

Understanding the performance of corona aged epoxy nano micro composites

Belaguppa Manjunath Ashwin Desai¹, Palash Mishra¹, Nilesh J. Vasa², Ramanujam Sarathi¹ ✉, Takahiro Imai³

¹Department of Electrical Engineering, Indian Institute of Technology Madras, Chennai 600 036, India

²Department of Engineering Design, Indian Institute of Technology Madras, Chennai 600 036, India

³Toshiba Corporation, Power, and Industrial Systems R&D Centre, 1, Toshiba-Cho, Fuchu-Shi, Tokyo 183-8511, Japan

✉ E-mail: rsarathi@iitm.ac.in

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Corona ageing of the epoxy nanocomposites surface exhibits a high influence on contact angle of the material. A reduction in corona inception voltage due to water droplet, upon corona ageing, is less with epoxy composites, which has Wollastonite as filler followed with nano-micro silica filler added epoxy composites. Charge accumulation studies indicate that charge retention time drastically reduces with corona aged epoxy composite specimen. Epoxy composites with Wollastonite as filler have shown higher mean charge lifetime. Adoption of laser induced breakdown spectroscopy (LIBS) technique for characterisation of samples is unique. Measure of threshold fluence and plasma temperatures through LIBS studies enables to classify the ageing condition of the composite material. Plasma temperature and threshold fluence are clear indicators to classify different materials. Plasma temperature is also an indicator of the hardness of the material. Epoxy composite with Wollastonite as filler is not affected by laser abrasion, which is in accordance with its superior performance with corona ageing, proving as discharge resistant material.

1. Introduction: Power equipment, irrespective of its operating voltage, improper insulation to it can lead to its catastrophic failure. Epoxy resin is one of the common insulating material having high performance. They exhibit better dielectric, thermal, mechanical, chemical and electrical properties. The properties of base epoxy are altered by adding fillers, its size, aspect ratio, the shape of the filler, particle/matrix interface adhesion and fabrication technique used are found to play a major role in achieving the desired properties of the material [1–3]. Recently, considerable works were carried out to understand the performance of epoxy resin using nano fillers such as clay, zeolite, alumina, boron nitride, silica, titania, magnesia [4–10]. It is possible to achieve required permittivity of the material by varying the percentage of filler content into the base material, uniform dispersion of nano fillers allows one to achieve a lower value of permittivity and loss tangent, resulting in improved breakdown strength and insulation performance [11]. Composites show increased partial discharge (PD) resistance and the inorganic fillers have improved damage resistance due to PDs [12].

Hashem *et al.* have observed uniform dispersion of titania particle having good interfacial bondage and increased thermal stability with polyimide (PI)/TiO₂ composite compared to virgin material [13]. The theoretical models indicate that characteristic variations at the interface region have high impact on permittivity of the material, they provide methodology to achieve desired dynamic mechanical properties [14, 15]. Thus theoretical models can allow one to acquire desired properties of the material, for any specific application.

Having known the advantages of reinforcing filler in the polymer, in this Letter, an attempt has been made to understand the impact of silica and Wollastonite as fillers in epoxy matrix. Tomášková *et al.* [16] have observed better electrical and thermal properties upon adding nano and micro silica into epoxy. Tanaka and Imai [17] have observed that silica epoxy nanocomposite is more resistant to PD compared to other composites. Iizuka *et al.* [18] have observed increased treeing and PD resistance with nano silica filler in epoxy. Research on Wollastonite composite's electrical behaviour is scanty. Thus in this Letter, three different epoxy composite materials were studied to understand their performance, to use as an insulant in power apparatus. The details of the material are provided in Table 1.

Sarathi *et al.* have studied water droplets on epoxy nanocomposites with montmorillonite (MMT) clay as nano filler, under high electric field and have indicated the formation of water droplet initiated corona radiating ultra high frequency (UHF) signal and damage to insulation due to arcing between water droplet and the electrodes. Also, they have measured the local temperature during arcing through optical emission spectroscopy [19, 20].

One of the main causes for the formation of surface discharge activity on insulating material is the charge accumulation and once the tangential electric field exceeds a certain limit, surface discharge activity prevails. Thus it is essential for one to understand the surface charge accumulation characteristics.

Laser induced breakdown spectroscopy (LIBS) is one of the promising technique for elemental analysis. Meyer *et al.* [21] have adopted laser technique to understand the ageing condition of the insulating material. Laser irradiated damage studies of the epoxy nano/nano micro composites (NMCs) are scanty.

