Laser Ablation of Cryogenic Dielectrics

L.H. Truong and P.L. Lewin

Tony Davies Laboratory, University of Southampton, UK

I. INTRODUCTION

Cryogenic dielectrics are essential for the concept of High Temperature Superconducting (HTS) applications. In a HTS device, solid dielectrics are immersed in liquid nitrogen (LN2). Liquid nitrogen is acting as a coolant as well as a part of the insulation system. Although the intrinsic breakdown strength of LN2 is high, comparable to mineral oil [1], the liquid is very susceptible to partial discharges (PD) which results in density change streamers. Since LN2 is often used close to its boiling temperature, phase change can occur even by a small increase of the liquid temperature. The density change channels if fully developed can lead to total electrical breakdown of the liquid. Therefore, LN2 failure has often been observed at much lower voltage than that of its intrinsic breakdown strength [1].

Due to its simple chemical composition, liquid nitrogen is not degraded after PD or even total breakdown. The only effect is the generation of clouds of gaseous bubbles which will gradually disappear providing that sufficient cooling is available. The damage caused by PD streamers on the solid part of in a composite insulation system, on the other hand, is irreversible. The degradation of solid dielectrics can be accumulated over time which can result in premature failure of the materials. Therefore, besides dielectric properties, erosion performance is also important in improving lifetime and stability of the insulation system.

This paper investigates the aging phenomenon of solid insulation under cryogenics conditions caused by PD streamers in LN2. Previous work has shown that laser ablation is an effective method for studying surface erosion of insulting materials since it can be used to simulate the thermal degradation associated with electrical discharges [2]. The principle of this method is to apply a known energy to a localised region of the tested sample using a high power laser source. Two distinct processes have been suggested to be involved with ablation of materials namely photochemical and thermal. UV radiation with short wavelength, e.g. 193 nm, can produce chemical reaction which leads to bond breaking of the materials. Photons from a visible or infrared laser do not have enough energy to cause photochemical effects. The energy in this case is more likely to be absorbed by the solid material and resulting in localised heating [3]. Since this work focus on thermal degradation, an infrared laser was used.

The discharge experiments utilise a point-to-plane configuration with an insulation barrier placing over the planar electrode. This PD source was subjected to a 50 Hz Ac voltage over a period of 30 minutes. After the experiments, degradation was found on the surface of the tested materials indicating that PD streamers in LN2 can cause significant damage on the solid barrier in between the LN2 and a metal electrode. The damage caused by discharges is compared with laser ablation results of the same materials in order to study the mechanism governing the erosion phenomenon under cryogenic conditions.

II. EXPERIMENT

The schematic diagram of the experimental technique is shown in Fig. 1. The PD source consists of a sharp tungsten needle of which tip radius was measured at 2.5±0.5 μm and a flat planar electrode. The two solid dielectrics investigated were glass-fibre reinforced plastic (GRP) and polytetrafluoroethylene (PTFE). The thickness of these barriers was 2.5 mm and 3 mm respectively. The tungsten needle was placed 4 mm away from the planar electrode. The whole configuration was submerged in LN2 as 77.5±0.5 K and at atmospheric pressure. Experiments were taken inside a cryostat which allowed pressure and temperature controlled as well as PD-free conditions up to 50 kV [4]. The vessel was carefully vacuumed before each test to eliminate contaminations such as oxygen and water from the liquid. PD was measured by an Omicron Mtronix MPD600 PD measurement system which is compliant with the IEC 60270 standard. The bandwidth of the device was set at 10-1000 kHz. A high speed camera in conjunction with a high density light source was used to observe the discharge location during the testing period.

