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Regeneration of electroformed metal–insulator–metal devices: a new model

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Abstract. The electroforming of Cu–SiO_x–Cu sandwich devices has been observed in ultrahigh vacuum (UHV) in devices with highly porous insulating layers. This shows that filament creation and regeneration are not dependent upon the presence of organic adsorbates and thus the filaments cannot be carbonaceous. Electroforming has also been observed at cryogenic temperatures. The time dependence of the regeneration of these filaments at room temperature has been studied and successfully modelled by assuming that a dynamic equilibrium exists between the rates of filament rupture and regeneration. The growth of the emission current, during filament regeneration, has been studied for the first time and it is found that low-voltage, room temperature emission is linked to the rate of filament regeneration.

1. Introduction

The electroforming of metal–insulator–metal (MIM) devices leads to the growth of filaments across the insulating layer [1]. The voltage-dependent rupture and regeneration of these filaments causes a region of voltage-controlled negative resistance (VCNR) in the I–V characteristics of the device [2, 3] when ramping the voltage up or down. The filaments could be created by the electromigration of metallic fingers into the insulating layer [1] or they could be carbonaceous: created by the decomposition of organic vapour which is assumed to be present due to residual pump oil in the high-vacuum systems usually used to manufacture devices [3]. The need to resolve this controversy has been highlighted by the recent suggestions that hydrogenated-amorphous-silicon-based barrier structures might also conduct in a filamentary manner [4] and that electroformed MIM devices could be utilized as 'electronic catalysts' [5]. However, attempts to detect the filaments directly by transmission electron microscopy (TEM) [6, 7] have failed because the spatial resolution obtained was inadequate. Hence, it is necessary to use less direct methods to investigate the nature of the filaments.

The carbonaceous model was first proposed by the Darmstadt group [3, 8, 9] as a result of finding that both planar (gold-on-quartz) devices [8] and sandwich (Au–SiO_x–Au) devices [9] were incapable of electroforming in ultrahigh vacuum (UHV) and that dosing either type of device with organic vapours tends to produce a large increase in device conductivity [9, 10]. The forming of MIM devices requires two processes to take place:

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the creation of pores in the insulator into which filaments can grow and the growth of the filaments themselves. Blessing *et al* [8] called the former process basic (or B-type) forming and the latter adsorbate (or A-type) forming (since they postulated that the growth of filaments was due to the decomposition of carbonaceous adsorbates). They assumed that UHV not only prevented A-type forming occurring but also inhibited B-type forming.

The Darmstadt group further propose that regeneration of filaments requires the presence of a continuous supply of organic vapour so, for instance, transferring devices, formed in a high-vacuum system, into a UHV system prevents broken filaments from regenerating [9]. They assumed that the filaments consist of a chain of carbonaceous links and that regeneration requires the decomposition of organic vapour to fill the 'holes' that appear in the chain as a result of previous operation at higher voltages [6, 11]. They postulated that the probability of a given filament 'hole' becoming filled at time t' is proportional to $\exp\{-t'/\tau_f\}$ where τ_f is a characteristic time for filament repair. Thus, the probability of a given filament conducting at time t after a switch from higher voltages is given by

$$P(t) = [1 - \exp\{-t/\tau_f\}]^\alpha \tag{1}$$

where α is the number of 'holes' in the filament. This model is found to predict the behaviour of room temperature regeneration well if $\alpha \simeq 1$, but is incapable of correctly predicting regeneration at lower temperatures (<220 K). Recently, an alternative model of cryogenic filament regeneration has been proposed, based on 'concerted regeneration'—the regeneration of filaments close to nearby intact filaments due to the local high temperature caused by power dissipation [12]. This model, which does not assume that the filaments are carbonaceous, fits the low-temperature characteristics well.

A number of other results do not appear consistent with the carbonaceous theory. The electroforming of planar (gold-on-SiO_x and gold-on-glass) devices at a pressure of 10^{-9} mbar has been reported [13]. Furthermore, it is found that controlled dosing with diffusion or rotary pump oil vapour does not produce an increase in device conductivity [10]. Indeed, bell jar evaporators of the sort used to manufacture most devices probably do not contain substantial quantities of pump oil vapour. Such evaporators are also used to manufacture MIM devices for use in inelastic tunnelling spectroscopy (IETS), a technique that is capable of detecting submonolayer quantities of oil and, in order to detect such oil, it is found to be necessary to deliberately dose some into the vacuum system [14]. Also, the I–V characteristics found when deliberately dosing controlled quantities of organic vapour onto a formed device in a UHV chamber do not tend to show smooth VCNR which would be expected since the amount of such vapour should be able to be controlled to match that present without having been intentionally introduced in other vacuum systems. In addition, it is not clear how organic vapour can be decomposed to create carbon. It has been argued that local temperatures as high as 4000 K can be obtained prior to the existence of a filament [15] but this seems implausible especially in sandwich devices where there is no need to create a microslit by Joule heating.