Having known all these aspects, the following studies were carried out to understand the performance of the epoxy composite material. (i) Variation in contact angle on corona ageing of epoxy nanocomposites, (ii) variation in corona inception voltage (CIV) due to water droplet on epoxy nanocomposites aged due to corona discharges, (iii) characteristic variation in surface charge accumulation of material with virgin and corona aged specimen, (iv) analysis of composite material characteristics adopting LIBS.

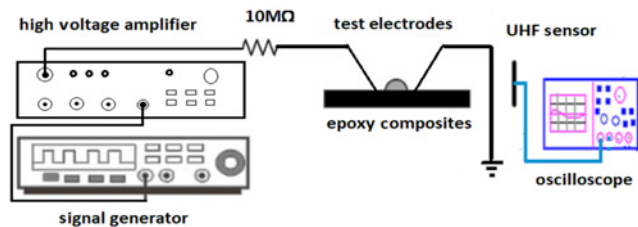
2. Experimental studies

2.1. Experimental setup: The experimental setup used for understanding water droplet initiated discharge is shown in Fig. 1. The experimental setup can be sectionalised into three parts, namely the high-voltage source, the test electrode, and the UHF sensor connected to the high bandwidth digital storage oscilloscope.

2.1.1. High-voltage source: The high DC and AC voltages were produced by using a Trek amplifier (Model 20/20C) in current limit mode with input to it from a signal generator (Tektronix 3051C). The generated AC and DC voltages were measured using resistance and capacitance dividers, respectively. The voltage was applied to the test at the rate of 200 V/s.

Table 1 Sample composition

Sample acronym	Composition, wt%
IN	epoxy resin with Wollastonite micro filler (56 wt%)
MC	epoxy resin with crystalline SiO ₂ micro filler of 67 wt%
NMC	epoxy resin of SiO ₂ 1.6 wt% nano filler and crystalline SiO ₂ (66 wt%) micro filler

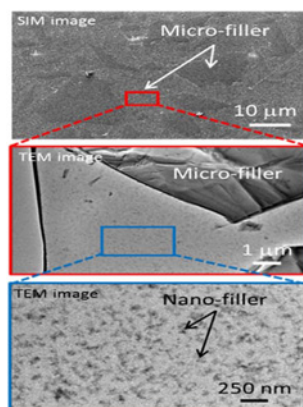
**Fig. 1** Experimental setup

2.1.2. Test electrode: The test electrode arrangement comprises of two stainless steel electrodes with its tip cut at 45° to enhance tangential electric field in the electrode gap (according to IEC 60112 [22]) set on 3 mm thick epoxy composite material. The electrodes are separated by a gap distance of 10 mm. One electrode is connected to the high-voltage source through a resistance of 10 MΩ and the other electrode connected to ground. 10 μL of deionised water was used as droplet.

2.1.3. UHF sensor: In this Letter, non-directional broad band UHF sensor was used by keeping the sensor at a distance of 20 cm from water droplet initiated discharge test setup. The output of the UHF sensor signal was measured using a high bandwidth digital storage oscilloscope (LeCroyWavepro 7300 A, 3.5 GHz bandwidth, operated at 20 GSa/s).

2.2. Sample preparation: The different samples evaluated along with their weight compositions are listed in Table 1. Standard shearing mixing, degassing, curing procedures were used to prepare samples with epoxy as base matrix [23].

Fig. 2 shows structured illumination microscopy (SIM)/transmission electron microscopy (TEM) picture of NMCs. The thin film for TEM studies was prepared by using focused ion beam (model np FB2200, Hitachi Hi-Technologies Corporation, Japan) and uniform distribution of nano fillers in nanocomposites were

**Fig. 2** Typical SIM/TEM pattern of epoxy nanocomposites

visualised using field emission transmission electron microscope (model no HF-2200, Hitachi Hi-Technologies Corporation).

2.3. Corona ageing: The corona was produced by using high-frequency damped sinusoidal AC voltage of 15 kV. Samples were cut into 3 cm × 3 cm and were put beneath the corona electrode with a 2 mm gap. In this Letter, the specimens were exposed to corona treatment for specific time for analysis of corona caused damage studies.