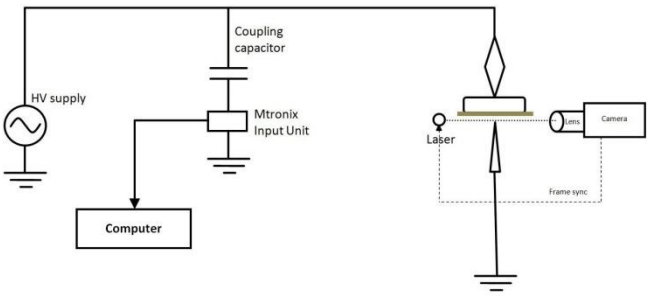


Figure 1. Schematic diagram of experimental arrangement

PTFE and GRP sheets were cut into 30x40 mm^2 samples for the laser ablation study. The prepared samples were radiated by a carbon dioxide laser with a wavelength of

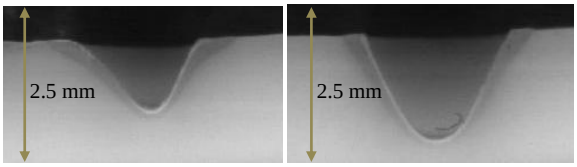
10.5–10.6 µm. The laser was located inside a fully enclosed aluminium box with an interlock system that only allows the laser to be radiated when the cabinet is closed. Laser power output and duration is controlled from the outside of the interlocked box. The samples were located horizontally and at 200 mm away from the laser aperture. The diameter of the laser spot was 3mm. The samples were tested at various laser powers (2 W to 28 W) and durations. The energy presented was calculated by multiplying the laser output and the exposure time. The surface damage was quantified by the pit-depth as well as weight loss. Each sample was weighed on a digital scale of which accuracy was 0.1 mg before and after the laser ablation test. A simple subtraction then will give the weight loss by laser radiation. After being weighted, the radiated samples were bisected in order to measure the pit-depth.

III. RESULTS

3.1 Laser ablation results

In the case of PTFE samples, ablation damage appears in form of holes at the laser contact point as shown in Fig. 2. The higher power and energy of the applied laser, the more materials was ablated indicated by the increase of the hole depth. In both cases presented in Fig.2, the tested samples show a V-shape profile which has been previously reported for unfilled materials in contrast to the spherical shape observed in laser ablation experiments with filled polymeric composites [5]. In all PTFE samples, laser ablation damage was from vaporisation of materials, almost none carbonisation was observed. Fig. 3 shows the variations of the erosion depth as a function of the laser energy. These samples were exposed to laser radiation for a fixed period of 10 s while the laser output increased from 3 W to 27 W. The hole-depth increases almost linearly with the laser energy. Compete puncture of the 3-mm PTFE samples occurred when the energy exceeded 35 W.

While the damage caused by laser ablation on PTFE was simply the removal of material, the phenomenon for the GRP samples was more complicated. This is because of the glass/epoxy structure of the composite. The epoxy resin can be degraded at much lower temperature, a few hundred degrees; meanwhile the degradation of glass fibre only occurs well above a thousand degrees [6]. Therefore, at lower laser energy (below 100 J), only the epoxy layer was removed as shown in Fig. 4 (a). As the energy increases above a few hundred Joules, the melting of the glass fibre was observed (Fig. 4 (b)). Different to the PTFE materials, carbonisation was the dominant phenomenon due to the pyrolysis of the organic epoxy. Especially under high energy radiations, carbon products were found up to tenths of millimetres away from the laser contact point. As shown in Fig. 4 (c), degradation happened far away from the 3-mm circle of the radiated area. On the other hand, using the same power and energy, vaporisation of PTFE was localised around the exposure region. Immediately after being radiated PTFE was found softened or in a liquid form in a few-millimetre circle around the contact area. If the sample then was left at room temperature for a few minutes, liquid PTFE would return to the original solid form. The foregoing observation is the result of thermal conduction which leads to an increase in temperature of the surrounding environment. Although this rise in temperature is high enough to liquidity PTFE, it is not sufficient to cause vaporisation of the polymer. Meanwhile, in the case of GRP, the thermal conduction would cause thermal decomposition of the epoxy resin which results in the carbon products observed far away from the laser contact point.



