Slower Impedance Collapse with Hot Anode and Enhanced Emission Cathode

N. R. Pereira and A. Fisher

***Abstract*— As most high power diodes, our nominally 1 kA and 300 kV diode shorts out during the pulse. This so-called impedance collapse can be affected by the anode temperature and by surface conditions on the carbon fiber cathode. With the tantalum anode at room temperature, impedance collapse occurs after about 200 ns. Collapse takes 100 ns longer when the anode is hot, 800–1200 K. Covering the carbon fibers in the cathode with cesium iodide delays collapse by another 100 ns. These observations are consistent with pre-shot removal of loosely bound adsorbates off the anode by the high temperature, and with easier electron emission from the cathode out of smaller, less violent explosive emission spots.**

***Index Terms*— Electrode effects, heating, surface contamination, vacuum discharge.**

I. Introduction

SPACE charge limits the current in a vacuum diode to a maximum value that depends only on the diode's geometry and the voltage. In traditional low power vacuum tubes the current is indeed constant at a given voltage. However, at high pulsed powers [1] the current I usually increases during the pulse, while the voltage V decreases at the same time as dictated by the circuit. The result is a decreasing impedance $Z = V/I$. When the current increases rapidly, and the voltage across the diode vanishes, the diode has shorted and the impedance has collapsed. Impedance collapse is caused by plasma coming off the electrodes. The observations in this paper demonstrate that impedance collapse is delayed by changing electrode parameters such as temperature (of the anode) or electron emission properties (of the cathode).

The modest vacuum (1–10 mPa or 10^{-6}-10^{-5} torr) typical of pulse power devices ensures that any surface is covered with many layers of loosely adsorbed water molecules and hydrocarbons. These are knocked off the anode by electrons, or released when the anode surface suddenly heats up by hundreds of degrees after receiving a dose around 500 kGy to 1 MGy (500 J/g to 1 kJ/g) in the discharge [2]. The just desorbed atoms become plasma, which modifies the diode's space charge and eventually causes the diode to short.

Low power vacuum diodes usually have thermionic cathodes. These can emit a continuous stream of electrons at up to 10 A/cm^2. In practice the cathodes operate at much lower current density to extend their useful life time to many years. In contrast, high power diodes are single shot, and they usually have cold cathodes that emit electrons only when the electric field exceeds some lower limit [3]. In this case the cathode phenomena are substantially more complicated and not yet fully understood. It is thought that the rapidly rising electric field early in the pulse pulls the electrons out of the surface, in specific spots. Electric field enhancing metallic whiskers were once believed to be the main cause of localized emission, but at the present time one recognizes many more possible ways in which emission from specific points on a surface is easier than from neighboring points. Whatever the initial mechanism, once the initial current exceeds some limit it heats up the initial spot and its immediate surroundings. New electrons leave even easier, the electron current to the spot continues to increase, ions may start to neutralize the electron space charge, and eventually the spot explodes [4]. Material emitted from the different spots becomes a bumpy plasma that covers the cathode.

The various mechanisms on anode and cathode create plasmas that are dense and hot, and therefore under pressure. These plasmas expand into the diode vacuum, where they modify the space charge and change the impedance. Sometimes the change in impedance is represented by a changing anode-cathode distance $d(t)$ in an applicable formula for the vacuum impedance $Z = Z(d)$, e.g., for a one-dimensional (1-D) diode the Child–Langmuir law $Z_{CL} \propto V^{1/2}/d^2$. The rate of change in the impedance is then $dZ/dt = Z' v_c(t)$, where Z' is the derivative of the vacuum impedance with respect to distance d, and $v_c(t)$ is the instantaneous closure speed. Often the closure speed v_c is assumed to be constant during the pulse, and d decreases from its initial value d_0 as $d(t) = d_0 - v_c t$. At the closure time t_c the gap is zero, and the average closure speed is $v_c = d_0/t_c$. We refrain from quoting a closure speed, because our data are not good enough to verify that the closure speed is constant during the pulse. Even if we were to attempt such a measurement, it is not so clear which impedance formula applies because our diode geometry is neither point-plane nor 1-D.