2.4. Surface charge measurement: Needle plane configuration was used to spray charges on the composites by generating corona at 12 kV +DC and –DC. The surface charge accumulation in epoxy composites was measured using electro static voltmeter (ESV) (Trek model 341B). The Letter was conducted with deionised water droplet. The sensor measures the deposited charge present in 5 mm radius when it is placed 3 mm above the water droplet placed on the surface of the specimen. The sample was charged at position 1 and was measured at position 2 as in Fig. 3. The charge Q measured on the surface of epoxy composite material is

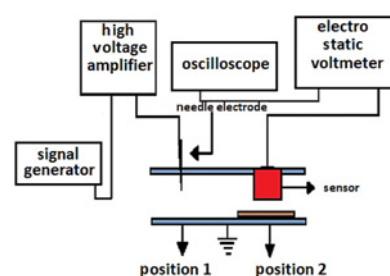
$$Q = \frac{V \cdot \epsilon_0 \cdot \epsilon_r \cdot A}{d} \quad (1)$$

where A is the area of the curved surface of the sensor. d is the distance between sensor surface and specimen. V is the voltage measured by the electrostatic voltmeter. ϵ_0 , ϵ_r are the permittivity of free space and relative permittivity of the specimen, respectively.

2.5. Laser induced breakdown spectroscopy: In LIBS, a pulsed laser is focused on to the sample, which creates optically induced plasma when energy provided by laser pulse is greater than threshold fluence of the material. The excited elements in plasma, while coming to lower energy states, emit electromagnetic radiations. Wavelengths of these radiations are fingerprints of particular elements present in the material [24]. An Nd³⁺ YAG laser was focused on the sample using 25 cm focal length lens, the optical emission is captured using 100 cm focal length lens and guided to a spectrometer (Ocean Optics) through the multimode optical fibre of core diameter 400 μm, 0.22NA as shown in Fig. 4. One second integration period was used to get the spectra.

3. Results and discussion

3.1. Corona inception voltage: Inception voltage is obtained based on first UHF signal captured because of corona inception in the electrode gap. It is observed that the contact angle of the specimens varies like micro composite (MC) > Wollastonite (IN) > NMC (Fig. 5 virgin case). Water droplets sitting on polymeric insulating material, under high electric field, the characteristic change in shape of the water droplet initiating corona discharges under AC/DC voltages are different [25]. Under AC voltage, the droplet undergoes characteristic oscillations which

**Fig. 3** Charge measurement setup

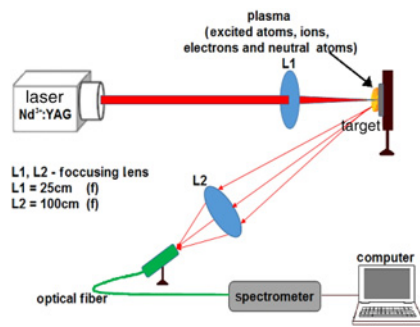


Fig. 4 LIBS experimental setup

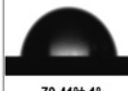
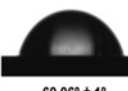
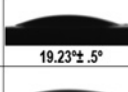
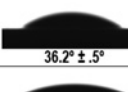
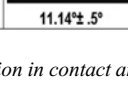
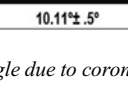
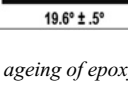
	MC	NMC	IN
virgin	 79.41° ± 1°	 61.43° ± .5°	 69.96° ± 1°
10 mins aged	 19.23° ± .5°	 20.07° ± .5°	 36.2° ± .5°
20 mins aged	 11.14° ± .5°	 10.11° ± .5°	 19.6° ± .5°

Fig. 5 Variation in contact angle due to corona ageing of epoxy composites

depends on the size of the droplet, frequency of the supply voltage and the local electric field at the triple point formed at the interface of water droplet, insulating material and the air medium. Under DC voltages, the water droplet deforms in the direction of electric field but no oscillations are observed [26]. Similar phenomena were observed during experimental studies. As water droplet spreads more in the electrode gap CIV decreases. It is observed that CIV due to water droplet under AC/DC voltages varies like MC>NMC>IN (Fig. 6 virgin case). Fujii *et al.* have indicated that the electrostatic force on the water droplet vary proportionately to square of the electric field [27]. Thus the voltage profiles have high influence on CIV due to water droplet. Also, it is observed that NMC specimen having higher CIV than IN specimen indicates that the contact angle alone is not the influencing factor for corona inception.