On the other hand, it is well known that metallic particles electromigrate in the presence of a sufficiently high electric field, thereby making the incorporation of metal into the insulator plausible. The incorporation of metallic particles into MIM tunnel junctions (with insulating layers <50 Å thick) has been implicated in giving rise to ageing effects [16–20] and is known to eventually lead to dielectric breakdown [21]. A number of studies have found that metal is incorporated into the insulator during forming, e.g., aluminium compounds and chains of SiO_4 have been found in the insulating layer of Al–SiO_x–Al compounds [22] and gold has been found in the insulating layer of Al–Al_2O_3–Au devices [23, 24]. Also, it has also been reported that other Al–SiO_x–Al devices would not be

formed unless a small amount of copper was deliberately incorporated into the insulator during manufacture and even then annealing prior to applying a voltage prevented forming [25]. In addition, the ability of an MIM device to form is strongly dependent on which metal is used in its construction [1] as would be expected if metallic filaments needed to be created.

In this paper we present evidence of a metallic filamentary model. (We shall use the term 'metallic filaments' here and below to mean filaments that are created by the incorporation of metal into the insulating layer. These might be purely metallic or involve some interaction between electromigrated metal particles and the insulating material.) We report the electroforming of MIM sandwich devices with porous insulating layers in UHV. Hence, we postulate that, although adsorbates may play a role in the creation of pores (B-type forming), they are not required for the production of the filaments themselves. Thus, the filaments cannot be carbonaceous. In addition, we propose an alternative model for regeneration based on a dynamic equilibrium between the rates of filament rupture and regeneration rather than on continuous carbonaceous adsorbate decomposition. Also, we report the effect of filament dynamics on the emission current. It is found that a link exists between low-voltage electron emission and the rate of filament regeneration.

2. Experimental procedure

Cu–SiO_x–Cu devices were manufactured by successive thermal evaporation of copper, silicon monoxide and copper films onto pre-laid contacts on a glass microscope slide in an ultrahigh-vacuum (UHV) system. The UHV chamber (the upper part of a two-level system) was pumped by an oil diffusion pump with a liquid nitrogen cold trap. This was normally operated at a pressure of 5×10^{-8} mbar although it could be baked to achieve a pressure of around 7×10^{-10} mbar (i.e. true UHV).

The sample was held in place by a purpose-built sample holder which was capable of being cooled by liquid nitrogen. Thermocouples were attached to both the sample holder (which attained a minimum temperature of about 80 K) and the front of the glass slide (which attained a minimum temperature of about 125 K).

Copper was evaporated from a molybdenum boat (R D Mathis ME8) and silicon monoxide from a tantalum baffle box (R D Mathis ME1), using a purpose-built UHV evaporator. The sources were outgassed prior to use, allowing evaporation to proceed (in the unbaked case) with an insignificant pressure rise. During silicon monoxide evaporation, air was let into the system via a leak valve to pressures up to 5×10^{-5} mbar in order to try to prevent shorting of the insulating layer due to pinholes during the subsequent evaporation of the top layer. The thickness of the layers could be monitored during deposition by a quartz crystal oscillator and (in the case of metal layers) by measuring the drop in resistance between the pre-laid contacts (evaporation was stopped once the resistance had fallen below 40 Ω). For some samples, the thicknesses of both the copper and silicon monoxide films were measured after operation of the device using a stylus profilometer (Sloan Dektak II). However, it is difficult to accurately determine thicknesses below 500 Å using this method. It is estimated that the thickness of the bottom metal layer was 1000–1500 Å thick while the insulator thickness varied between 300–500 Å and the top metal layer was 200–300 Å thick.

The I–V characteristics of the devices were obtained via a computer-controlled four-wire measurement using a Thurlby PL320 dual d.c. voltage supply, a Thurlby 1905a Intelligent Digital Multimeter to measure the current, a Blackstar 4503 Intelligent Multimeter to measure the voltage and a Keighley 485 Autoranging Picoammeter to measure the emission

current. The emitted electrons were detected and their energy analysed with a retarding-field analyser.