Some early experiments [5] with electron beam diodes intended to drive electron beam inertial confinement fusion (ICF) explored the influence on diode impedance of electrode parameters such as material, surface conditions, and temperature. These experiments showed the same effect of anode heating as we see, namely, a delay in impedance collapse. Heating cathodes has not helped: at least up to 1700 K the impedance does not change with cathode temperature, [6] but still hotter cathodes apparently do work better [7].

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It is intriguing why these early experiments did not lead to universal acceptance of high temperature anodes in high power diodes. Perhaps the effect may not have been spectacular enough to make or break the electron beam fusion program that inspired the work, or perhaps in part because some claims to success [8] were based only on a single shot. Practical reasons may have contributed: some X-ray producing diodes use thin anode foils that are hard to keep straight when the foils stretch on heating.

Various more recent experiments have shown similarly positive results in other settings. Ion diodes needed for the light ion ICF program have been improved substantially [9] by heating, gold coating, and other measures that affect electrode surfaces [10]. High pulsed power microwave tubes also work better [11] with similar measures, notably heating of the entire diode and putting CsI on the cathode [12]. Related work on rep-rated vacuum arcs is also relevant [13].

II. Observations

The following figures show the current from shots with different treatments of anode and cathode. For the baseline shots, the diode is handled with the care characteristic of many pulse power laboratories. After leaving the setup open overnight the vacuum vessel is evacuated first thing in the morning by an oil-filled roughing pump. After reaching about 5 Pa (50 mtorr) the system is pumped to high vacuum with an 8 in cryopump at the end of a 2 m long and 200 mm diameter conduit. From an initial high vacuum pressure of around 100 mPa the pressure decreases to around 5 mPa (50 microtorr) after 2 h, when the shot takes place. In our diode, the cathode is a 13 mm ring of 8 mm long carbon fibers, placed at 19 mm distance from a 50 mm diameter tantalum anode. The voltage pulse increases to about 300 kV with a 50 ns rise time: later the voltage stays roughly constant. The pulse is reproducible to within 10%, much better than the 50–100% effects we find.

A variac and a transformer heat the anode with abut 200 W, driving 8 A ac current through a tungsten wire. The wire is embedded in a heat insulating backing to the anode. The assembly is mounted on a 300 mm long and 13 mm diameter thin-walled stainless steel tube held in place with six screws inside a slightly bigger support tube. Pressed against the anode is a chromel-alumel thermocouple that is insulated from the support tube with a ceramic tube holder. A window looks straight through the support tubes to the back of the tantalum anode, providing a satisfactory visual corroboration of the thermocouple's temperature readings. The current pulse is measured with a current transformer around the anode support.

Fig. 1 is the current versus time under three conditions of the electrodes. All three currents are the same early on. Later, in the space charge limited regime at full voltage, the electrode conditions affect the current. When the electrodes are treated as normal in a pulse power laboratory, i.e., no special care at all, the current in the diode is typically as shown by the dashed line. After the initial 50 ns the current rises slowly from about 0.7–1.0 kA in about 150 ns. At constant 300 kV voltage the increasing current corresponds to a diode impedance decreasing from 500 Ohm to 300 Ohm. During this time the diode current is limited by the space charge in an equivalent diode gap that slowly decreases, as the electrode plasmas enter the gap. Thereafter, at the collapse time, the current begins to rise rapidly. The plasmas have filled the gap, the impedance has collapsed, and the current rise time reflects the inductance and resistance in the remainder of the circuit.

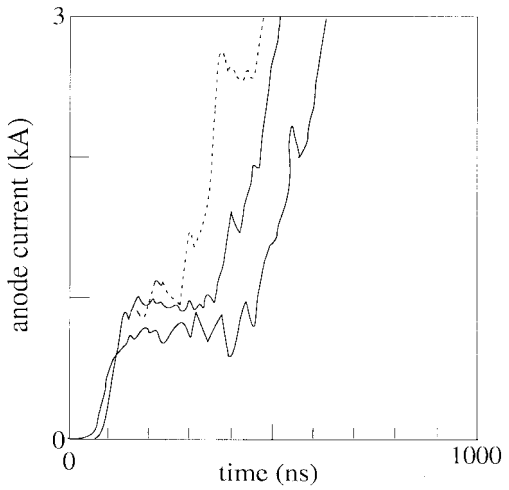

Fig. 1. Anode current for three cases: 1) (dashed) baseline, cold anode without CsI on the carbon fiber cathode; 2) (left-solid) hot anode without CsI on the carbon fiber cathode; and 3) (right-solid) hot anode with CsI on the carbon fiber cathode.