3.2. Corona ageing: Philips *et al.* have indicated that the low-energy discharges occur on the surface of the polymeric insulating material, and the hydrophobicity of the material gets reduced [28]. Thereby, the water droplet sitting on the epoxy sample spreads in the electrode gap and at the thin edge of the elongated water droplet, the charge accumulation occurs thereby reducing the CIV. Reynders *et al.* have indicated that the reduction in hydrophobicity occurs with corona aged material [29]. In this Letter, the samples were subjected to corona ageing for 10 and 20 min, then contact angle and CIV of corona aged specimen were determined (Figs. 5 and 6). As already mentioned that under the unaged condition, under all three voltage profiles MC has higher inception voltage than NMC followed by IN. Among the corona aged specimens, it is observed that CIV due to water droplet under AC/DC voltages varies like IN>NMC>MC (Fig. 6 aged case). Same order can be observed in their contact angles (Fig. 5 aged case). Ten minutes aged sample showed a drastic drop in their CIV as well as contact angle. A lower CIV was recorded for the 20 min aged sample than the 10 min aged sample; however, the fall was not high in line with fall in the contact angle (Fig. 5).

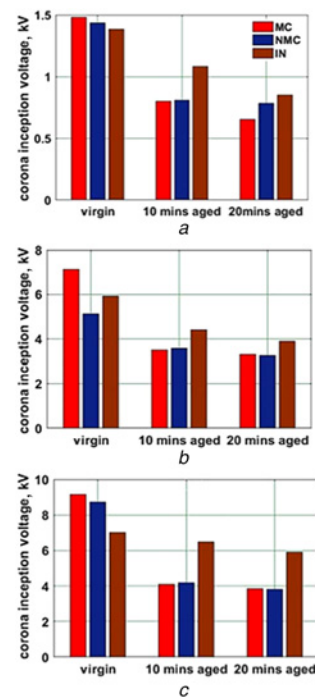


Fig. 6 Variation in inception voltage with corona ageing
a AC
b +DC
c -DC

Interestingly, IN performs better among aged samples, it has highest contact angle and inception voltage than other samples upon ageing, though virgin IN sample performance is not good compared to MC and NMC. This can be attributed to Wollastonite filler in IN which acts as fire resistant and reduces the corona damage. This can be easily observed in level of damages that occurred due to discharges (Fig. 7). IN is least damaged on arcing. All samples were allowed to arc at same voltage for the same interval of time, also it was observed that damage is least with AC voltage.

3.3. Surface charge accumulation: The charge decay process can be quantified as

$$Q(t) = Q_0 e^{-\lambda t} \quad (2)$$

where λ is the decay rate. The mean lifetime ' τ ' can be written as

$$\tau = 1/\lambda \dots \quad (3)$$

Fig. 8 shows the surface charge decay characteristics of the virgin and corona aged samples with the droplet placed at the point of charge injection. It is observed that all virgin samples have a marginal difference in their mean lifetime (Fig. 8a). The amount of initial charge stored by the virgin MC sample is higher than

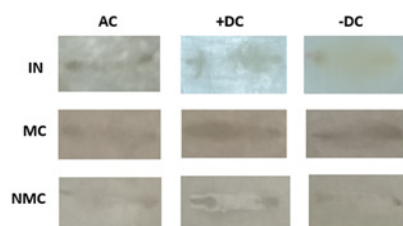


Fig. 7 Damage caused due to arcing on the samples between water droplet and the electrodes

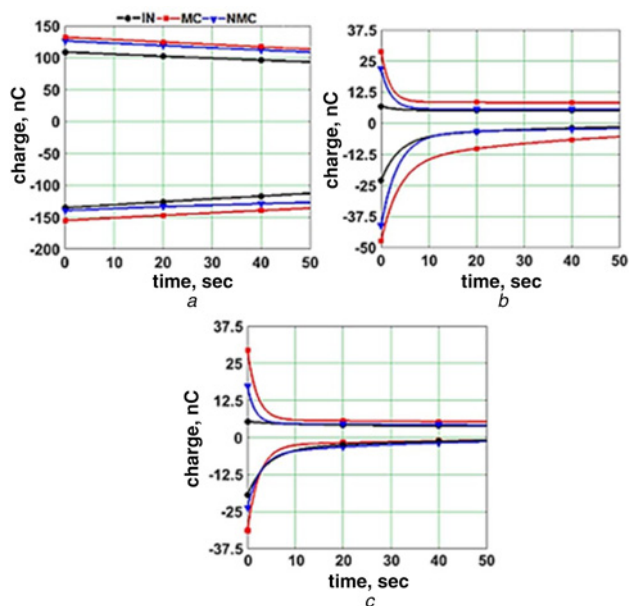


Fig. 8 Surface charge decay characteristics for
a Virgin specimens
b 10 min
c 20 min, corona aged specimens under positive DC and negative DC charging

the other two samples with a higher mean lifetime. Table 2 shows the initial charge and mean lifetime for virgin specimens. Du *et al.* studied surface charge accumulation studies epoxy titania nanocomposites, using ESV. They have concluded that the charge decay relies upon the local condition that gets altered by the nanoparticles [30].