(a) (b)

Figure 2. Cross sections of erosion pits in PTFE formed by laser ablation at (a) 9 W, (b) 24 W

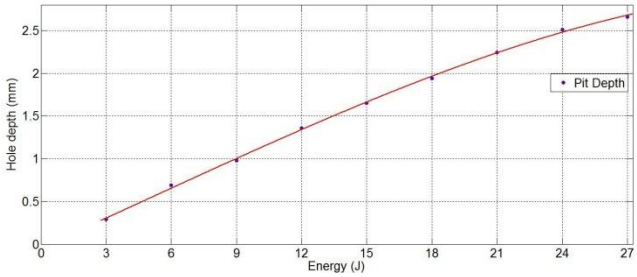
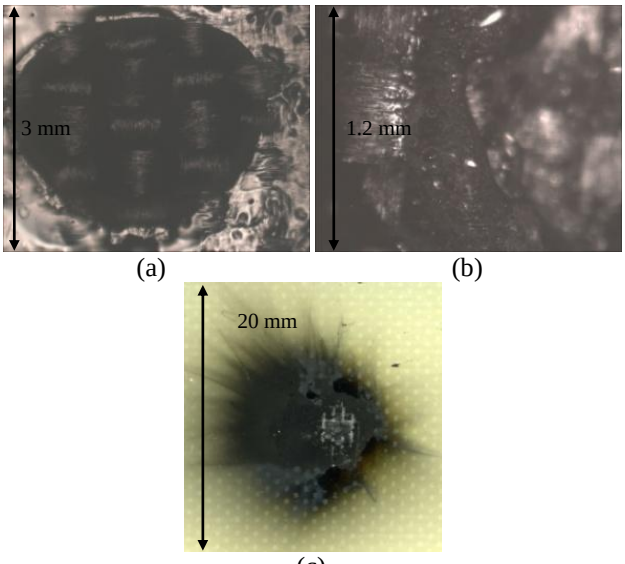


Figure 3. Hole depth as a function of laser energy



(a) (b)

(c)

Figure 4. Laser Ablation of GRP samples at (a) 15 W and 15 J, (b) (c) 28 W and 420 J

The mass loss data for ablated GRP and PTFE samples are shown in Fig. 5. The power laser was set at 50% and 95% corresponding to 15W and 28 W respectively. Below 800 J, for PTFE samples, the mass lost data is very similar for both power levels. As the energy increases, the results during 15-W tests saturated at just below 55 mg while the mass loss by 28-W laser continued to increase. For a given laser power and regardless of energy, PTFE samples experienced more weight reduction compared with the other material. The observation is due to different ablating mechanisms for the two materials. Under the same laser radiation, PTFE samples experienced a lot of material removal due to vaporisation while glass fibre was hardly melted. The weight loss from the epoxy part of the GRP is very limited due to the fact that majority of the carbon products were still remained on the samples.

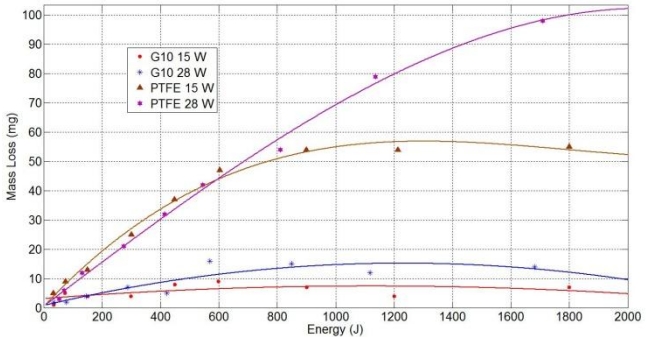


Figure 5. Laser ablation data for G10 and PTFE samples at 15 W and 28 W.

3.2 Discharge in LN2

Pre-breakdown streamers were observed above 15 kV. The phase-change channels initiated from the needle tip and propagated towards the planar electrode under the influence of the applied voltage. However, close to PD inception voltage, the streamers only propagated less than 1 mm and did not reach the solid barrier electrode (Fig. 6 (a)). As the voltage increased, the phase change channels lengthened and eventually bridged the liquid spacing gap above 18 kV (Fig. 6 (b)). Once reaching the solid insulation, the channels continue propagating along the surface of the barrier. The attachment of streamer channels does not necessarily lead to degradation of the solid barriers. In fact, up to 30 kV and for 30 minutes of applied voltage, no significant damage was observed on the surface of the insulation barriers. Surface erosion on both materials were seen when the applied voltage was increased to 35 kV.