3. Results and discussion

3.1. Electroforming in UHV

Figure 1 shows the I–V characteristics obtained from a Cu–SiO_x–Cu device (with an insulator $\simeq$500 Å thick) manufactured *in situ* at room temperature in the baked UHV system described above (at a pressure of 7×10^{-10} mbar). The presence of a VCNR region and of electron emission clearly demonstrates that electroforming has occurred despite the pressures being much lower than those usually used to manufacture such devices. This is the first report of the electroforming of a sandwich MIM device in UHV. Indeed, no difference in forming ability was found between devices manufactured in the system when baked or and those manufactured when unbaked. Even in the latter case the ambient pressure (5×10^{-8} mbar) is much lower than that usually used to form devices.

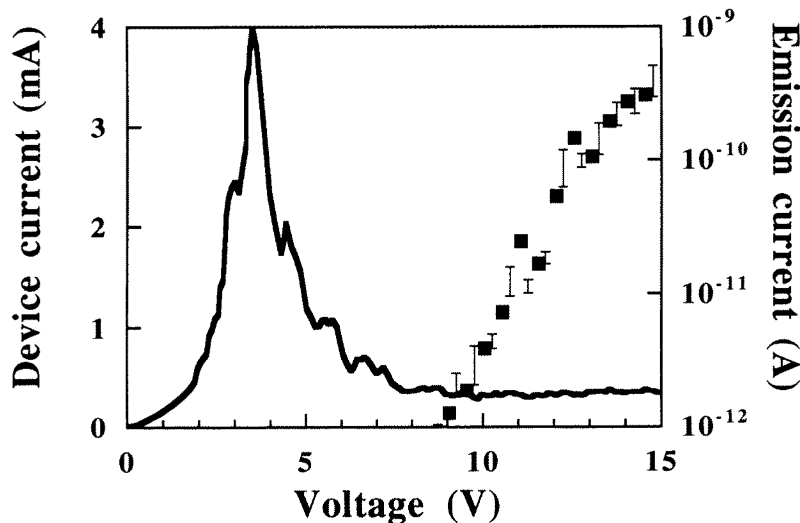

Figure 1. Typical device and emission current characteristics of a Cu–SiO_x–Cu device electroformed *in situ* after manufacture in UHV.

Figure 2 shows the residual gas analyser (RGA) spectrum of the system at a pressure of 1.9×10^{-9} mbar demonstrating that the partial pressure of organic adsorbates is less than 10^{-10} mbar. At this pressure it would take several hours for even one complete monolayer of organic adsorbates to accumulate on the device. In sandwich structures, such as those used in this study, these adsorbates then have to be absorbed into the device through the top metal layer in order to reach the insulating layer. However, devices have been formed when only half an hour has elapsed from the start of the manufacturing process to the application of the forming voltage. This result in itself does not disprove the carbonaceous model since it is possible that some organic material is incorporated into the device during manufacture.

In section 1 we saw that the presence of a continuous supply of organic adsorbates is required by the carbonaceous model not just for filament creation but also for filament

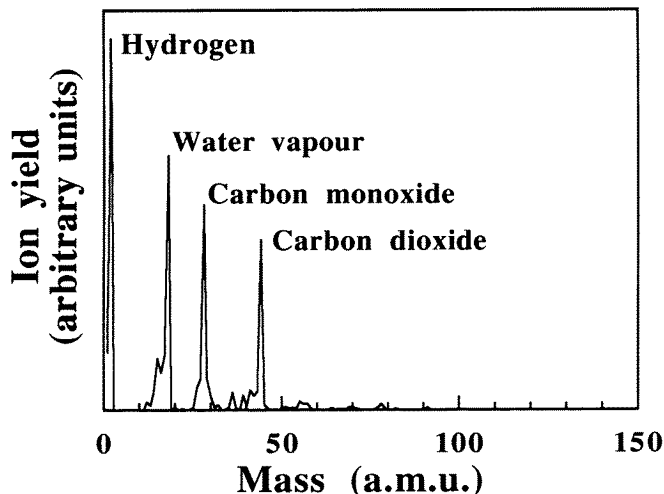

Figure 2. The residual gas analyser spectrum of the vacuum in the baked UHV chamber used to manufacture the Cu–SiO_x–Cu devices.

regeneration. This is an easier criterion to monitor than the total accumulation of organic material in the device and has been proposed as the best test of the presence of carbonaceous filaments by the Darmstadt group [3, 26]. VCNR has been consistently observed, in the absence of organic adsorbates, in these devices when ramping the voltage either up or down (at a ramp rate in the range 0.01–1 V s^{-1}). Thus, filament regeneration readily occurs. This result confirms that the filaments are not carbonaceous.

