is tape-sealed in a jar before removing from the glove box to trap in the low humidity nitrogen gas.

The next step is to use a ball mill process to thoroughly blend the chemicals for several hours. Afterward the mixture is transferred to a platinum crucible (the crucible should only be partially filled to halfway in order to allow room in the crucible for expansion without spilling over). Chemical reactions occur when the mixture is heated in an air furnace for 4 hours at 1100 degree C. The product expands and froths as CO_2 gas is released, and then it shrinks to form a solid mass while cooling. The comminution processes that follow consist of crushing and milling to modify the shape of particles, to further disperse the mixture, and to reduce the average particle size of the material.

Continuing, the solid mass is removed from the crucible after cooling and then dry ground with a mortar and pestle to a fine powder, ~ 200 (75μm) mesh size, before placing in a

planetary ball mill for one-hour minimum to reduce to a finer powder size as a final mixing step. The entire mixture is again transferred to the platinum crucible for re-heating in an air furnace for 2 hours at 1450 degree C, where it completes the reaction, releasing any remaining CO_2, producing the Leucite crystalline phase $K_2OAl_2O_3*4SiO_2$, and cools to a solid mass. After removal from the crucible, it is re-ground once more with mortar and pestle to produce the final particle-size powder distribution needed for coating substrates.

The particle-size powder distribution was found to be an important factor in minimizing defects, such as, warping, distortion and cracks on the alumino-silicate layer. Particle size separation was done with continuous screening and batch-sieving operations. Two powder sizes for the coating process are separated using wire sieves. Fine particle-size 400-mesh (< 37μm) powder is chosen as an initial pre-coat layer on porous tungsten substrates to penetrate, and lodge into the ~15μm diameter pores of the tungsten material. The main coating layer consists of a coarser particle-size distribution between 200 (75μm) and 270 (53μm) mesh powder. To further reduce contamination, mechanical vibration sifting is used through a stack of stainless steel wire sieves; on top is a 200-mesh wire sieve, followed by a 270-mesh and a 400-mesh, then a catch pan at the bottom. Typically, it has been convenient to produce powder batches of ~170grams.

IV. Coating Technique

There are many forming processes based on different forming techniques and principles. Each application method will require a consistency state of ceramic material, for example; dry (no liquid), granules, plastic body, paste, and slurry. Several examples of wet application methods include dipping, slushing, flow coating (enamel material applied to a metal substrate using a directed stream of material with drainage), wet spraying (compressed air through air gun), wet electrostatic spraying (100-200kV electric charge imparted to each particle for attraction to metal substrate), and electrophoretic deposition (consists of electric cell with the metal part as the anode and negatively charged frit particles attracted to the substrate forming a dense, thick, uniform, coating).

A thin layer of ceramic can also be formed by pouring an aqueous slurry onto a surface and then moving a blade over the surface to produce a thin film. This method has advantages of

better powder dispersion in low viscosity liquid and the complexity of product shape permissible. The coating technique developed applies a uniform adhering finish of potassium aluminosilicate mixed with de-ionized water to cover the large area concave surface of an 80% density porous tungsten substrate. Fine particle-size 400-mesh (<37μm) powder is chosen as an initial pre-coat layer on porous tungsten substrates to penetrate, and lodge into 15μm average-size diameter pores of the tungsten material. The main coating layer consists of a coarser particle-size distribution between 200 (75μm) and 270 (53μm) mesh powder.

Our coating process occurs in two steps. The initial step is to start with 400-mesh powder and mix it well in a glass beaker with high purity de-ionized water to form an aqueous slurry suspension. Very dilute systems of slurry material are often called suspensions. Because of the abrasive nature of ceramic powders, an alumina ceramic rod is used for all mixing procedures to limit contamination. The 400-mesh aqueous mixture is then applied with an eyedropper to distribute suspension droplets over the entire curved surface, effectively imbedding small particles of fine 400-mesh (< 37μm) aluminosilicate powder into the pores of the tungsten substrate as a pre-coat layer. After a 12-hour drying period under normal atmospheric and temperature conditions, the top layer of 400-mesh dry powder, which should appear opaque white when dried, is gently brushed off using an acid brush. Care should be taken with the tips of the bristles on the acid brush, as they can easily be severed, and particle contamination in the ceramic material will result in pluming during the firing phase leading to blistering or craters on the surface.

The next step is to apply the main 15-mil coating layer, consisting of a coarser particle-size distribution between 200 (75μm) and 270 mesh (53μm) potassium aluminosilicate powder, and mixing well with high purity de-ionized water to form a thicker slurry. To apply a 15-mil coating, ~ 0.05g/cm^2 of potassium aluminosilicate powder is needed, therefore, the 10cm diameter substrate of total surface area, 82.38cm^2, would need ~ 4.2g. A major difficulty in producing this main coating is due to the large diameter and non-planar surface.

A blade-scraping tool was designed, matching the spherical radius of the concave shaped substrate (minus 15-mil to match the change in spherical radius due to a 15-mil coating on top of this concave surface). The blade-scraping tool is made of 0.25” lucite plate material, ~ 4.5” in length to cover the entire substrate diameter, machined to 7.985” (20.82cm) spherical radius with a smooth knife-edge down the center, and the knife-edge has a 30-degree taper from the horizontal on both sides (Photo 2). The scraping tool is attached to a 0.5” diameter, 5” length

aluminum support rod (Photo 3). Accurate horizontal and vertical alignment of the scraping tool is essential to the coating process, with a tolerance of ~ 1-mil. A milling machine can be used for this step to take advantage of the digital positioning features that come as standard equipment on many of them. The aluminosilicate slurry is poured to cover most of the emitting concave surface of the tungsten substrate that has been positioned in a Lucite die mold (Photo 4) and clamped to the milling machine. Because the porous tungsten absorbs water readily, it will be necessary to frequently apply de-ionized water droplets to keep the mixture near aqueous in viscosity. Initiate the scraping action by hand rotating the scraping tool slowly at the base of the aluminum support rod and applying de-ionized water droplets and adding slurry as needed to cover depleted, or uncovered areas. The support rod should be shielded (heat shrink tubing works well) to limit gloved hand-to-metal contact, which can result in aluminum particle contamination and defects to the ceramic coating. Patience is required to steadily apply a uniform coating across the entire surface. The coated material gradually increases in viscosity as it dries and is nearly ready for the "final lifting" of the knife-edge tool off the surface. The scraping tool, as it rotates, will tend to accumulate slurry material at the knife-edge. It is best to raise the tool and remove most of this slurry mixture at the knife-edge surface before the "final lifting" of the tool off the surface, otherwise the excess material tends to stay on the coated surface as the tool is raised. An ~1-mil high thin track is typically left by the knife edge tool during "final lift off". This is usually not a concern because the track will disappear when the layer of alumino-silicate melts during the heat firing process. With accurate digital vertical position control provided by the milling machine, it is possible to reset the scraping tool each time after removing excess slurry material, and then continue to form the layer until achieving a uniform thickness. By slowly rotating the knife-edge tool, in contact with the slurry, to form a smooth layer, and letting the slurry slowly dry before removing the molding tool, a uniform 15-mil coating has repeatedly been achieved. After the coating process has been completed, the drying process should begin immediately.

V. Drying

In developing our coating techniques, the rate of drying and the particle packing density are essential factors of the forming process. If the rate of drying is too fast, shrinkage of the

ceramic slurry will cause coating defects to occur. Drying cracks, tears, and blisters can be directly attributed to incorrect drying rates. Rapid drying by vacuum or heating can leave voids in the coating layer that develop into defects due to shrinkage stress. A ceramic part dried too rapidly forms a hard surface film, which traps the moisture and leads to tearing at the surface. Hydrostatic forces, imparted to the mixture by water, are removed through the drying process. During the forming process there is a distribution of particle shapes and sizes, and particle packing density. A higher particle packing density will effectively reduce shrinkage. Shrinkage will occur during drying as the liquid between the particles is eliminated and the interparticle separation distance decreases. Forming the ceramic material at a lower liquid content and increasing the mean particle size will reduce drying shrinkage and thus reduce coating defects (Fig. 6). We have found that shrinkage defects occur using small particle size 400 mesh alumino-silicate powder for the main 15-mil coating layer, while shrinkage defects did not occur using larger particle size 200 mesh powder with proper drying techniques (Photo 5).