GRP and PTFE sheets were subjected to 35 kV Ac voltage for 30 minutes. During this period, pre-breakdown streamers were observed repeatedly. The liquid was vaporised vigorously resulting in clouds of bubbles sitting at the solid barriers. Plasma channels were observed together with intensive light emission as shown in Fig. 6 (c) and (d).

Discharge data for GRP and PTFE samples was displayed in Fig. 7. The presented graphs were measured during the first 5 minutes of applied voltages. Since the planar electrode was connected to the HV supply while the needle was earthed, the polarity of the tip is opposite to that presented by the voltage waveform in Fig. 7. For all samples, discharges were located primarily in the first and third quadrant of the applied voltage due to space charge accumulation on the surface of the barriers. Different discharge patterns were observed for the two studied dielectrics. Maximum magnitude was much higher in the case of GRP samples compared with PTFE samples, 200 nC and 71 nC respectively.

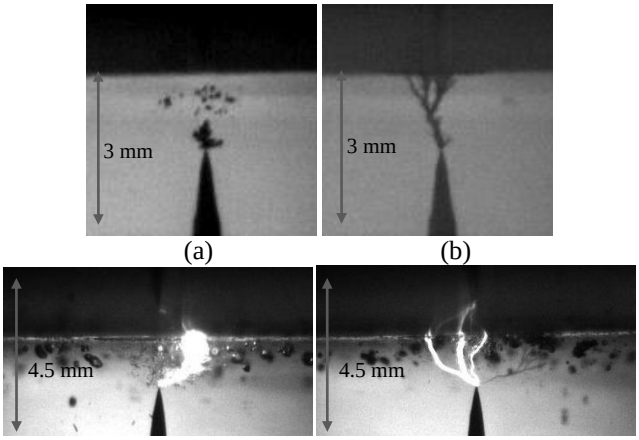


Figure 6. Density change streamers in LN2 at (a) 15 kV (b) 20 kV, (c) (d) 35 kV. GRP sample, LN2 at 77.5 K, atmospheric pressure.

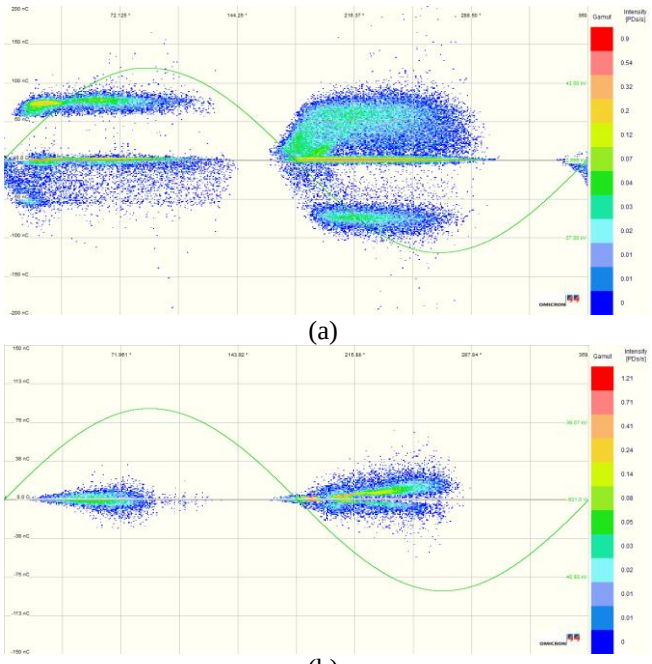


Figure 7. Accumulated PD pulses over the first 5 minute of applied voltages: (a) GRP, (b) PTFE.

