## USE OF PHASE RESOLVED PARTIAL DISCHARGES FOR STUDYING THE INCUBATION PERIOD OF ROOM TEMPERATURE FLASH SINTERING OF ZINC OXIDE

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Several attempts have been reported in the recent literature to lower the temperature of the flash sintering process. Flash sintering on zinc oxide was successfully carried out at room temperature under high electric fields by controlling the atmosphere (nature of the gas, pressure, humidity). In particular, Liu et al. [1] have shown that the water content strongly reduces the electric field at which the flash event happens. This phenomenon is likely to be caused by partial discharges within the porous sample and subsequent dielectric breakdown.

In this work, we study the early stages of the incubation period through the acquired Partial Discharges (PDs) patterns obtained by Phase Resolved Partial Discharge (PRPD) method. The setup consists in placing ceramic cylindrical samples in a climatic chamber between two platinum planar electrodes in the same configuration as flash sintering experiments. In this work, porous cylindrical samples of zinc oxide (average dimensions of 8 mm in diameter and 3.5 mm in height) are studied between 5°C and 65°C in humid air atmosphere whose relative humidity (RH) ranges between 10% and 90%. A 50 Hz AC voltage is applied to the sample and increased gradually up to 4 kV. We record either the PRPD patterns for 30 s at fixed voltage amplitude (increasing gradually the voltage amplitude for each experiment) or the time evolution of the PD amplitude (regardless of their phase) when linearly increasing the AC voltage. Then we examine the number, the amplitude and the pattern shape of the electric discharges. The average discharge current (in the 40 kHz - 800 MHz bandwidth) corresponding to the partial discharges occurring during the acquisition period exhibits a sharp increase out of the noise level of ~10 pA up to an almost constant value ranging between 10 nA and 1µA depending on the temperature and the relative humidity. The threshold electric field, for a given average discharge current of 1 nA, strongly depends on the relative humidity as it falls down from 4 kV/cm at 35°C and 10%RH to 3 kV/cm at 50%RH and 2.36 kV/cm at 90%RH at the same temperature. The threshold electric field is also affected by the temperature but does not follow a monotonic evolution. In addition to the modification of the threshold electric field, the maximum amplitude of partial discharges is strongly attenuated when the relative humidity increases. The pattern evolution with increasing AC voltage presents different characteristics that can be associated to various phenomena either within the porous sample volume, at its surface or at the interface with the electrodes. This study is supported by ERDF and the Walloon Region, in the frame of IMAWA-FLASHSINT research project (program 2014-2020).



[1] J. Liu, H. Rongxia and R. Zhang et al. / Scripta Materialia 187 (2020) 93-96

Figure 1 – Phase Resolved Partial Discharge patterns acquired for 30 s on porous ZnO samples at 7 kV/cm at 35°C and relative humidity of (a) 10%, (b) 50% and (c) 90%.