Common methods used to control the drying rate include humidity drying, microwave drying, and supercritical freeze-drying, among others. We have chosen to control the drying rate by controlling the humidity. For this task, an ordinary humidifier chamber was built by flowing dry air through a de-ionized water-filled flask to create water vapor flowing jointly with dry air into a flow-controlled manifold combining these two inputs to a 4-liter chamber (Photo 6). The air velocity is kept low for slow drying rate with two identical flow meter systems for each of the two inputs, dry air and water vapor. Each flow meter system consist of a 0.5 SCFH and a parallel 10.0 SCFH flow meter to improve control and adjustment. Humidity is monitored at the output tube of the chamber with a digital humidity meter connected to a tube elbow fitting. The 4-liter humidifier chamber should be pre-set at >90% humidity, ~ 5.0 SCFH water vapor as the only input before inserting the coated source. Immediately after coating, substrates are quickly placed in the humidifier chamber and slow dried over a period of several days by starting with ~ 90% humidity, gradually lowering the humidity in 10% increments, and ending with <10% humidity. This averages to a 10% increment change every 5-6 hours and careful monitoring is recommended as the drying rate can change depending on the size and water content of the porous substrate, the larger the substrate; the more the drying rate may change abruptly. Normally, the water vapor flow system is set to ~0.25 SCFH, and the dry air flow system is usually < 5.0 SCFH as the dry air input will vary depending on the desired humidity setting. Complete slow drying is essential, before heat firing in a vacuum furnace.

After the drying cycle, the coated substrate is removed from the chamber and any residue alumino-silicate powder on the non-emitting surfaces of the source is carefully brushed off with an acid brush. At this point, the uniformly coated source is ready for the final heat-firing step.

VI. Heat Firing

Heat firing is the final process step that produces a melted layer of alumino-silicate that bonds to the tungsten substrate. The bonding is primarily mechanical so substrate porosity is essential because a rough surface provides the basis for mechanical gripping. There is thermal expansion mismatch between the metal substrate and alumino-silicate because metals usually have a higher thermal expansion coefficient than ceramics. Typically, the lower thermal expansion coefficient leaves ceramic coatings in compression after heat firing thus reducing its tendency to failure. Conversely, coated convex surfaces or edges will be more prone to resultant stress damage at room temperature as these areas exhibit maximum strain. Surface cracks may form when material near the surface becomes brittle and differential shrinkage within the material produces stress that exceeds the tensile strength. Poor coating thermal shock resistance can lead to cracking and chipping at the surface. An effective way to enhance thermal shock resistance is to apply as thin a coating as possible with a high ceramic to metal bond.

Defects to the ceramic material can occur, such as, shrinkage cracking, vapor blowhole, edge rounding, slumping, hour glassing, blistering, and discoloration. Additionally, the vacuum furnace has inherent temperature gradients limiting the maximum size of substrates. Craters, or blowholes, will result from contamination such as dust or metal flakes on the surface or within the material vaporizing during heat firing.

The coated substrate is centrally placed in a vacuum furnace to reduce inherent temperature gradients. During the heat firing process, the vacuum furnace pressure is maintained in the 10^{-5} - 10^{-6} Torr range. Potassium alumino-silicate starts to melt at 1550 degree C. Typically, our heat firing process is to bring the temperature up to ~1565 degree C for 45 minutes at a heat-up and cool-down rate of 10 degree C per minute. Automatic temperature control is done with thermocouples up until ~1500 degree C, then two optical

pyrometers are utilized to achieve accurate temperature control near the melting point. An optical pyrometer employing the incandescent disappearing filament method was compared to a single wavelength infrared optical pyrometer. Many factors can affect the single wavelength infrared optical pyrometer reading, such as, field of view, focusing distance, and the changing emissivity of the melted surface. The K-alumino-silicate surface emissivity, $\epsilon = 0.91$, was calibrated by focusing on a blackbody, $\epsilon = 1.00$, in the vacuum furnace and then compared with a coated K-alumino-silicate material at the same temperature, 1570 degree C, in the heat zone region. Utilizing both optical pyrometers improved the accuracy of the temperature measurements to +/- 5 degrees C. The resultant phase structure is dependent upon the maximum heat firing temperature and the aluminosilicate coating thickness. Observed phase transformations, for uniform 15-mil coatings, range from glassy vitreous structure, ~1555C (Photo 7), snowy dull-like bubble structure, ~1565C (Photo 8), to crystalline needle-like structure, >1570C (Photo 9), and combinations of each. As an experiment, one particular substrate was intentionally coated with an uneven thickness distribution of between 10-mil and 20-mil and heat fired at ~1565C, and the observed phase transformations ranged from glassy vitreous structure, ~20-mil, snowy dull-like bubble structure, ~15-mil, to crystalline needle-like structure, ~10-mil (Photo 10). The glassy vitreous structure has desirable characteristics of lower defects and high current emission properties. Shrinkage of K-aluminosilicate coating after the heat firing step is ~30%, a 15-mil coating, for example, will shrink to ~10-mil.

The maximum heat firing temperature must be approached slowly to avoid overfiring temperatures. According to SEM analysis, overfiring causes crystal growth and depletion of potassium (Photo 11). The absorption depth of molten ceramic material into the porous tungsten substrate has been detected to depths of ~ 25-mil to ~50-mil. Tungsten substrates may be recycled and surfaces re-machined to grind away previous coating materials. It is necessary to remove all the previous coating material before re-etching because electro-chemical etching, using KOH solutions, does not etch evenly in areas with remnants of alumino-silicate coating.

VI. Storage

Finished alumino-silicate sources should be stored in a dry environment and kept away from potential contaminations. Ion sources that have been stored for several years were found to work as well as new ones. So far we have not been able to determine their storage shelf life.

VII. Conclusions

Previous attempts at fabricating ion sources, without following these outlined technical improvements, have resulted in uneven surfaces displaying surface tension effects, such as, shrinkage cracking and blistering resulting in uncoated bare tungsten regions throughout (Photo 12). After applying these significant coating technique improvements, ion sources have displayed much higher quality, uniform phase structure, and fully coated surfaces (Photo 13). As with any technique, there is always room for improvement. For instance, there appears a tendency for the accumulation of coating material at the center of a concave substrate causing a thicker coating region, ~1cm diameter, that is assumed to be due to gravity during melting in the heat firing process. It is believed that a thinner coating tolerance across the entire concave curved surface, ~10-mil, will improve this negative effect of gravity, along with slightly lowering the maximum heat firing temperature to adjust to a thinner applied coating thickness. Furthermore, optimization for any of these other processes is not complete. For instance, the drying rate takes several days, but conceivably this drying time could be considerably shortened and has not yet been fully investigated. Another optimization factor to be investigated concerns the total time at maximum heat firing temperature. It is reasonable to assume that shortening this total time at maximum heat firing temperature from 45 minutes to ~5 minutes may preserve more of the K+ alkali content and improve source lifetime. The effect on surface quality will be a determining factor.

VIII. Acknowledgements

I would like to acknowledge my sincere gratitude to all our LBNL staff whose experience and expertise has contributed to this effort, and in particular, Warren Chupp, Ralph Hipple, John Jacobsen, Tak Katayanagi, Wayne Greenway, and Ron Beggs.

IX. Bibliography

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Ceramic Film and Coatings, Wachtman & Haber, 1993

Ceramic Processing, James Reed, 1995

X. Vendor Information

Semicon Associates
695 Laco Drive
Lexington, Kentucky 40510
Telephone: (859) 255-3664
Contact Person: Jim Tartar
Website: http://www.semiconassociates.com/
Supplies: 1.00” diameter x 0.125” thick, 80% density tungsten disks, unit price $75, and 4” diameter HCX 80% density tungsten sources, unit price $1250.