The left-most solid line in Fig. 1 shows that the diode stays open about 100 ns longer when the anode is hot, about 900 K. When in addition the cathode has a coat of cesium iodide the collapse is another 100 ns later, as seen by the right-most solid line. Compared to the 200 ns the diode normally remains open (dashed line), the diode with an anode at 900 K and cesium iodide on the cathode stays open twice as long, 400 ns. An experimental flaw causes the space charge limited current for the shot with CsI to be only 0.7 kA instead of the usual 0.9 kA: on this shot the generator's charging voltage happens to be 10% lower than intended.

Fig. 1 illustrates that simple measures such as heating the anode or putting cesium iodide on the cathode have desirable effects, at least for our diode with a relatively modest current and charge density. In the parameter regime explored there did not appear to be an influence of the rate of anode heating (rapid, in 10 min or so, or slow, over 1 h), the temperature (red-hot at about 900 K or orange at about 1100 K), or a delay in shooting (from zero to 1 h) while the anode is hot. These observations suggest that heating in this range affects the tantalum surface only, as expected from tantalum's known properties [14]. At our temperature, tantalum is a getter that absorbs hydrogen: removing all gases from tantalum's bulk demands up to 2500 K.

As in many high power bremsstrahlung diodes, in this experiment the anode surface melts as evidenced by a melt spot. Tantalum's enthalpy from 1000 K to incipient melt is about 400 J/g. A 400 ns pulse of 1 kA puts 0.1 mC into the tantalum, where the 300 keV electrons lose about 1.25 MeV-cm^2/g. To get to a surface dose sufficient for melting suggests a

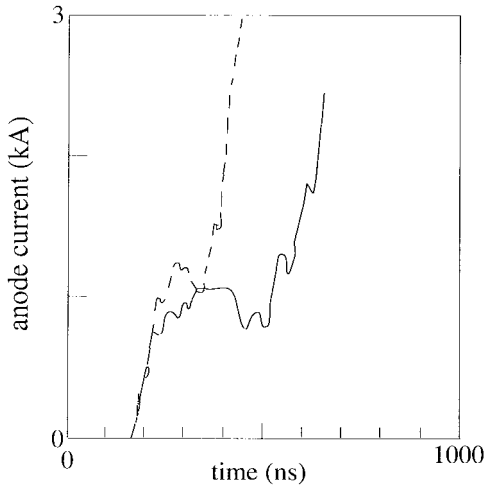

Fig. 2. Anode current for the first shot with a hot anode and CsI on the carbon fiber cathode (solid line), and a subsequent shot with a cold anode after opening up to air.

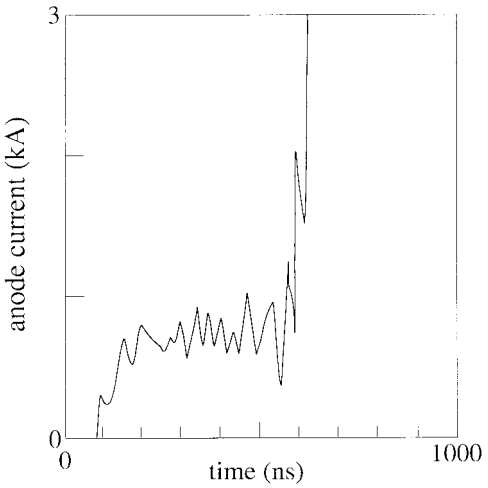

Fig. 3. Delayed collapse on the second shot with a hot anode and CsI on the carbon fiber cathode.