It is well observed from Figs. 8b and c and Tables 3 and 4 that a drastic reduction in the mean lifetime and the initial stored charge is observed for the samples as compared with their respective unaged virgin specimens. MC and NMC sample behave similarly even after corona charging though MC stored higher charge and has a higher mean lifetime. It is also observed that IN sample stores almost negligible charge under positive corona charging and has the highest mean lifetime among aged samples. Though charge stored is less

Table 2 Initial charge and mean lifetime of epoxy composite virgin specimens

Polarity	Positive DC		Negative DC	
	Initial charge, nC	Mean lifetime (τ)	Initial charge, nC	Mean lifetime (τ)
Sample				
IN	110.34	310.89	135.76	334.22
MC	155.88	340.90	155.13	374.67
NMC	132.97	326.78	136.42	367.80

Table 3 Initial charge and mean lifetime of specimens after 10 min of corona ageing of epoxy composite

Polarity	Positive DC		Negative DC	
	Initial charge, nC	Mean lifetime (τ)	Initial charge, nC	Mean lifetime (τ)
Sample				
IN	7.52	4.43	23.75	4.60
MC	28.75	2.89	47.50	3.79
NMC	22.53	2.44	41.25	3.45

Table 4 Initial charge and mean lifetime of specimens after 20 min of corona ageing

Polarity	Positive DC		Negative DC	
	Initial charge, nC	Mean lifetime (τ)	Initial charge, nC	Mean lifetime (τ)
Sample				
IN	5.28	4.13	20.0	3.60
MC	25.88	1.99	30.02	2.58
NMC	17.56	1.53	23.75	2.44

for IN sample higher mean lifetime indicates less damage to sample as compared to MC and NMC. A similar trend has been observed under either polarity but under negative polarity, more charge is stored this could be due to higher affinity of the surface for negative charges. Twenty minutes aged samples are further degraded compared to 10 min aged samples with lower mean lifetime.

3.4. Libs analysis: Fig. 9 shows emission spectra of the three samples in order IN, MC and NMC. In all the samples C, H, O

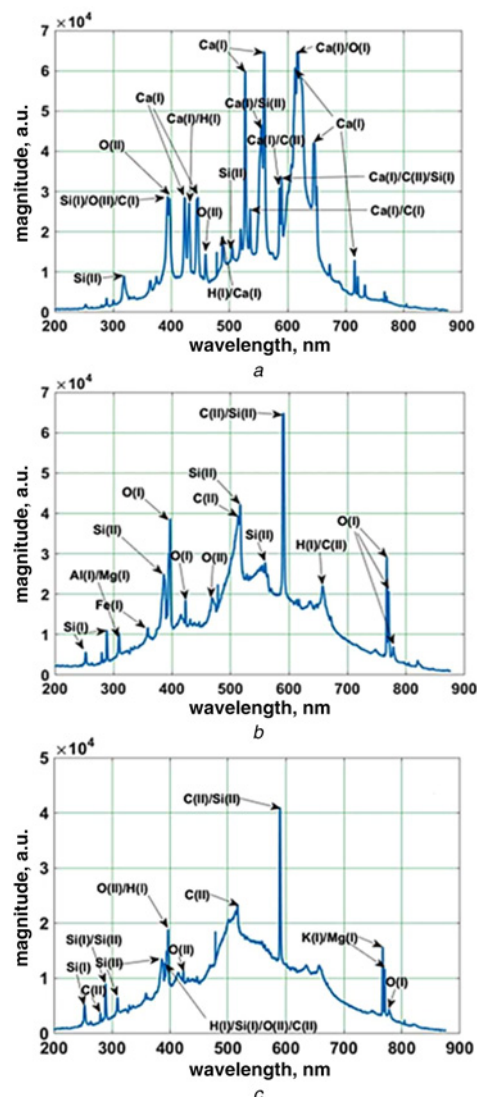


Fig. 9 Emission spectra of the samples in
a IN
b MC
c NMC

Table 5 Threshold fluence, plasma temperature and hardness of the epoxy composites (bracketed elements peaks are chosen to calculate plasma temperature)

Sample	Threshold fluence, mJ	Plasma temperature, K	Micro Vickers hardness
IN	6	17,276.78 (Ca)	41.9
MC	10.5	10,316.62 (O)	32.4
NMC	17	10,804.59 (Si)	35.4

peaks were observed. While in IN Ca was observed due to Wollastonite filler and Si peaks were observed in MC and NMC due to silica filler. The identification of all emission lines is based on comparison with NIST database [31].