Spectra-Mat
100 Westgate Drive
Watsonville, CA 95076
Telephone: (831) 722-4116
Contact Person: Anne Simmons
Website: http://www.spectramat.com/
Supplies: 1.00” diameter x 0.125” thick, 80% density tungsten disks, unit price $104.

Alfa Aesar
30 Bond Street
Ward Hill, MA 01835

Telephone: (800) 343-0660

Website: http://www.alfa.com/

Supplies: SiO_2: Silicon (IV) oxide, 1kg, 99.9%, -325 Mesh Fused Amorphous Powder, S.A. (Surface Area): $5m^2/g$, Part # 89709, CAS # 7631-89-9

Sigma-Aldrich

Telephone: (800) 325-3010

Website: http://www.sigmaaldrich.com/

Supplies: Potassium carbonate: K_2CO_3, 50g, 99.995%, Part # 36,787-7, CAS # 584-08-7

Supplies: Aluminum oxide: Al_2O_3, 500g, 99.8%, -325 Mesh, Part # 26,549-7, CAS # 1344-28-1

Figure 1: NTX K+ Aluminosilicate source current vs. diode voltage. Maximum ion source current density = 15.79 mA/cm^2

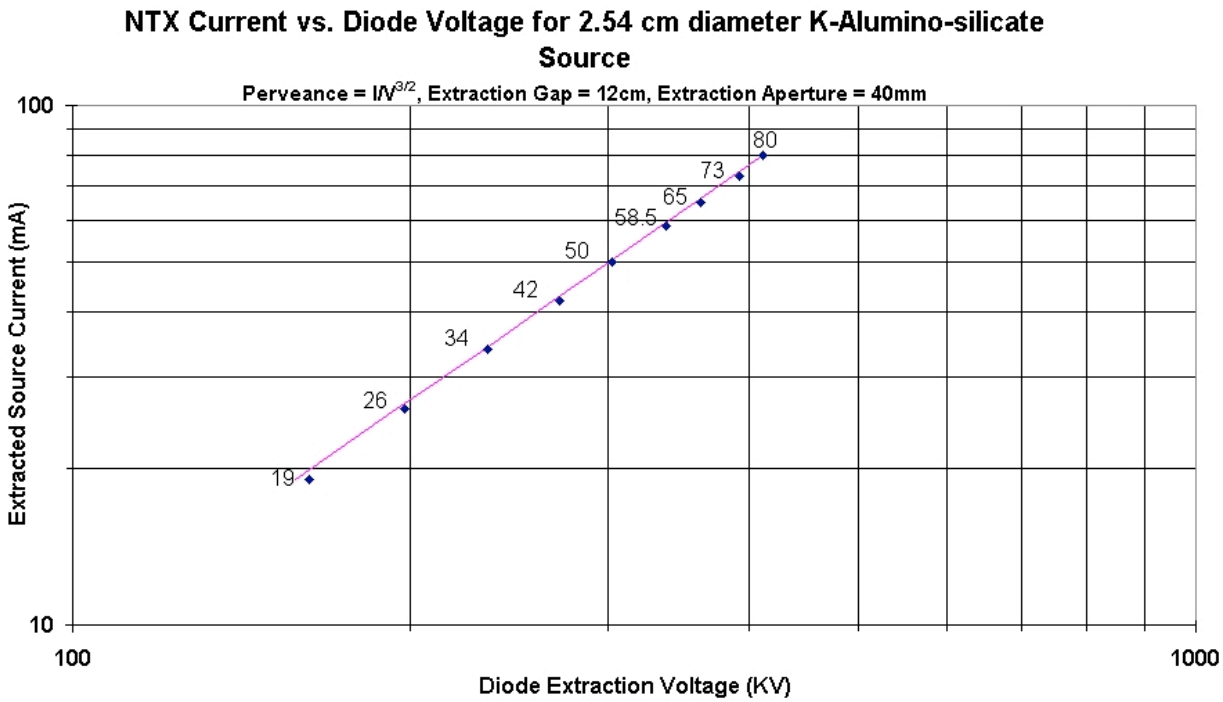


Figure 2: HCX K+ Aluminosilicate source current vs. extraction voltage. Maximum ion source current density = 9.2mA/cm^2

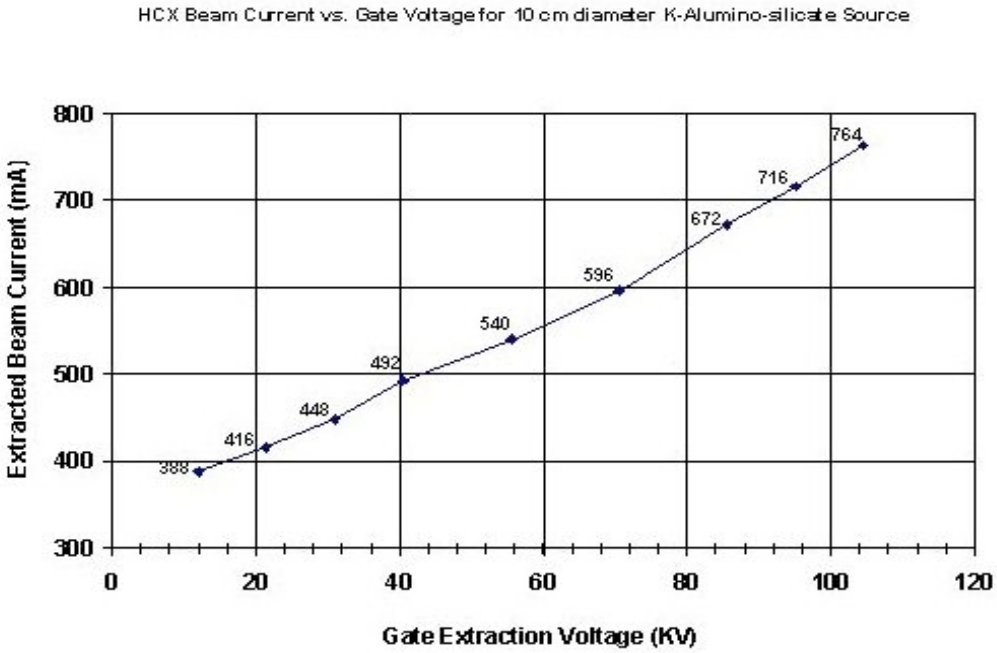


Figure 3: J – V curves at various temperatures for a fully activated 6.35 mm diameter K-aluminosilicate source after 2 weeks of continuous operation. Maximum ion source current density = 80mA/cm2