The devices created by the UHV evaporator were found to have a high conductivity prior to forming (in some cases this even made the detection of the forming threshold voltage difficult). This indicates that there is a high defect density in the insulating layer which might encourage the incorporation of filamentary material. The density, stoichiometry and refractive index of evaporated silicon monoxide films are known to be strong functions of the ambient conditions during deposition—e.g., source temperature and the partial pressures of oxygen and water vapour [27, 28]. In fact, silicon monoxide is a getter of water vapour [29] and the partial pressure of water was observed to fall rapidly once SiO_x evaporation started and rise rapidly once evaporation ceased. This leads to high porosity which might explain why the samples used in this study do form in UHV, while other authors are unable to form devices under similar circumstances. Hence, it seems likely that previous reports of devices that failed to electroform in UHV [8, 9] were due to the manufacturing conditions of those devices which gave rise to less porous insulating layers thus requiring the pores to be created subsequently (B-type forming). Various adsorbates (including organic ones) might help to increase the porosity of an insulating layer, and thus the lack of such adsorbates would inhibit B-type forming in such devices where the pores are not already present. However, for the devices made in this study, B-type forming has already occurred due to the manufacturing conditions.

Some of the samples produced were found to be initially shorted due to metal, evaporated for the top electrode, finding a path through the insulator (probably a pinhole) to reach the bottom metal layer. This is another indication of the high porosity of the insulating layer

produced by the method of manufacture used in this study. As has been previously observed in other such shorted devices [30–32], if a sufficiently large voltage is applied, the short breaks and the device then forms normally. In fact, forming in such cases occurs exceedingly quickly probably due to the high porosity of the insulator and the large amount of metal already incorporated.

In addition, it was found that electroforming was possible if a device was cooled to 125 K prior to applying a voltage. The current flowing through the device grew for a number of voltage ramps until steady I–V characteristics were obtained. VCNR was observed to occur repeatably after this, when ramping the voltage either up or down. However, after the device was left overnight, normal behaviour returned, i.e. VCNR was only seen in the first voltage ramp after cooling since filament regeneration was inhibited. This property has only been reported once previously [33] (and those devices subsequently behaved in an anomalous fashion) although a number of authors have found that steady I–V characteristics cannot easily be obtained in unformed devices under such conditions [34, 35]. The effect is again probably caused by the high porosity of the insulating layer as a result of manufacture which would help forming to occur in conditions in which it was previously thought to be impossible.

Hence, electroforming has been observed in conditions in which the partial pressure of organic adsorbates is negligible but highly porous insulating layers have been created due to the manufacturing conditions. This implies that, although organic adsorbates may aid in the creation of pores in the insulator (B-type forming) if these are not present as a result of manufacture, the filaments themselves are not carbonaceous. In the devices used in these experiments, the manufacturing conditions are such that forming can occur readily in UHV or even at cryogenic temperatures.

Forming and filament regeneration in UHV is direct evidence that carbonaceous adsorbates are not necessary for these processes. However, as mentioned above, the carbonaceous model has also provided a model which adequately explains room temperature filament regeneration and an explanation of the increase in the device conductivity observed when various adsorbates are dosed into the system. In the following section we provide alternative explanations for these effects.

3.2. Room temperature regeneration: the dynamic equilibrium model

Figure 3 shows the regeneration (I–t) characteristics obtained by recording the increase in current with time that occurred after the voltage across a Cu–SiO_x–Cu device, manufactured and formed in the UHV system described above, had been lowered, at room temperature, from 10 V to various regeneration voltages. As can be seen, the I–t characteristics generally show a large, approximately linear rise in current with time in the first few minutes. The value of the regeneration voltage does not have a large effect on this initial regeneration rate after which the rate of current growth slows down. However, saturation is seen to occur only at low voltages (<4 V) whilst at higher voltages there tends to be a small, noisy rise in current with time. The higher the voltage, the more noisy the current appears to be.