Typical degradation of PTFE and GRP due to PD in LN2 is shown in Fig. 8. For GRP samples, erosion appears in form of a circular hole together with further cracking on the surface of the material. Different to laser ablation results, not only epoxy layer but also glass fibre was removed. Recorded shadowgraphs reveal that the eroded hole was the attachment area of streamer channels (Fig. 6 (c) and (d)). Therefore, it can be hypothesised that the major damage area, the circular pit, is caused by initial streamer attachments while the cracking is the consequence of streamer propagation on the surface of the insulation. Microscopic images (Fig. 7 (c)) show that the glass fibre was broken rather than melted as in the case of laser ablation. In fact, free particles were observed only during the aging of GRP samples as shown in Fig. 7 (d). The particles had to be the products of discharges into GRP sheets because of two reasons. Firstly, the cryostat was vacuumed which means impurities must be produced during the experiments. Secondly, the particles were not observed during PTFE tests, and GRP samples at low-voltage conditions under which the composite sheets were not damaged. Thus, the mechanisms for the eroded composite material can be suggested. From the laser ablation results, the epoxy layer is very susceptible to thermal decomposition. The attachment of hot plasma channels could damage the epoxy resin leaving the fibre weaves exposed to LN2. Under cryogenic conditions, the glass fibre becomes very brittle. Therefore, while the fibre has good resistance to thermal effects, it can easily be degraded mechanically and broken into smaller pieces which are when the free particles are observed. Thermal contraction at cryogenic temperature and the pressure shock waves from streamer propagation [7] are the two possible causes for the breaking of glass fibre weave.

For PTFE samples, typical erosion profile from PD in LN2 experiments is shown in Fig. 8 (b). PTFE appears to be more resistant to degradation from streamers than GRP. The damage remains only on the surface and does not penetrate into the bulk of the material. Laser ablation results show significant vaporisation and mass loss while the damage from experiments in LN2/PTFE only appears on the surface and almost no weight loss was seen. Such difference is due to the fact that under cryogenic conditions, thermal degradation becomes less effective. Any localised heat dissipation can quickly dissipate by the surrounding liquid nitrogen. Chemical reaction is very unlikely to happen due to the lack of oxygen. Therefore, the eroded surface of the PTFE must be a consequence of interactions between high energy charges within the gaseous channels and the solid molecules.

Figure 9 shows the total charge transferred per a 20-ms cycle. The number was averaged from data captured from 1000 cycles at a certain time during the 30-minute of applied voltage. The plot shows the change in the amount of apparent charge over time. For PTFE samples, the total charge varies between 700 and 800 nC without any defined trend. Experiments with GRP/LN2, on the other hand, show an increase in apparent charge of PD pulses over time. The difference between the two materials is certainly related to their erosion behaviour. The glass fibre composite degraded severely overtime as the pit depth increases. The reduction of the thickness of the GRP barrier would result in higher magnitude PD pulses. Therefore, the total charge transfer rises during the testing period. On the other hand, PTFE samples did not experience such erosion. Consequently, the total charge remains fairly constant over the stressing period.