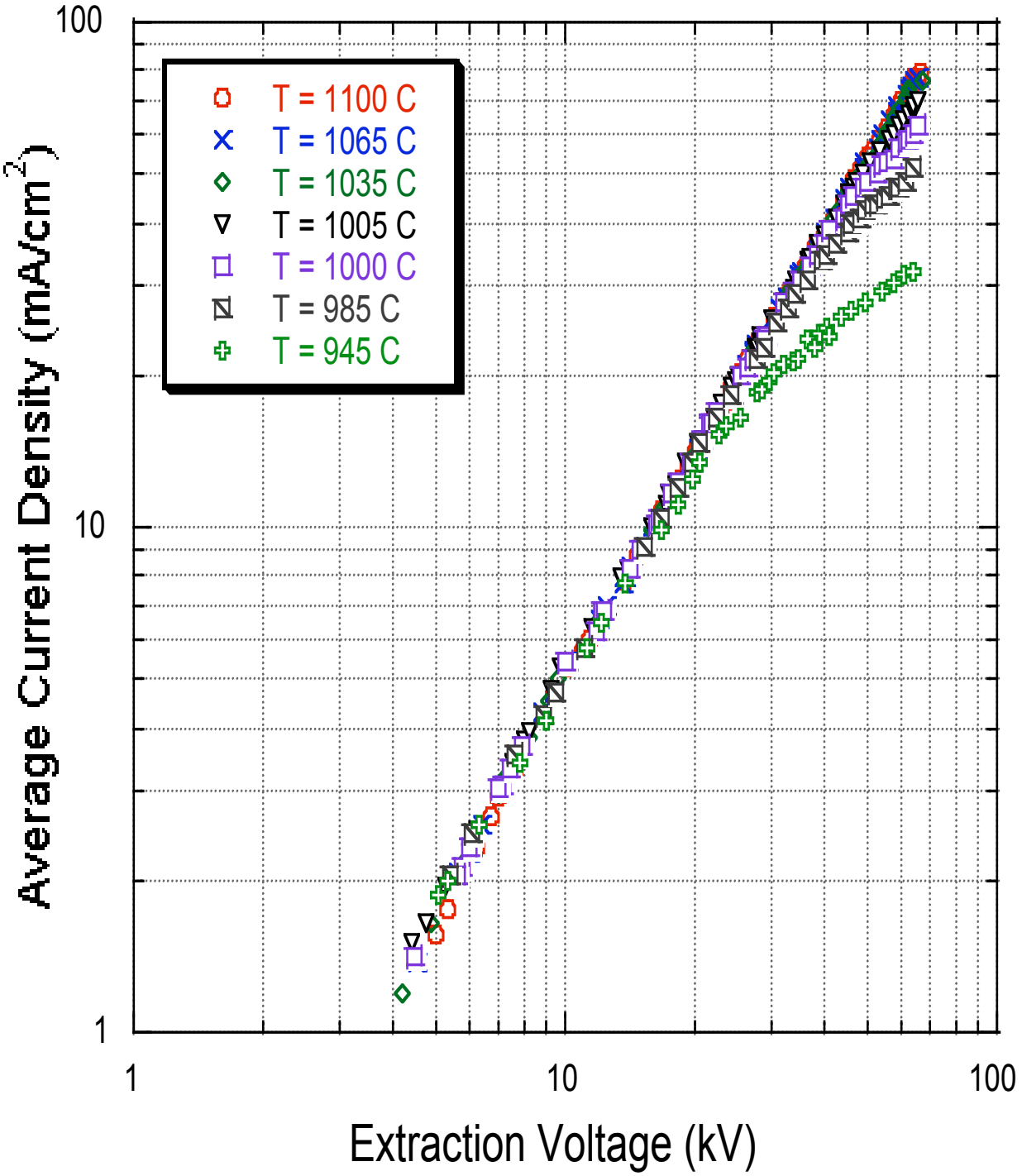
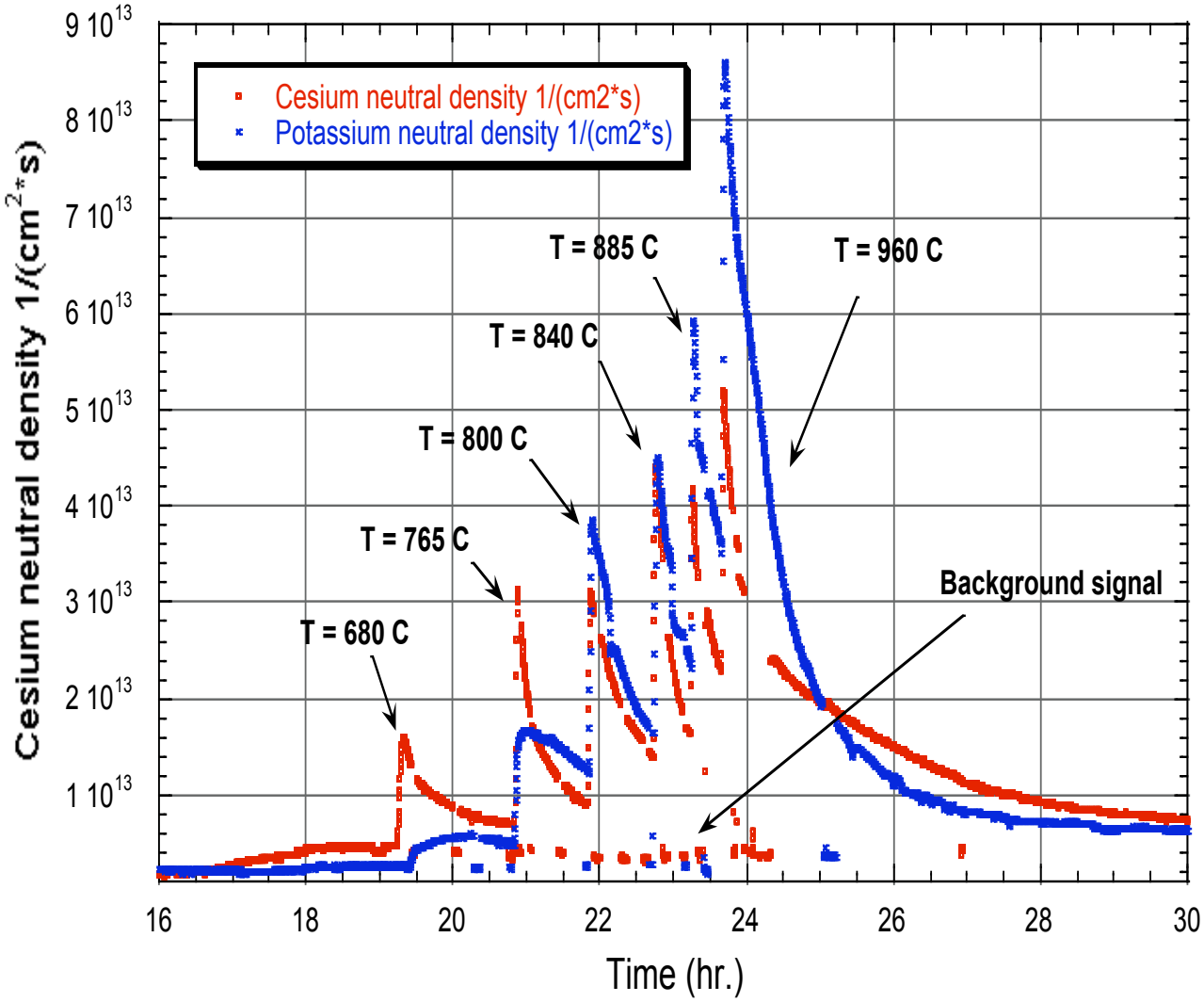


Figure 4 : Data set of a heating cycle for Cs and K contact Ionization sources measured with neutral detector.



Contact ionization source (doped source) neutral emission levels at operating temperatures > 1,000 °C are under 1 μA/cm2 current equivalent, typically between 0.3 and 0.4 μA/cm2, decreasing over time. In the case of the alumino-silicate ion sources, no neutral emission is detected during the heating cycle equivalent to that shown in this figure. Nevertheless, at high operating temperatures ~1,200 °C, neutral emission levels of a fraction of a μA/cm2 (~0.2) were observed.

Figure 5 : K_2O - Al_2O_3 - SiO_2 Phase Diagram

(Diagram reproduced from "Phase Diagrams for Ceramists" by the American Ceramic Society, 1964.)

