Enhancement of Dielectric Properties of Polyamide Enamel Insulation in High Voltage Apparatuses Used in Medical Electronics by Adding Nano Composites of SiO₂ and Al₂O₃ Fillers

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Abstract – In recent days, there was a significant development on the electrical, thermal, mechanical, physical, chemical, magnetic and optical properties of the polyamide enamel, varnish and other insulating materials by the addition of nano fillers to it. Enamel was used in High Voltage Apparatuses used in Medical Electronics as insulation. Insulating materials determine the life time of the electrical apparatuses. The life time of the insulating materials was improved by the addition of nano fillers to it. Hence the life time of the electrical apparatuses was improved by the mixing of nano fillers to the enamel. In this research, the basic dielectric properties of the enamel and enamel mixed with nano composites of silica and alumina were analyzed and compared with each other. The addition of nano fillers has improved the quality factor and capacitance of the enamel. It was also observed that the addition of nano fillers has reduced the dissipation factor and dielectric losses of the enamel. Heat produced by the dielectric losses was also reduced by adding nano fillers to it.

Keywords: Ball mill, SEM, Enamel, SiO₂, Al₂O₃, Medical electronics, Space electronics

1. Introduction

In Insulating materials are also called as Insulators or Dielectrics. Enamel and Epoxy resins are used in High Voltage Apparatuses which are used in medical electronics, TV and Radio Receivers and Space electronics. The satisfactory operation of the electrical apparatuses depends to a great extent upon the properties of the insulation used. Therefore the proper choice of insulating materials for the electrical apparatuses is of considerable importance [1]. In general the insulating materials used for the electrical apparatuses should have the following characteristics.

1. It should have higher insulation resistance to avoid the leakage current.
2. It should possess higher dielectric strength to avoid electrical break down in the electrical apparatuses.
3. It should have higher mechanical strength to withstand the mechanical stresses produced in the electrical apparatuses due to friction and mechanical handling of the apparatuses.
4. It should be non-hygroscopic. It should not absorb moisture from the environment. The moisture will decrease the insulation resistance and quicken the breakdown of the insulation.

5. It should be non-inflammable.
6. It should be economic.
7. It should not react with other chemicals.

All the dielectrics are insulators but all the insulators were not dielectrics. In dielectrics all the electrons are bound to their parent molecules and there were no free charges [2]. The dielectrics are non metallic materials with high specific resistance and have negative temperature coefficient of resistance. The dielectric characteristics of insulating materials are determined by the dielectric constant or relative permittivity of that material. The dielectric constant is used to determine the share of the electric stress which is absorbed by the material. It is defined as the ratio between the permittivity of the insulating medium and the permittivity of free space. Since it is the ratio of the same quantity it has no unit. It is used as a measure of the polarization in the dielectric material [3]. Polarization is defined as the process of producing dipoles in the dielectric materials by an external electric field. There are different types of polarization such as electronic polarization, ionic polarization, oriental polarization and space charge polarization. Polarization is measured in terms of polarization vector. The dipole moment per unit volume of the dielectric material is called as polarization vector. Greater the value of the dielectric constant, greater will be the capacitance of the insulating material [4].

The word nano means 1×10^{-9} m. The diameter of a single atom may vary from 0.1 to 0.5 nm. Generally they are obtained as colloids. The colloidal particles will have the tendency to remain as single crystal and hence they are called as nano crystals. Nano crystals possess electrical,

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electronic, mechanical, thermal, magnetic, physical, chemical and optical properties [5]. The nano particles exhibit an electronic behavior governed by the quantum physics and hence they are called as quantum dots. Nano materials are the materials having the size less than 100 nm at least in one dimension. Nano materials are layers in one dimension such as thin films or surface coatings [6]. Nano materials are tubes in two dimensions such as nano tubes and nano wires. Nano materials are particles in three dimensions such as precipitates, colloids and quantum dots. Nano science is defined as the study of phenomena and manipulation of materials at atomic, molecular and macro molecular scales. Nano technology is defined as the design, characterization, production and applications of structures, systems and devices by controlling the shape and size at the scale of 10^{-9} m. Nano fillers are added to the enamel to avoid tracking in polymeric insulation [7]. In the recent years, SiO_2 , TiO_2 , CNT, Barium Titanite, ZNO, ZrO_2 , Al_2O_3 are used as fillers for polymeric insulation such as enamel, varnishes, Bakelite and so on. Fillers can be added in the form of micro and nano level [8]. Nano fillers are added to the polymeric insulation to increase the performance and life time of the electrical apparatuses [9]. One such study is carried out based on the addition of SiO_2 , TiO_2 , CNT, ZNO, ZrO_2 , Al_2O_3 nano filler with enamel or varnish to improve the physical, chemical, thermal and electrical properties of the enamel or varnish. This research is conducted to improve the properties of the dielectric materials by using nano technology. The ultimate aim of this project is to improve the performance of the dielectric materials by adding nano fillers to it.