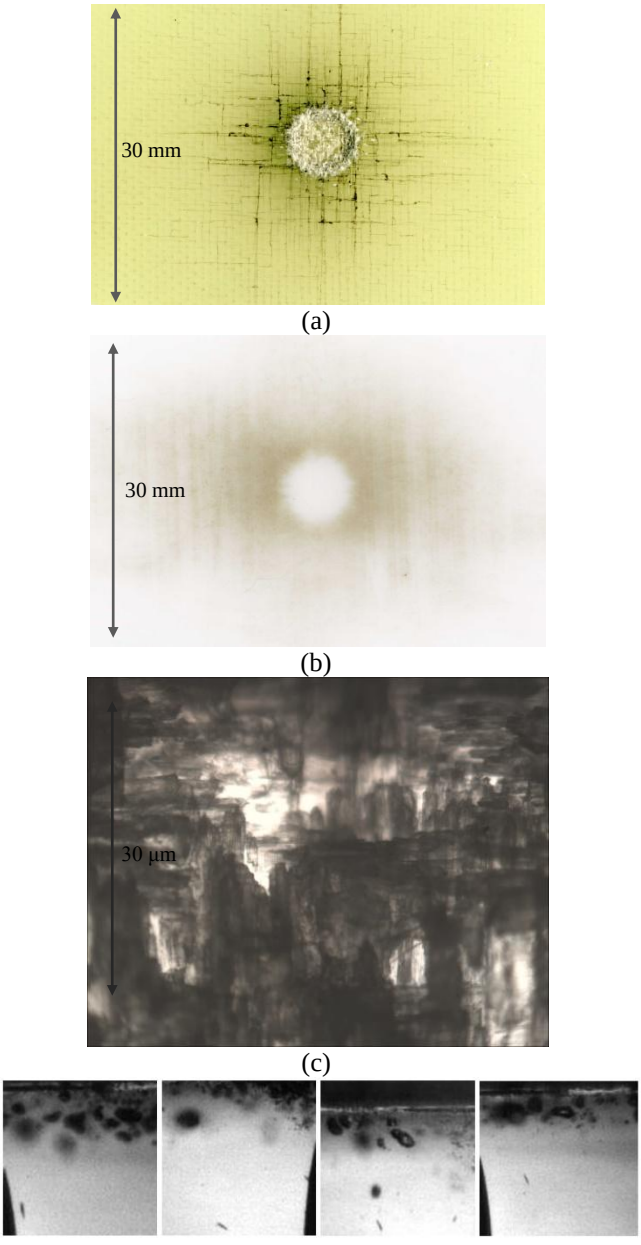


Figure 8. Surface Erosion (a) GRP, (b) PTFE, (c) 1000x magnification of the eroded hole in (a), (d) particles generated during GRP experiments

3.3 Effects of sample thickness

PTFE sheets with thickness of 2 mm were used instead of the 3-mm samples. Other experimental conditions remained unchanged. Similar to previous results, at 30 kV, no visible damage was seen. The samples then were tested under 35 kV. Breakdown happened randomly after 1-5 minutes of applied voltage. Fig. 10 (a) shown the PD pattern captured during a period of 30 second prior to breakdown. The discharge pattern and magnitude is very similar to the result from 3-mm samples as shown in Fig. 7 (b). This implies that the thickness of samples does not affect the discharge signature. The insulation barrier material, on the other hand, can greatly influence the discharge pattern as shown in Fig.7.

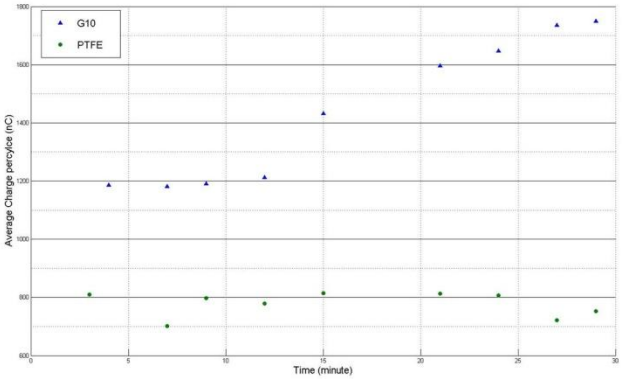
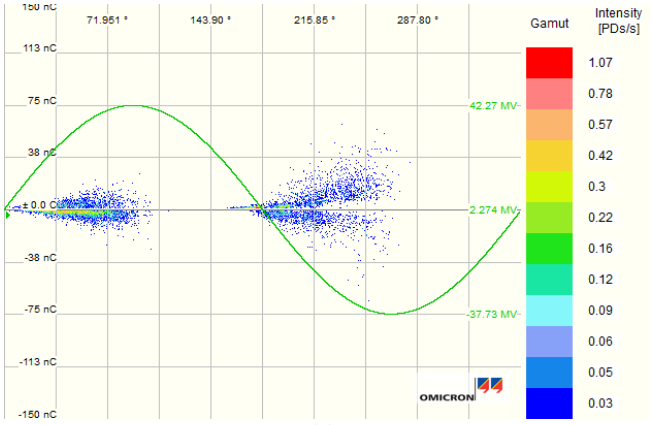
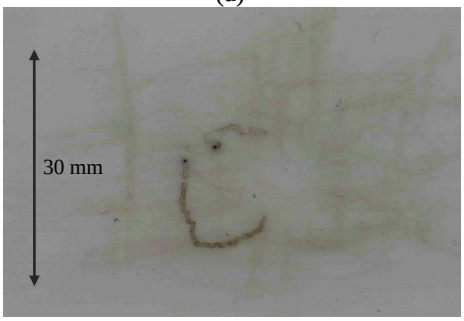


Figure 9. Charge transferred per cycle over 30 minutes of applied voltage.