Metal Oxide Systems

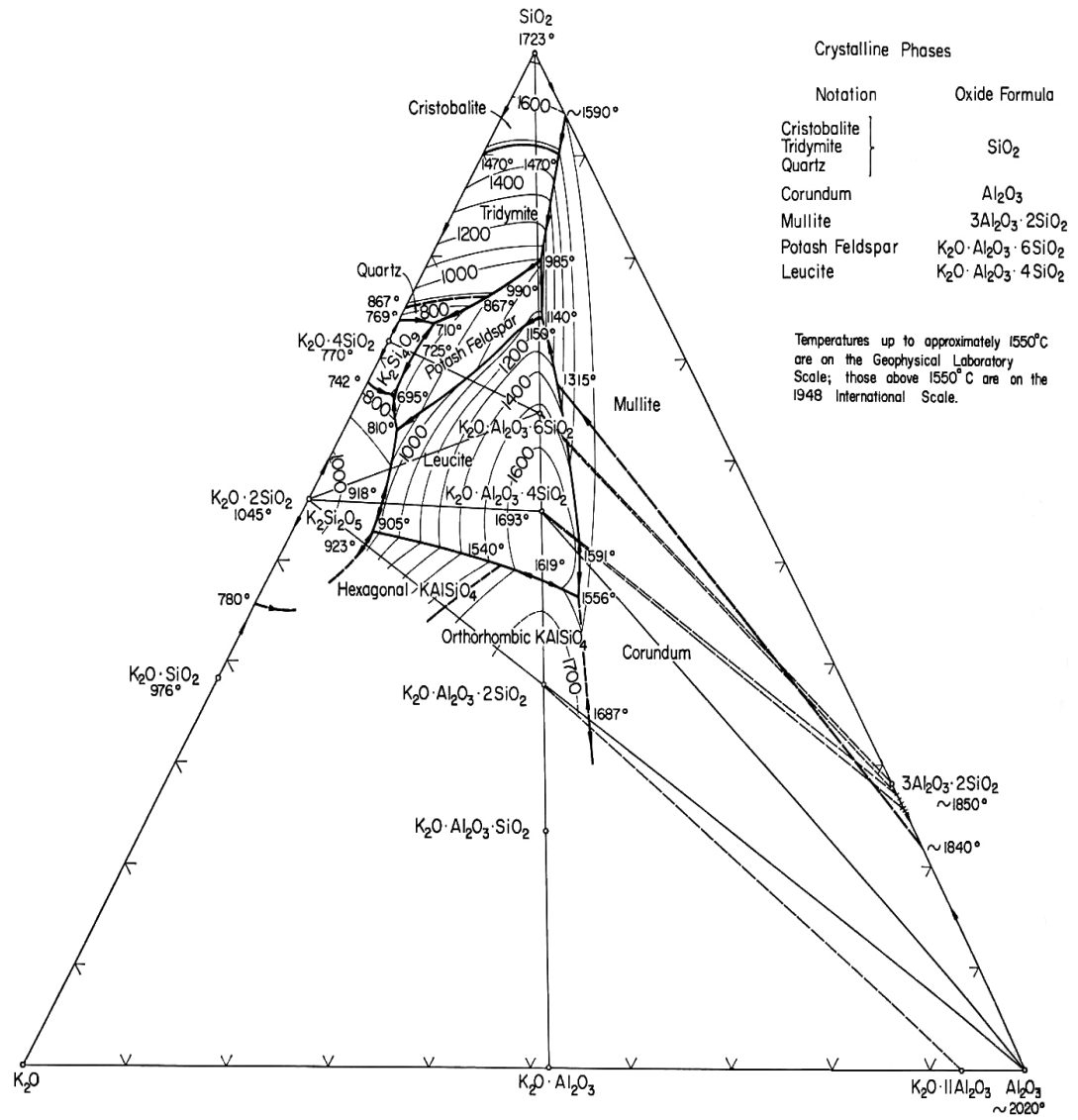


FIG. 407.—System K_2O–Al_2O_3–SiO_2; composite.

E. F. Osborn and Arnulf Muan, revised and redrawn "Phase Equilibrium Diagrams of Oxide Systems," Plate 5, Published by the American Ceramic Society and the Edward Orton, Jr., Ceramic Foundation, 1960.

Figure 6 : Drying Rate and Volume Shrinkage vs. Moisture Content
(Reproduced from "Introduction to Ceramics", W.D. Kingery, 1960, page 57.)

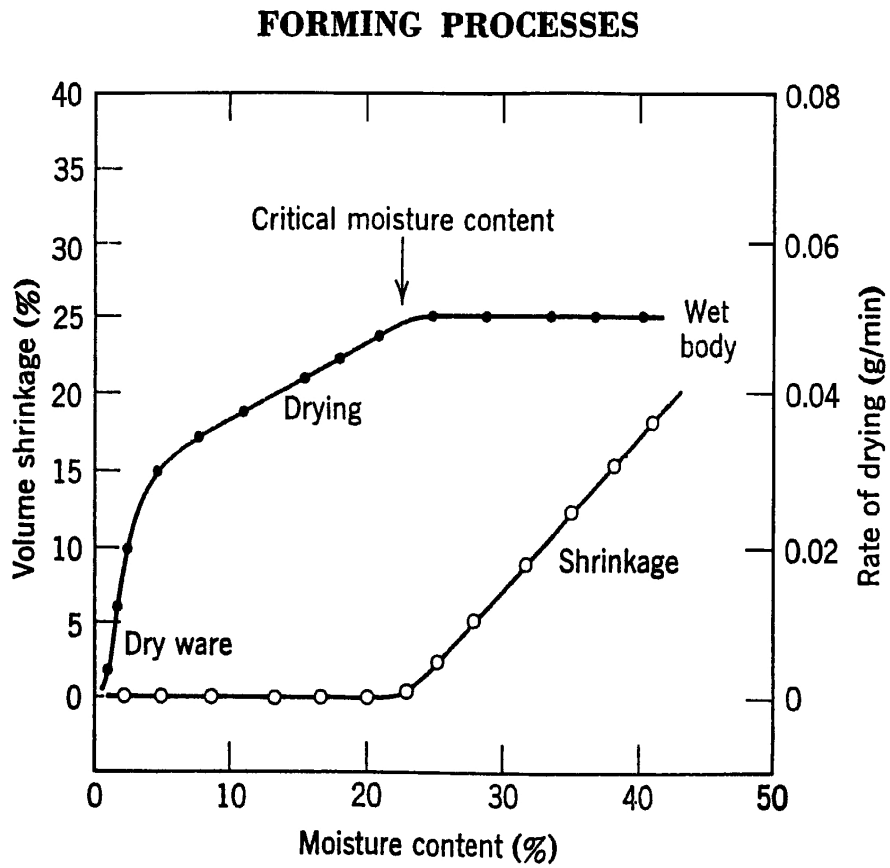


Fig. 3.20. Rate of drying and drying shrinkage for a ceramic body. At a critical moisture content which corresponds to the solid particles coming into contact, the rate of drying begins to decrease and the shrinkage stops.

Table 1 : Ionization Potentials and Work Functions of Several Elements

Element	I (Volts)
H	13.527
Li	5.363
Na	5.12
K	4.318
Rb	4.159
Cs	3.87
Ca	6.09

Element	Work Function(Volts)
Mo	4.2
W	4.52
Pt	6.35
Ir	5.3
Pd	4.97
Zeolite (Spodumene analog)	6.26

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Photo 1 : SEM photo of etched, porous, 80% density tungsten surface (50μm scale), with average pore size ~15μm.

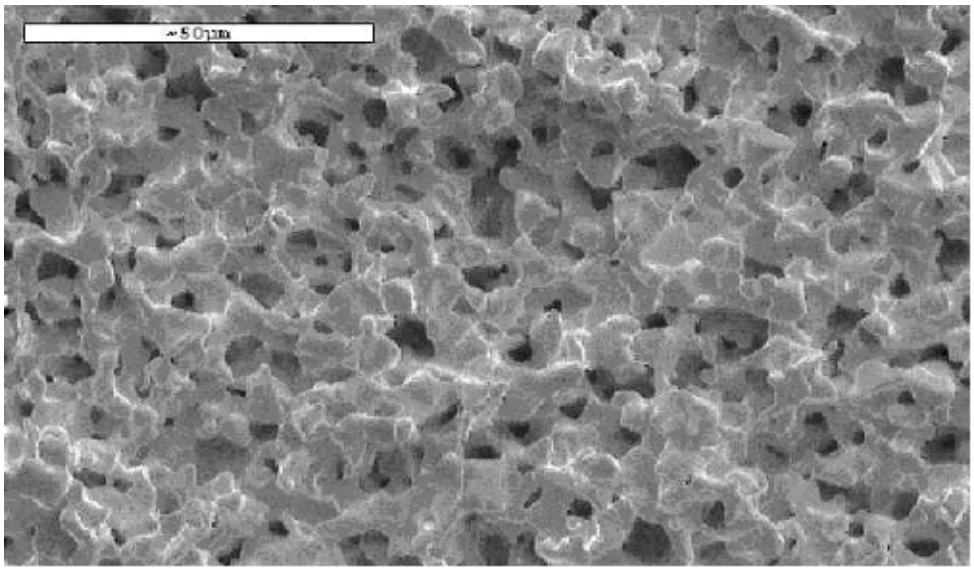

Photo2: Blade-scraping tool, knife-edge has a 30-degree taper from the horizontal on both sides.