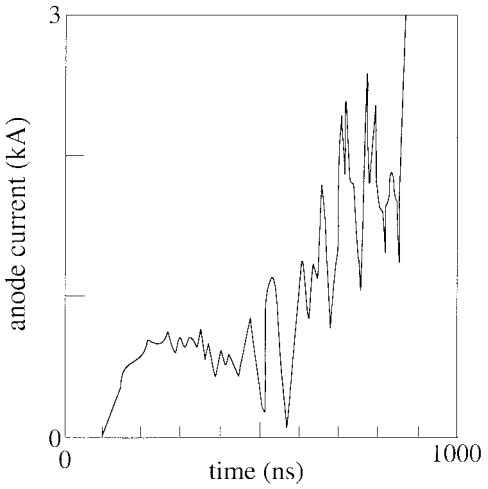

Fig. 4. Delayed and erratic collapse on the third shot with a hot anode and CsI on the carbon fiber cathode.

current density exceeding 750 A/cm^2. The actual surface dose is higher than estimated because under some circumstances up to half the electrons can scatter out of the tantalum back into the diode, from where they reenter the anode surface with much lower energy and much higher energy loss. After many shots the size of the melt spot is roughly 5 cm^2, which is consistent with the current density estimates and the likelihood that the melt spot moves around on different shots.

Tantalum's surface dose in the present experiment is comparable to the dose in typical high power bremsstrahlung diodes. Here the pulse is usually an order of magnitude shorter (40 ns, say) but with an order of magnitude larger (kA/cm^2) current density at an order of magnitude higher voltage (e.g., 1.5 MeV). The charge per unit area is then roughly the same as here. The larger voltage has a minor effect on the dose because the faster electrons penetrate proportionally deeper into the anode material. Therefore, our diode's parameters are quite similar to those of higher power and more interesting bremsstrahlung diodes that are much harder to work with.

Fig. 2 contains data from a series of ten sequential discharges. The solid line is the first shot in the best configuration, a hot anode (at about 1000 K) and CsI on the cathode. The current is fairly constant around 1 kA, with little or no change in impedance. After just under 400 ns or so the impedance collapses. The dashed line is the last shot in the sequence. This last shot is done after letting the diode up to air, having it sit for 1 h, and re-evacuating to the same pressure. The cathode is covered by whatever CsI has remained, and the anode is now cold. There is no visible effect of the nine previous shots: most of these had a hot anode, but steadily changing amounts of CsI on the cathode (some are shown below). It appears that a diode re-exposed to the atmosphere behaves as if the electrodes were fresh. Apparently, to get a delayed collapse the anode must be hot during the shot, while the history of the anode material is a minor effect.

"Conditioning" describes the gradually improving performance of many high voltage devices from a series of shots, or from exposure to gradually increasing voltages. In our test there is little or no conditioning for a hot anode when the carbon fiber cathode misses CsI: repeating shots without exposing the diode to atmosphere give substantially the same impedance behavior. However, when the same diode has its carbon fiber cathode covered with CsI, the impedance improves erratically on subsequent shots, reminiscent of conditioning. Figs. 2–5 contain some of the data. The solid line in Fig. 2 is the current on the first shot in a diode with a hot (about 1100 K) anode and a CsI-covered cathode. In this case the impedance collapses in slightly less than 400 ns. The solid line in Fig. 3 is for the second shot, done a few minutes later without opening the vacuum vessel. Now the impedance collapses 100 ns later, after about 500 ns. On the two shots thereafter, e.g., shot three in Fig. 4, the current is more erratic than usual, and the collapse time is not defined so easily. A best guess is a collapse time more than 700 ns. After two erratic shots the diode settles down: for the six shots thereafter, shots five to ten, the diode's collapse time settles into a reproducible 500 ns. The solid line in Fig. 5 is the current on shot ten, the last of these six nominally identical shots.

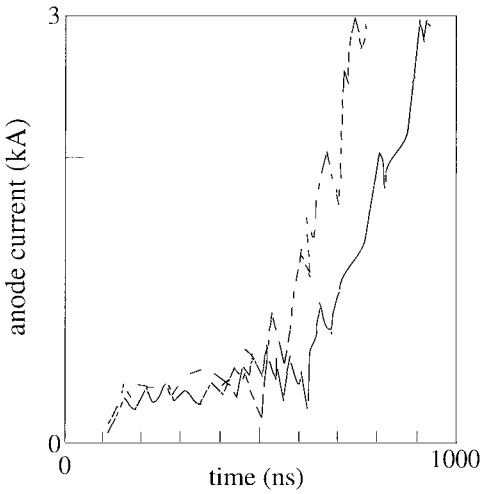

Fig. 5. Anode current while the anode is hot (solid) and while cooling down (dashed).