Erosion on the 2-mm sheets shows similar characteristics to the thicker sample. Damage appears in a large area but only on the surface of the insulation. However, eroded area is less clear than that in Fig. 8 (b) because of much shorter stressing duration. Two breakdown holes which completely punched through the sample can be seen. The paths leading to the breakdown holes suggest that the breakdown positions are not the attachment points of the streamer channels. Upon contact, streamer channels continue to propagate until weaker insulation paths are found. Because the density change channels are considered to be conductive, together with high amount of charge at the streamer head, the electric field at the streamer tips is very high enough to breakdown the sample. Breakdown did not happen for 3-mm samples since the thicker materials mean the higher the breakdown voltage is required. Dielectric failure of the insulation is suggested to be due to a particular single event rather than an accumulation of repetitive PD. The duration before breakdown (several minutes) is the time it takes for the streamer channels to find the weakest link of the polymer rather than to accumulatively age the material.



(a)



(b)

Figure 10. Experiments with 2-mm PTFE sheet, breakdown happened in less than 3 minutes. (a) PD data, (b) image of surface erosion after test.

IV. CONCLUSION

This paper investigates the erosion phenomenon on solid dielectrics under cryogenic conditions. Previous works [2] have shown correlative results between laser ablation and discharge tests at room temperature such as incline tracking tests. The results presented here suggest that at LN2 temperature, laser ablation data cannot be used for ranking materials for cryogenic applications. This is because thermal decomposition is much less effective at low temperature and other degradation mechanisms must be considered.

Partial discharge streamers in LN2 can cause significant damage to insulation barrier on its way. Fibre/epoxy composite has been found to be less resistant to aging than PTFE although laser ablation results suggest otherwise. Once the epoxy layer has been degraded, the glass fibre which is very brittle at cryogenic temperature is very susceptible to mechanical damage due to thermal contraction and pressure shockwave from streamer propagation. The result is a hole is formed at the streamer attachment points. The damage on the PTFE samples on the other hand only appears on the surface. This is believed to be the result of electron interactions with the solid molecules rather than thermal or chemical effects. Further study on different materials and experimental conditions is

required to aid understanding of the degradation mechanism of solid dielectric under cryogenic conditions.

References

[1] *G. J. Gerhold, "Cryogenic Liquids-A Prospective Insulation Basis for Future Power Equipment", IEEE Trans. Dielectr. Electr. Insul., vol. 9, no. 1, pp 68-75 (2002).*

[2] *D. Srinivasan, G. G. Karady, "Development of the dry-band arc on all dielectric Self-Supporting cables during laboratory tests", IEEE Trans. Pow. Del., vol. 19, pp. 1746-1750 (2004).*

[3] *B. J. Garrison, R. Srinivansan, "Laser ablation of organic polymers – microscopic models for photochemical and thermal processes", J. Appl. Phys., vol. 57, pp. 2909-2914 (1985).*

[4] *D. J. Swaffield, P. L. Lewin, G. Chen and S. G. Swingler, "Variable Pressure and Temperature Liquid Nitrogen Cryostat for Optical Measurements with Applied Electric Fields", J. Meas. Sci. Technol., vol. 15, no. 11, pp. 2325-2332 (2004).*

[5] *I.L. Hosier, M.S. Abd Rahman, E.W. Westenbrink and A.S. Vaughan, "Laser Ablation of Polymeric Composites", Proc. 2010 IEEE International Conference on Solid Dielectrics, Germany, 455-59 (2010).*

[6] *K. C. A. Crane, J. R. Brown, "Laser induced ablation of fibre epoxy composites", J. Phys. D – Appl. Phys., vol. 14, pp. 2341-2349 (1981).*

[7] *A. Denat, "High Field Conduction and Prebreakdown Phenomena in Dielectric Liquids", IEEE Trans. Dielectr. Electr. Insul., vol. 13, no. 3, pp 518-525 (2006).*