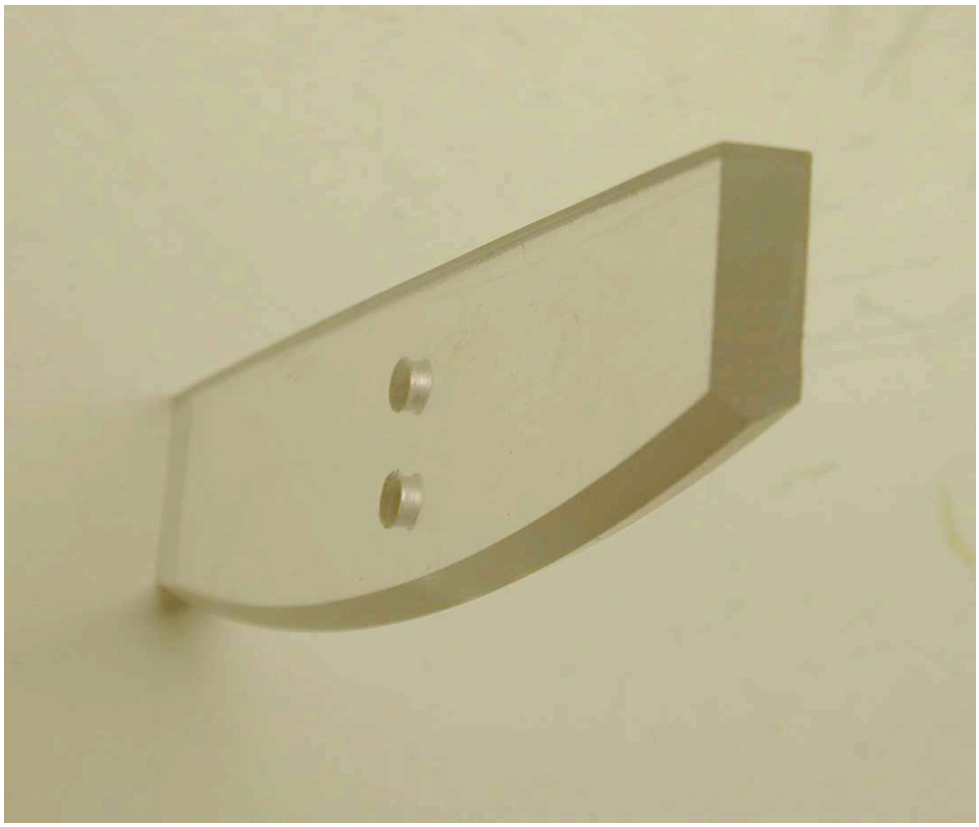

Photo 3 : Blade-scraping tool attached to a 0.5” diameter, 5” length aluminum support rod.

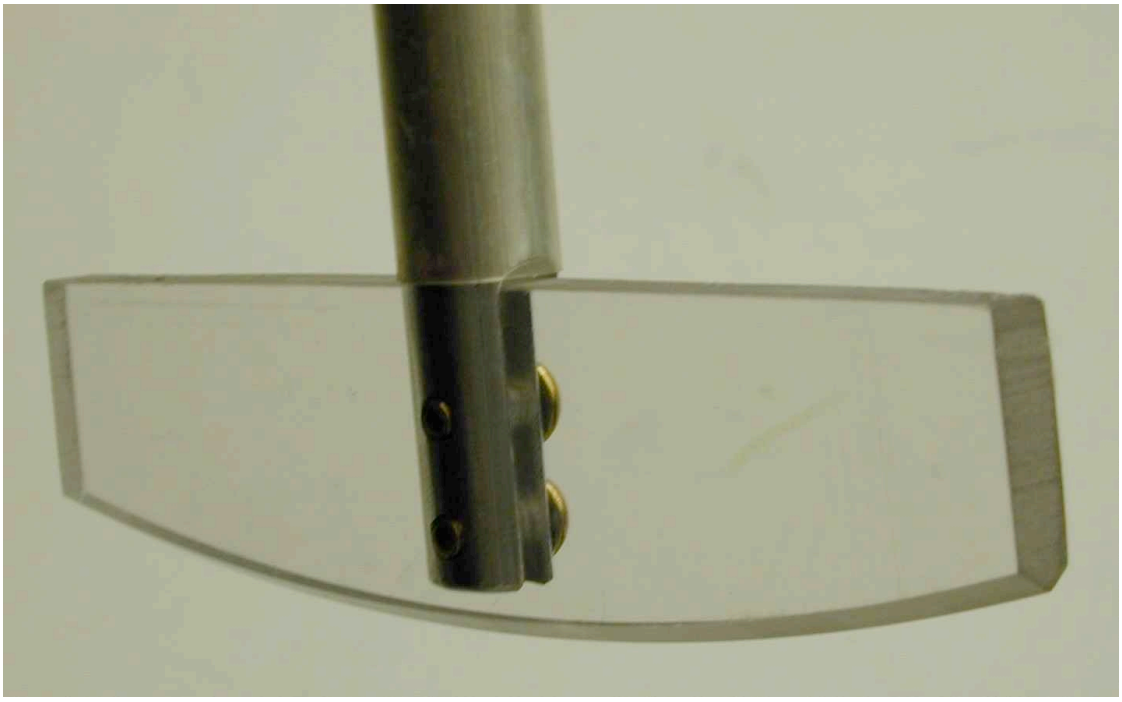

Photo 4 : Tungsten substrate positioned in Lucite die mold with coating.

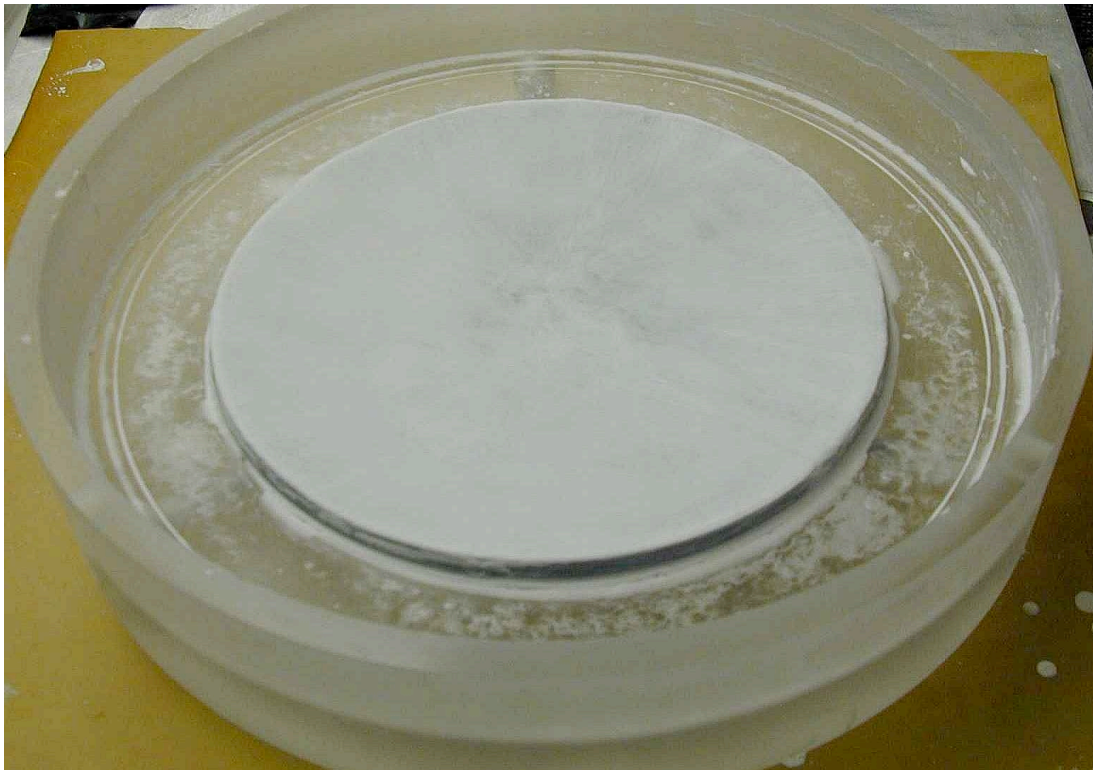

Photo 5 : Coating shrinkage defects induced by particle packing density.