In order to create plasma, the threshold fluence of the sample must be smaller than fluency of incident laser. Laser damage threshold or threshold fluence is intensity below which no laser induced damage is likely to occur. Laser damage may be broadly classified into two types: absorption-driven and dielectric-breakdown damage. Which type dominates depends on the material properties (absorption coefficient, specific heat, melting temperature, as well as defects that cause scattering and concentrated electric field effects), geometric properties of the sample (thickness, homogeneity, surface morphology), and of course the properties of the laser beam itself [32]. As shown in Table 5, NMC has highest threshold fluence than MC followed by IN.

Plasma temperature can be calculated based on measured spectroscopic quantities considering local thermal equilibrium using Boltzmann–Saha equation [33]

$$T_e = 1.44 \times \frac{E_2 - E_1}{\ln \left[\frac{I_1 \lambda_1 A_2 g_2}{I_2 \lambda_2 A_1 g_1} \right]} \quad (4)$$

where E_1 and E_2 are excited energy levels, g_1 and g_2 are statistical weights of excited energy levels 1 and 2, respectively, A_1 and A_2 are transition probabilities of states, I_1 and I_2 are intensities of particular atomic species at λ_1 and λ_2 wavelength, respectively, and T_e is the plasma electron temperature under the condition of local thermodynamic equilibrium.

IN has highest plasma temperature. Cowpe *et al.* [34] explored the impact of the hardness of bio-ceramic sample on plasma temperature and observed that the plasma temperature increases with increasing sample hardness. The outcome was additionally affirmed by the investigation led by Labutin *et al.* in which the temperature of Li–Al alloy plasma was proportional to micro-hardness of the alloy samples [35]. They supposed that less material is ablated with higher hardness of the sample, thus the plasma is featured by a higher temperature value, that is smaller the number of particles ablated higher the energy per particle, this tells us that IN sample has high hardness, same was observed with measured micro hardness value of the sample as shown in Table 5.

There is a marginal difference in plasma temperatures of MC and NMC as their elemental composition is same and they have a marginal difference in their hardness value. While the difference in threshold fluence among MC and NMC can be attributed to the presence of nano fillers in NMC. This shows that plasma temperature and threshold fluence are clear indicators of the different type of materials.

Fig. 10 shows a typical 3D picture of the crater formed by the laser pulse. Samples were subjected to an equal amount of laser energy as well as an equal number of pulses and depth of the crater formed was measured (Fig. 11). It is observed that the sample IN has the least depth of crater, while MC and NMC have nearly same amount of damage. This is in line with the expected

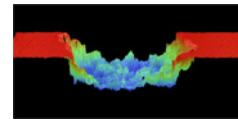


Fig. 10 Typical 3D photograph of the crater formed by the laser pulse

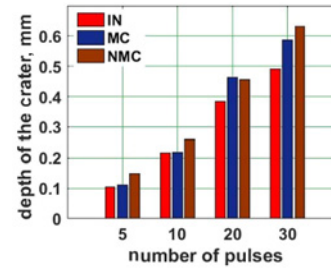


Fig. 11 Variation in depth of the crater with number of laser pulses applied

superior performance of IN sample which has higher plasma temperature and higher hardness.

4. Conclusions: Important conclusions arrived at based on the study are the following:

- Under the unaged condition, MC performance is better than NMCs followed by IN. Upon corona ageing IN performs better than MC and NMC due to its filler content, which reduces the damage due to corona ageing. Also, it is observed based on experimental studies that contact angle measurement alone is not sufficient to predict the variation in CIV.
- Charge accumulation studies show that both charge retention capability and mean lifetime of the samples reduced drastically upon corona ageing. IN performed better under aged conditions among the three samples with higher mean lifetime indicating less carbonisation of the surface.
- LIBS studies indicate that plasma temperature and threshold fluence are good indicators to classify the performance of the materials.
- Plasma temperature measured is also an indicator of the hardness of the material. The damage to the IN specimen is the least by laser abrasion, indicating higher resistance to discharges.

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