With the limited diagnostics available in this experiment it was not possible to identify the reasons behind the changes in diode behavior. For the moment, we expect that as yet ill-understood cathode processes will turn out to be responsible. The observations in this shot series are compatible with gradual changes in CsI coverage. Perhaps the cathode has developed an optimum coverage of CsI during the first two shots, which manifests itself in an unprecedented delay of collapse on the two erratic shots. The stable impedance behavior on subsequent shots is compatible with carbon fibers without CsI, the CsI having been removed in four shots.

The dashed line in Fig. 5 is for one final shot, taken with an anode temperature of 750 K while the anode was cooling down. The impedance collapse is about 100 ns faster than six shots with an 1100 K anode temperature and a reproducible collapse time of 500 ns (Fig. 5's solid line). Apparently, the diode deteriorates when the anode temperature falls below some threshold, perhaps because contaminants from the vacuum may have settled back down on the tantalum. Proof of a temperature threshold demands a future experiment, wherein many nominally identical shots still vary in a reproducible manner with temperature and with threshold-affecting parameters. One of these may be the pressure of the vacuum, which was not specifically recorded during this series. Another influence may be cleaning of the anode by the discharge itself. Electron stimulated desorption by individual electrons combined with high-temperature heating of the anode by the electron beam may have removed strongly bound oxides of tantalum, so that later shots would be affected mostly by anode contamination that is in equilibrium with the vacuum.

At room temperature the experimenter usually waits to shoot until the vacuum has reached an experimentally determined shooting range. This is typically a few mPa ("on the 10^{-5} torr scale"). As discussed further below, the gas remaining in the diode gap does not contain enough charge to affect diode behavior even at one or two orders of magnitude higher pressure than the shooting range. Therefore, an influence from vacuum pressure on diode behavior must come from the pressure's secondary effects. Of these the prime candidate is increased surface contamination: on any given material at a given temperature, the amount of surface contaminants increases with increasing base pressure.

A secondary effect such as increased surface contamination with higher pressure could be counteracted by higher temperature, because higher temperature decreases surface coverage by contaminants. When the anode is hot it should then be possible to shoot at higher base pressure, and this is indeed the case. Without CsI on the cathode, but with a 1200 K anode temperature and an order of magnitude higher base pressure (30 mPa or 3×10^{-4} torr) than usual, we observed that the current no longer has a rigorously constant region. Instead, the current increases slowly until the beginning of the collapse proper. The gradual increase in current is absent on the other shots, which are all taken at 2–5 mPa. The observation is consistent with higher coverage of adsorbates that are more loosely bound, as one might expect from the higher pressure. In future experiments we hope to explore this phenomenon further.

III. Discussion

Pulsed high power diodes are, lamentably, much less reproducible than most physics experiments. In part the problem may be extreme sensitivity to initial conditions, in which case reproducibility is hard to obtain even under the best of circumstances. Another part of the problem may be unintended variation of important parameters. Here we try to identify these parameters, so that they can be kept properly constant in future experimentation.

In practice it is already difficult to keep base pressure constant from shot to shot, despite its acknowledged importance: parameters whose importance is unknown are constant only by accident. This experiment reconfirms earlier findings [5], [8]–[10] of electrode temperature as an important parameter to be used to advantage in improving the behavior of the discharge. It is well understood why anode temperature affects the production of anode plasmas, by reducing surface and bulk contaminants. This experiment also confirms the benefits of using CsI on the carbon fiber cathode, first suggested in [11].

To a surface physicist, our high power vacuum discharge is a primitive desorption experiment that mixes electron stimulated desorption (ESD) and temperature programmed desorption (TPD) with a host of other mechanisms that each have their own acronym (a list of acronyms with short descriptions for the various techniques needs five pages [15]). For a good desorption experiment a purpose could be to measure binding energies. However, binding energies and even the surface structure of the many possible adsorbates on the many possible surfaces are already known: they fill a 135 page long table [16]. In pulse power equipment the surfaces are usually dirty and not well characterized (the so-called "technical surfaces") which makes it difficult to get reproducible data needed to understand the surface. Conversely, a badly characterized electrode surface makes it difficult to use modern understanding of surfaces to improve pulse power.