400-mesh (37μm) particle size, 94% humidity, shrinkage tearing defects visible after < 2 hours drying time.

200-mesh (75μm) particle size, 94% to <10 % humidity, no shrinkage defects after complete 3 day drying cycle.

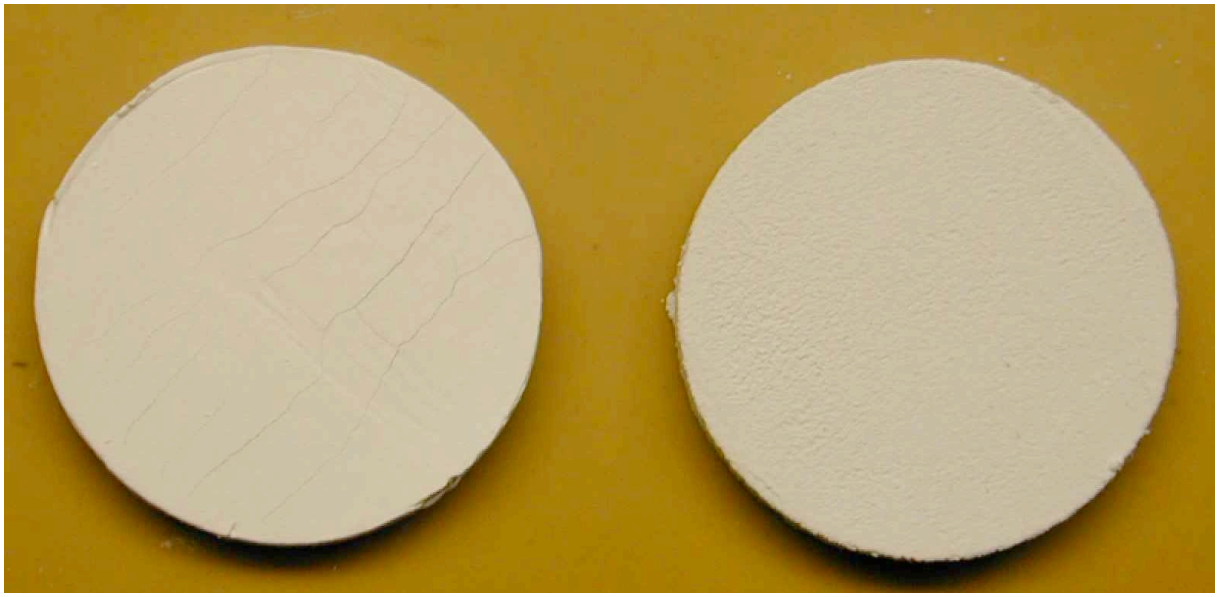

Photo 6 : Alumino-silicate drying apparatus, 4-liter humidity chamber.

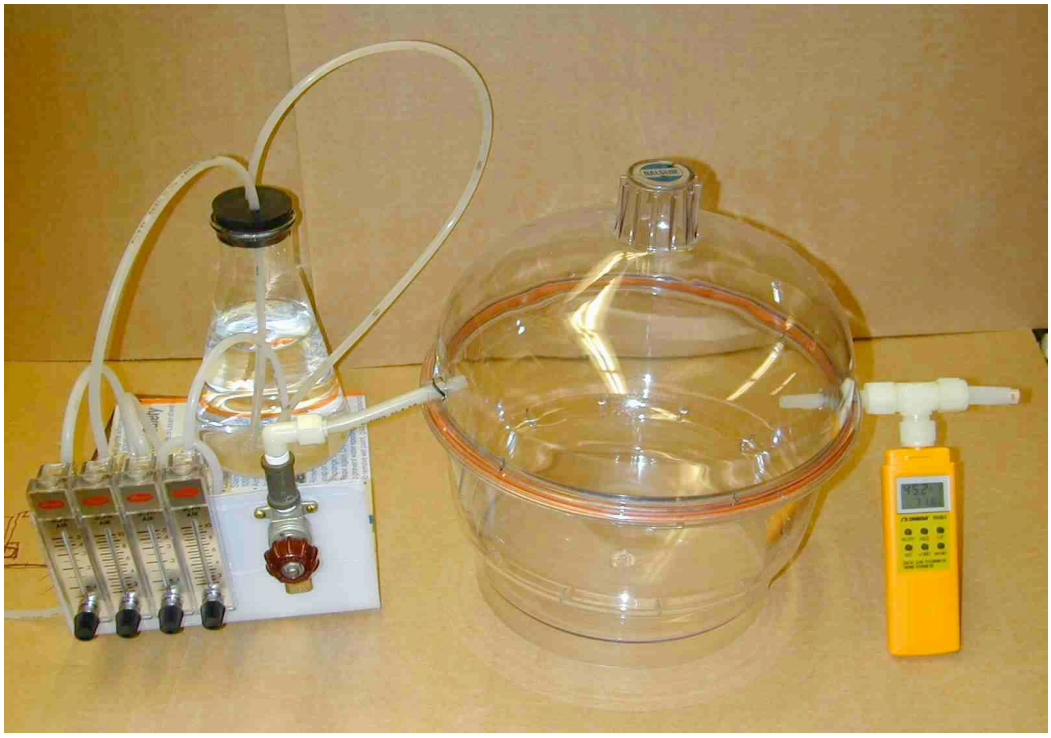

Photo 7 : K-Aluminosilicate phase transformation, glassy vitreous structure, ~30X magnification.

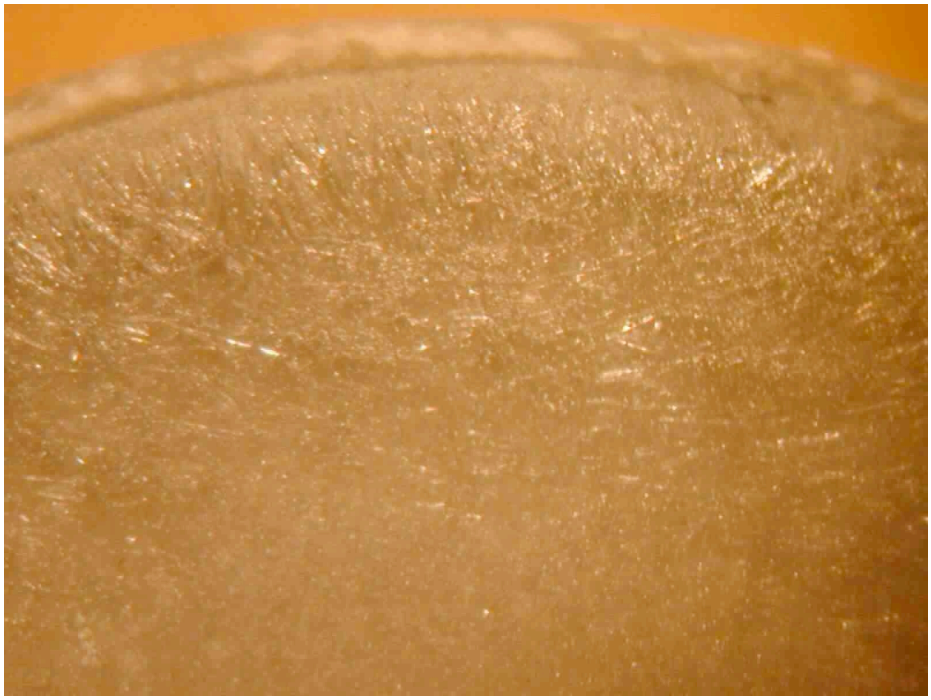

Photo 8 : K-Aluminosilicate phase transformation, snowy dull-like bubble structure, ~30X magnification.