Advantages of nano dielectric materials:

- i. It is based upon molecular self-assembly
- ii. It was used to create many new materials
- iii. It has impact on environment and economics.
- iv. It is the engineering of functional systems at the molecular scale.
- v. It is used to make high performance products.

Nano fillers were used in the field of electrical and electronic engineering. Nano filler added insulating materials were used in the generation, transmission, distribution and utilization of electrical energy and hence these divisions of engineering were collectively called as Nano Electrical Energy Systems Engineering. Nano fillers can be used in the insulating materials used for cables, insulators, capacitors, transformers, motors, lightning arrestors, bushings, current transformers, potential transformers and CVT. Nano fillers can be used for the improvement of the performance of the several electrical and electronic devices and materials such as Nano capacitors based filters, Nano transformer based SMPS, Nano cables, Nano insulators, Nano powders used for welding rods and electrodes, Nano based rectifiers, Nano resistance, Nano engineering materials, Nano SiC Arrestors,

Nano ZnO Arrestors, Nano technology used in receivers and transmitters, Nano alloys, Nano capacitor based microphones, Nano electrets, Nano nuclear engineering, Nano thermo electric materials and so on.

Nano fillers added to the polymeric insulation would have the following advantages:

1. Higher resistance to partial discharge
2. Enhanced thermal properties
3. Lacking of erosion resistance
4. Matching of coefficient of thermal expansion
5. Thermal conductivity enhancement
6. Improved mechanical reinforcement
7. Increased abrasion resistance
8. Improved life time

2. Synthesis and Characterization of Silica and Alumina Nano fillers

Ball mill method is used to synthesis the nano fillers. Ball mill is an efficient tool for grinding many materials into fine powder. The ball mill is used to grind many kinds of mine and other materials. It is widely used in chemical industry. There are two ways of grinding: dry process and the wet process. The ball mill is key equipment for regrinding. It is widely used for the cement, the silicate product glass, ceramics and etc. Here the Silicon dioxide and Aluminum Di-oxide nano material is synthesized by using ball mill to convert micro particles to nano particles. The micro powders of silica and alumina are grinded by ball mill for 36 hours. The pulverize 6 planetary mono mill is universally applicable for quick dry or wet grinding of inorganic and organic samples for analysis, quality control, materials. The sample material is crushed and disintegrated in a grinding bowl by grinding balls. The grinding balls and the material in the grinding bowl are acted upon by the centrifugal forces due to the rotation of the grinding bowl about its own axis and due to the rotating supporting disc. As a frictional effect, the grinding balls running along the inner wall of the grinding bowl, and impact effect, the balls impacting against the opposite wall of the grinding bowl. After that the particle size is augmented by SEM. SEM is used to characterize the prepared nano fillers.

In SEM, the image is built up by using an electron probe of small diameter which scans the surface of the specimen in parallel straight lines. During surface observations, the electron probe scans the specimen. Secondary electrons are emitted and these are collected. Current is amplified and hence the image is formed on a TV screen. Magnification is done up to 300000. Images are also formed by collecting the scattered electrons and transmitted electrons.

Advantages of SEM

1. Large specimens could be examined.
2. Longer depth of focus was possible.

3. Structural details of the specimen were resolved in a precise manner.
4. Easier.
5. Thicker specimens could be examined by using transmitted electrons.
6. No labour was involved in preparing replicas.
7. Composition of the specimen was obtained from the characteristic x-rays.

2.1 SEM analysis before synthesization

The particle size of SiO_2 and Al_2O_3 before ball mill method is shown in Figs. 1 and 2.

2.2 Figures

Fig. 1. SEM analysis of SiO_2 at 50 μm

Fig. 2. SEM analysis of Al_2O_3 at 50 μm

2.3 SEM analysis after synthesisation

From the analyzed SEM image the particles are in the form of nano metric range varies for one area to other. The sizes of the particles as shown in Figs. 3 and 4 are in the range from 40 to 100 nm size.