In this work we have kept in mind some simple considerations that are well known, but still often ignored in day to day practice. Older references such as [14], and newer ones as [17] and [18] are useful as a practical background, while

[15] and [16] are good examples of the state of the art in surface physics. For pulse power systems the role of electrode contaminants has been discussed in [9]: their attention to surface issues resulted in major improvements to ion beam diodes.

An anode in a high power diode heats up during the shot over a well-defined time scale, here a few 100 ns. The result is desorption of contaminants. In fact, temperature programmed desorption (TPD) is a well-known surface physics technique wherein a surface is heated slowly. During the temperature rise the pressure in the vacuum vessel shows one or more peaks. The temperature at peak pressure gives the binding energy of a specific contaminant, while the peak's integral relates to the amount of desorbed material. In TPD the temperature increases perhaps 1–10 degrees per second, while in a high power discharge the temperature rises a billion times faster, many degrees in 1 ns. Pressure gauges far away do not see the gases desorbed this rapidly, but they can be qualitatively inferred from the changing diode impedance. In our experiment heating the anode before the shot shows a pressure peak, as in TPD, but we did not feel comfortable to interpret the peak in terms of anode contaminants: desorption from a 50 mm diameter could have been swamped by material desorbed from the 1 m^2 area vacuum vessel wall that received radiation from the heated anode. In a future experiment we hope to use TPD for an estimate of reduced surface contamination on the anode before the shot.

Impedance collapse can not be caused by charge that could appear by ionization of leftover vacuum. The maximum pressure for a bremsstrahlung discharge is usually considered to be about 10 mPa (10^{-4} torr), but one typically strives for at least one order of magnitude better, 1 mPa (10^{-5} torr). A 1 cm^3 diode filled with 10 mPa hydrogen contains about 3×10^{11} particles and only about 50 nC, barely enough to sustain a 1 kA current for 1 ns. Therefore, a change in impedance can not come from ionization of the background vacuum. Instead, the vacuum influences the diode through its effect on the electrode surface.

One single layer of atoms has approximately 10^{15} atoms/cm^2. Evaporating one such layer into a 1 cm diode gap adds 0.4 Pa to the vacuum pressure. Therefore an electrode is a virtually infinite reservoir of atoms. When the electrode is clean the surface atoms are tightly bound (by the same forces that hold the metal together), and the surface atoms stay on the electrode when the temperature remains below sublimation. Unfortunately, at room temperature surfaces remain clean only with orders of magnitude better vacuums than the 1–10 mPa typical in pulse power work. Surfaces in pulse power systems are therefore always dirty, covered with an ill-defined mixture of water vapor, absorbed hydrogen and chemically bound oxygen (rust). The contaminant atoms come off the surface promptly during the power pulse. They immediately create a dense neutral layer above the electrode surface, which can rapidly ionize to form plasma that easily carries the current. The result is a change in impedance, and eventually impedance collapse.

How tightly bound are the foreign surface atoms and molecules [9], [15], [16]? On a clean crystal surface with a particular orientation, an atom or molecule has a specific binding energy E. These energies are electronic, about 1 eV/atom or 100 kJ/mol. Strong bonds between reactive molecule such as oxygen or CO and clean transition metals such as Ta or W are many 100's of kJ/mol, e.g., 800 kJ/mol for WO_3 (and correspondingly less per molecule of O_2). For hydrogen-metal bonds the energy per molecule of desorbed hydrogen is often around 100 kJ/mol. Binding energies of impurities on top of various stronger bound layers are usually an order of magnitude lower.

The binding energy determines how rapidly the molecules leave the surface by thermal processes. For a single molecule bound with energy E, the desorption rate is $A\exp(-E/RT)$, where $R = 8.3$ J/K-mol is the gas constant and $A = 10^{13}$/s is a typical time scale for motion on the surface. Strongly bound molecules with $E = 100$ kJ/mol have $-E/RT = -30$ at 400 K and a desorption rate of 1/s, while at 1200 K the exponent $-E/RT = -10$ and the desorption rate is almost nine orders of magnitude higher, 5×10^8/s.

Heating can be made more effective when assisted by suitable chemical reactions. As an example, stainless steel vacuum vessels are usually passivated by heating and flushing with oxygen, but the process is much more effective when using NO [18].