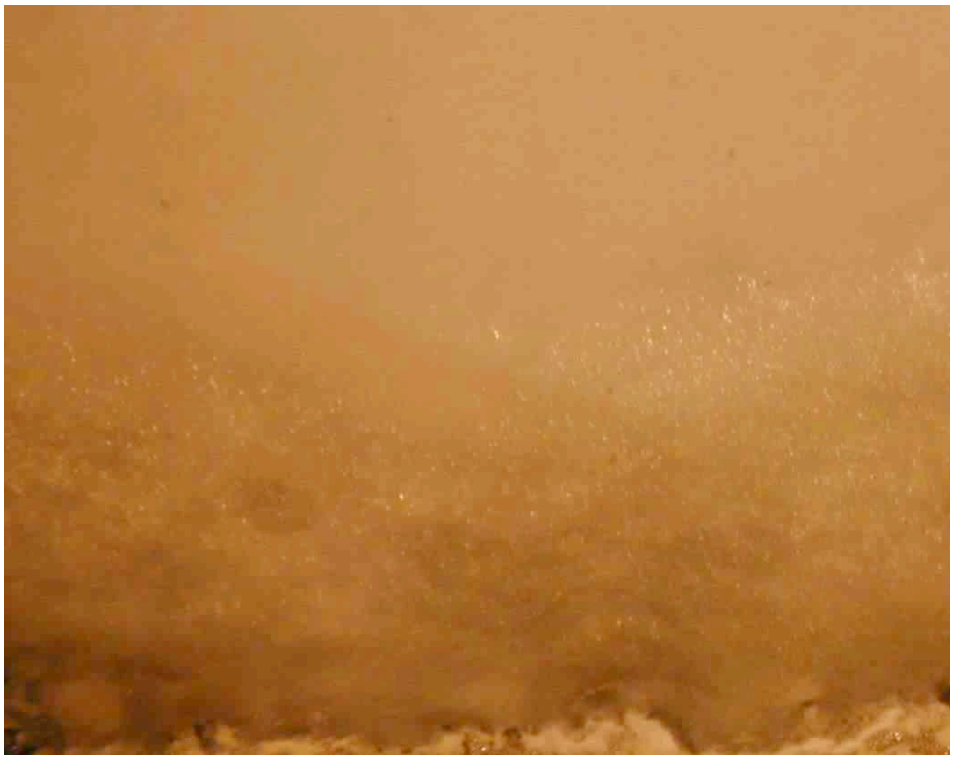

Photo 9 : K-Aluminosilicate phase transformation, crystalline needle structure, ~30X magnification.

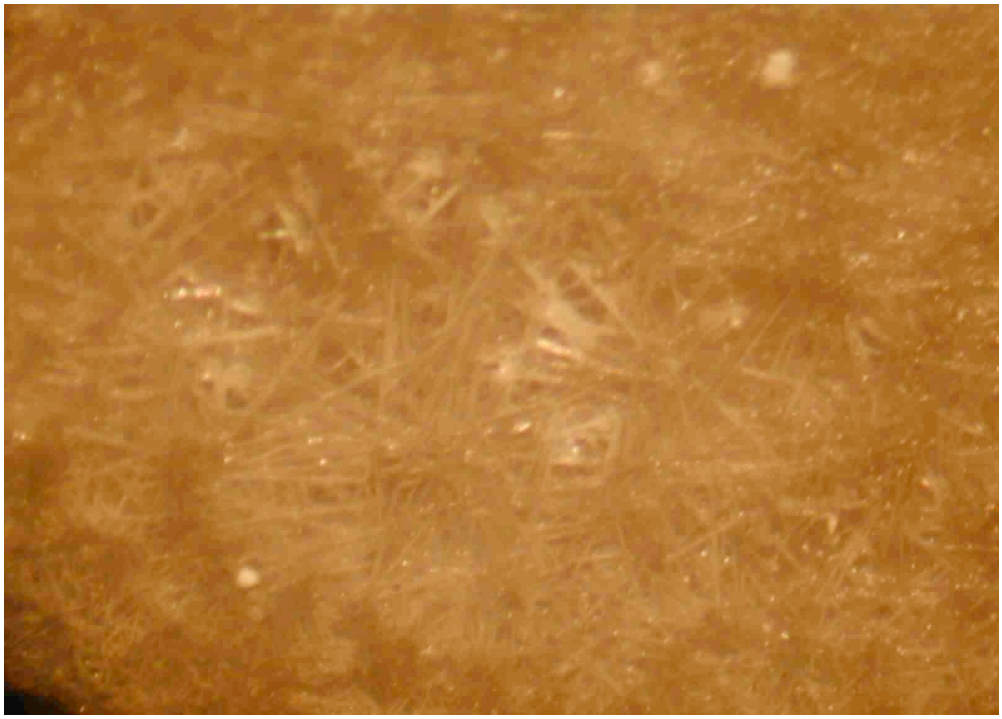

Photo 10 : K-Aluminosilicate coating thickness vs. phase structure on 10cm diameter substrate.

Photo 11 : SEM data of 1" diameter K-Aluminosilicate coated surface. The higher the maximum heat firing temperature, the lower the observed potassium content.

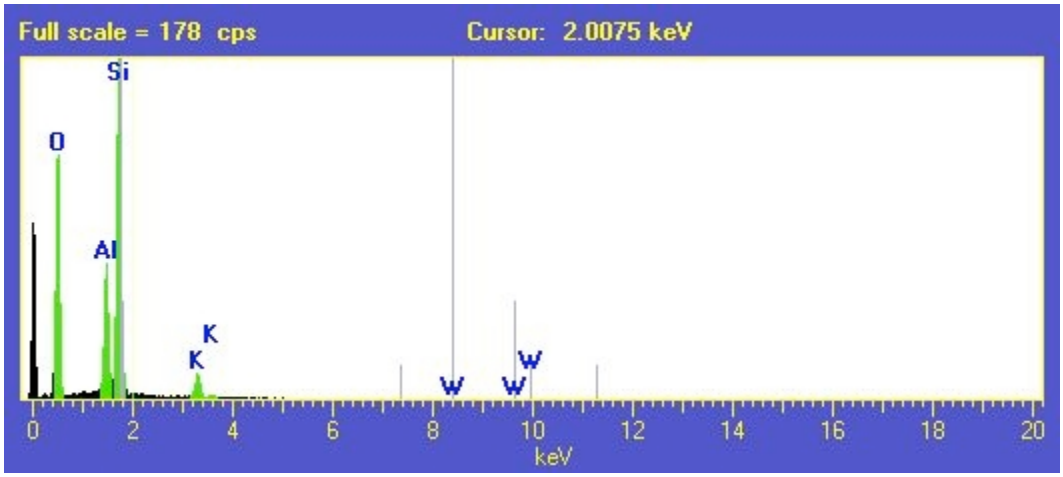

Photo 12 : Before coating technique improvements to K-aluminosilicate coated substrate. Surface tension effects and bare tungsten regions are evident on 10 cm diameter, 0.5 mm thick alumino-silicate coating on porous tungsten substrate.

Photo 13 : After significant coating technique improvements to fabricate 10cm diameter, 0.5 mm thick K-alumino-silicate coating on porous tungsten substrates for HCX source.