The carbonaceous adsorbate model [6, 11] fits these results well if it is assumed that there is only one 'hole' per filament, and thus that the current is proportional to $1 - \exp\{-t/\tau_f\}$. However, as mentioned above, this model has been shown to be not appropriate at low sample temperatures [12] and, since it assumes that a continuous supply of organic vapour is available, is not applicable to the devices used in this study at least. Hence, in this section, we propose a new room temperature regeneration model.

In fact, just modelling regeneration may not be enough. The characteristics do not

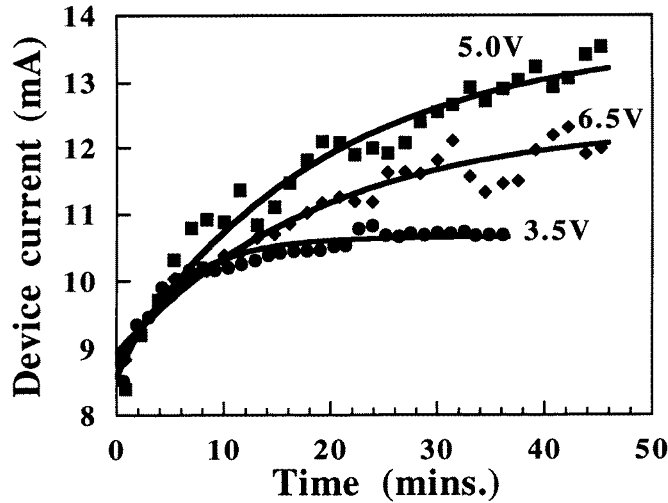

Figure 3. Device current regeneration (after operation at 10 V) at room temperature in an electroformed Cu–SiO_x–Cu device at various regeneration voltages: 3.5 V (circles), 5.0 V (squares) and 6.5 V (diamonds). The solid lines show the best fits obtained using the dynamic equilibrium model.

increase smoothly, and are noisy once saturation is reached, and indeed at some points the current decreases. This suggests that rupture of filaments must also be occurring. Indeed, since the VCNR region in the I–V characteristics is due to filament rupture if the voltage is increasing, and filament regeneration if the voltage is decreasing, it is reasonable to assume that both processes occur at any voltage above a threshold voltage ($\simeq$2 V). Thus, a steady current will only be obtained when the rates of filament rupture and regeneration are the same and thus a dynamic equilibrium is established. If we also assume that the rate at which the regeneration occurs is proportional to the number of broken filaments and that the rate at which the filament rupture occurs is proportional to the number of intact filaments, then we see that

$$\text{Rate of regeneration} = r_+(N - n(t)) \tag{2}$$

$$\text{Rate of rupture} = r_- n(t) \tag{3}$$

where N is the total number of filaments (assumed to remain constant), $n(t)$ is the number of intact filaments at time t and r_+ and r_- are both constants. Hence

$$\mathrm{d}n(t)/\mathrm{d}t = r_+(N - n(t)) - r_- n(t)$$

which, by integration, gives

$$n(t) - n_0 = (r_+ N/(r_+ + r_-) - n_0)[1 - \exp\{-(r_+ + r_-)t\}] \tag{4}$$

where n_0 is the number of intact filaments at $t = 0$ and $r_+ N/(r_+ + r_-)$ is the number intact as $t \to \infty$. Thus, we have obtained the same relationship as equation (1) predicts for one 'hole' per filament, and, if we assume that the current is simply proportional to the number of intact filaments, this agrees well with the observed I–t relationship as we have seen.

Eventually a situation is reached where all the filaments that are capable of remaining intact at the regeneration voltage without overheating are doing so, and the rate at which

filaments rupture and regenerate are the same. Hence, the noise level of the current can be expected to be dependent on which of these two effects (conduction due to stable versus conduction due to unstable filaments) is dominant. Since fewer filaments are stable at higher voltages it is to be expected, qualitatively, that there will be more noise at higher voltages. Similarly more noise is expected as the current approaches saturation at a set voltage since more filaments are rupturing. Both of these effects are seen experimentally (see figure 3).

Current saturation does not seem to occur, except at low applied voltages; a slow rise in current with voltage is seen instead. This could be caused by a slow rise in the operating temperature of the device due to steadily increasing power dissipation. This would raise the filament regeneration rate and thus, as more filaments would be intact at any given time, increase the filament rupture rate. Such an internal temperature rise is also postulated to fully explain the low-temperature regeneration characteristics [12].