It is believed that the lightest neutrals will lead to the most rapidly expanding plasma. If this were true it would be particularly important to clean the surface of hydrogen. Hydrogen can get onto the surface in many ways, e.g., after diffusion through the metal itself, and be chemically bound as a hydride or weakly adsorbed as water on top of many layers of other contaminants. This water should be largely driven off by keeping the surface hot.

The same is not true for chemically bound hydrogen. Different materials have vastly different affinities for hydrogen and for water, so that a proper choice of electrode material should help in minimizing the amount of strongly bound hydrogen on the surface. Strong bonding of all kinds of molecules, including hydrogen, occurs especially to the transition metals. Their active surfaces make these elements good catalysts (e.g., Pt, Pd, Ni, and Fe: [16]), good getters (e.g., Ti, Zr, Ta: [14], [17]), and the (related) strong interatomic bonding makes them refractory (Ta, W, Mo). Very high temperatures are needed to clean these transition metals: tungsten filaments are commonly cleaned by flash heating to 3000 K [14], [17].

For a bremsstrahlung diode the usual material is tantalum $(Z = 73)$. Tantalum is chosen because its high atomic number gives high radiation production efficiency, because of its ease in making thin foils (compared to tungsten), and because of its high melting point. Unfortunately, tantalum is an excellent getter for hydrogen and has a strongly bound oxide (used to advantage in electrolytic capacitors). The metal's properties depend on fabrication processes such as heat treatments, distillation or densification in an inert or vacuum environment, and degassing, but in practice the metal is used as received from the manufacturer, and possibly after many years of storage in an uncontrolled environment. In contrast to what used to be common practice [14] in the now-defunct vacuum tube industry, tantalum is not usually degassed or otherwise treated before use in a high power diode.

For a high intensity diode where the anode evaporates completely, a good alternative to tantalum is gold. Gold can be applied as a thin coating, and its beneficial effect for pulsed power electrodes has been well documented [9][1]. If a gold coating can not be done, bismuth might well be a good alternative. Bismuth adsorbs only oxygen, which might be chemically removable in situ by heating in a background of hydrogen (which is not absorbed by Bi). Bismuth is also a good radiation converter because its atomic number $(Z = 83)$ is the highest of all the radioactively stable elements. An unfortunate characteristic of a nonrefractory Bi anode is a more violent explosion from faster evaporation by the intense electron beam.

The discussion here mentions some of the extensive literature data that should help in understanding anode plasmas and their suppression in a predictable fashion. Unfortunately, cathode plasmas are not well understood, so that creative speculation still has a place in suggesting ways to suppress cathode plasmas. Our suggestions assume that field emission spots lead to explosive emission, which in turn produces plumes of ejected plasma. A larger number of smaller spots would result in a slower, more uniform layer of desorbed gas or plasma that would help to retard impedance collapse. Minimizing light atoms such as hydrogen in favor of heavier ions should help too. Both arguments support a coating of CsI on the cathode, and in our experiment the cesium iodide on the cathode clearly delays diode collapse. All shots with cesium iodide on the cathode show a remarkable amount of fluctuation in the current trace, as might be expected from explosive emission that forms plasma jets shooting into the diode. A logical extension is to speculate that cathode plasma might be suppressed altogether by using e.g., atomically clean thoriated tungsten hot enough to carry at least the early part of the current pulse in thermally emitted electrons.

IV. Conclusion

In agreement with previous work [5], [8], [10], and especially [9], this experiment clearly shows that a high power submicrosecond discharge is influenced by electrode parameters that affect the electrode surface. The theory is far from complete. Heating the anode may reduce the amount of gas that is desorbed, and thereby reduce the amount of plasma exploding into the diode vacuum. The mechanism for CsI's effect may be that covering the cathode with CsI produces slower and/or more uniform cathode plasma through easier emission at lower electric field. Combined implementation of these two improvements to the electrode surfaces, heating of the anode and CsI on the cathode, leads to longer pulses in our diode, and may result in longer pulses for still higher power diodes as well.

Acknowledgment

The authors appreciate the help of Dr. S. Gold at NRL, who put NRL's Long Pulse Accelerator at their disposal through a CRADA agreement.

[1] PIIR-9-80 (8) mentions improved operation of a bremsstrahlung anode when the tantalum is covered with 350 nm copper overlaid with 100 nm gold, but has little detailed documentation. Various other papers by R. D. Genuario repeat these claims.

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