This dynamic equilibrium model is entirely consistent with the 'concerted regeneration' model developed to explain low-temperature current regeneration [12]. The main difference is that, at low temperatures, regeneration is inhibited and therefore only occurs in those parts of the insulator in which there is a high local temperature due to the power dissipated by nearby, intact, 'active' filaments. Once a high proportion of filaments are intact the low-temperature regeneration characteristics are akin to those observed at room temperature since the effective temperatures of the insulators are similar in the two cases.

The increase in device conductivity observed when various organic adsorbates are dosed onto the devices has, as outlined above, been used as evidence that carbonaceous adsorbates are created [9, 10]. However, in a series of experiments in which Cu–SiO_x–Cu devices have been exposed to chloroform [36], it has been shown that the conductivity increase observed can be explained entirely in terms of a shift in the position of dynamic equilibrium caused by an increase in the regeneration rate. Indeed, if the dosing ceases, the device current returns to a value very close to the value prior to the start of the dosing. Thus, there is no need to postulate an increase in the number of conducting filaments and, hence, that new carbonaceous filaments are created.

3.3. The effect of filament regeneration on the emission current

Figure 4 shows the device current growth with time at room temperature that occurs in another Cu–SiO_x–Cu device at 5 V (after the voltage has been reduced from 15 V). This shows that the emission current is highest at the onset of regeneration (after the voltage switch) and decreases as saturation is approached. To the best of the authors' knowledge this is the first time that the time dependence of the emission current has been reported during regeneration. Previous authors have assumed that low-voltage, room temperature emission is caused by hot electrons created by filament rupture [37] (mainly because a local maximum in the emission current–voltage characteristics is observed in the VCNR region where lots of rupture occurs [38]). However, the dynamic equilibrium model predicts that, at saturation, the rates of filament rupture and regeneration are equal. The filament regeneration rate is likely to be high immediately after the voltage reduction (when most filaments are ruptured) and decrease as saturation approaches. This is the trend shown by the emission current so this study tentatively suggests that low-voltage, room temperature emission is linked to filament regeneration rather than rupture. This topic will be explored further in a separate publication [36].

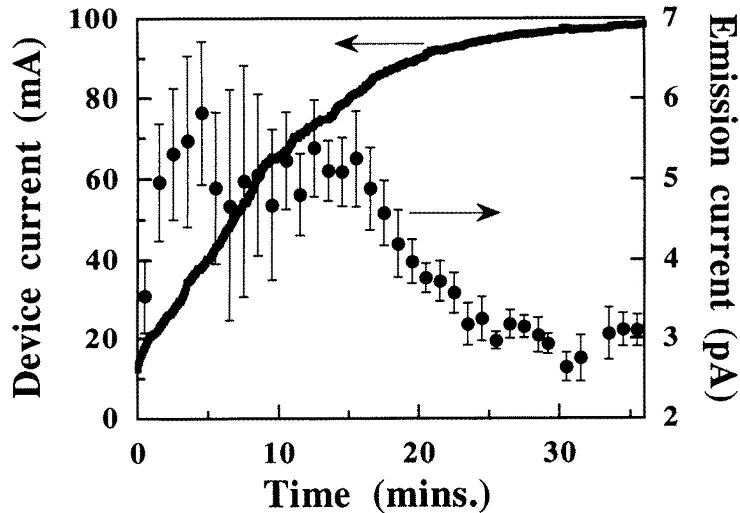

Figure 4. Device and emission current regeneration at room temperature in an electroformed Cu–SiO_x–Cu device at 5 V (after operation at 15 V).

4. Conclusions

We have shown evidence that the filaments in electroformed MIM devices, in the samples produced in this study, are not carbonaceous and that they are metallic instead. Forming has been observed in UHV illustrating that it can certainly occur in the absence of organic adsorbates. It has been postulated that the previously reported inhibition of forming due to the lack of such adsorbates [8, 9] is due to the inhibition of the creation of pores in the insulator rather than the lack of material required to create the filaments themselves. However, since nominally identical devices manufactured under different conditions can behave very differently, it is possible that carbonaceous filaments could exist in other samples and even that some samples might contain both carbonaceous and metallic filaments. In particular, it seems likely that, if they exist, carbonaceous filaments would occur preferentially in planar devices and metallic filaments in sandwich devices.

In addition, a new model of room temperature regeneration has been proposed which explains the observed current–time characteristics simply in terms of a dynamic equilibrium between the rates of filament rupture and regeneration, without the need to assume that the filaments are carbonaceous. The time dependence of the emission current during filament regeneration has been reported for the first time indicating that a link exists between low-voltage, room temperature electron emission and filament regeneration.

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