Fig. 3. SEM analysis of SiO_2 at 5 μm

Fig. 4. SEM analysis of Al_2O_3 at 5 μm

3. Sample Preparation

The solid samples of enamel and nano composites filled enamel are prepared by radical initiator curing method. For proportionality of 80% of varnish and 20% of epoxy resins are taken. Curing agent such as DDM (Diamino Diphenyl Methane) is taken in proportion to epoxy resin (For 1g of resin 0.27g of DDM). The DDM is melted for 10 minutes at 60-80°C. The polyamide enamel, resin and melted DDM are mixed in a beaker. Before pouring the mixture into the die, it should be coated by anabond 666. It is placed in an oven at a temperature of 80°C for 1 hour. After that, the mixture is poured into the die which was coated by a Teflon sheet. The die is heated at 80° C for 2 hours for epoxy curing and 120° C for 3 hours. The die is cooled for 1 hour. Seven series of specimens were produced, each one with different filler content. The process involved for preparation of nano composites was revealed in Figs. 5 and 6.

3.1 Figures

Fig. 5. Sample Preparation

Fig. 6. Proposed works

4. Measurement of Dissipation Factor and Quality Factor as the Function of Temperature and Frequency

When a dielectric material is subjected to AC voltage, the electrical energy is absorbed by the material and was dissipated in the form of heat. This dissipation of energy is called as dielectric loss. It is measured in terms of dissipation factor. For dielectrics, the dissipation factor should be low. Dissipation factor and quality factor of the insulating materials is measured by Dielectric Spectroscopy. It measures the properties of the insulating materials as the function of temperature and frequency. The different types of samples are subjected to Dielectric Spectroscopy. Dissipation factor was inversely proportional to quality factor. When the quality factor increases, the value of the dielectric losses will be less.

Dielectric power loss was given as

$$P = V^2 * 2\pi * f * C * \tan \delta$$

where $\tan \delta$ was called as dissipation factor of the dielectric material

The dielectric power loss was dependent on $\tan \delta$, voltage, frequency and capacitance. The rise of the temperature, humidity, voltage and frequency would increase the value of the dielectric loss. The dissipation factor and quality of the enamel varies with the concentration of the nano and micro fillers, temperature and frequency. The variation of the dissipation factor and quality factor with the nano and micro filler concentration of alumina and silica with temperature and frequency were experimentally proved and were shown in the Figs. 7 to 16. The exact mechanism and the reasons for the

variation in the dielectric properties of the enamel with filler concentration, temperature and frequency are not known experimentally. But, the variations in the dielectric properties of the enamel with the filler concentration are shown clearly. The main causes and mechanisms responsible for the enhancement of the properties of the enamel with the filler concentration are under research.

4.1 Figures

Fig. 7. Variation of Dissipation factor as the function of temperature and frequency at 50°C for various samples

Fig. 8. Variation of Quality factor as the function of temperature and frequency at 50°C for various samples

Fig. 9. Variation of Dissipation factor as the function of temperature and frequency at 75°C for various samples

Fig. 10. Variation of Quality factor as the function of temperature and frequency at 75°C for various samples

Fig. 13. Variation of Dissipation factor as the function of temperature and frequency at 125°C for various samples

Fig. 11. Variation of Dissipation factor as the function of temperature and frequency at 100°C for various samples

Fig. 14. Variation of Quality factor as the function of temperature and frequency at 125°C for various samples

Fig. 12. Variation of Quality factor as the function of temperature and frequency at 100°C for various samples

Fig. 15. Variation of Dissipation factor as the function of temperature and frequency at 150°C for various samples

Quality factor increases as the function of temperature and frequency. Many researches are conducted on the property analysis of nano filler added enamel and varnishes. The approximate reasons for the improvement of the properties of the nano filler added enamel is shown in some researches. But the exact cause for the variation in the properties of the micro and nano filler mixed insulating materials are still under the research. Some researchers

were mainly focusing on the mechanical properties of the nano filler mixed enamel whereas a few are only concentrating their full time effort on the physical, chemical and thermal properties of the nano and micro filler mixed insulating materials. Only a few material science engineers and mechanical engineers are focusing on the electrical properties of nano filler mixed insulating materials. In recent days only, interdisciplinary researches

Fig. 16. Variation of Quality factor as the function of temperature and frequency at 150°C for various samples

are carried out on the dielectric, mechanical, thermal, physical and characterization of the nano filler mixed enamel. In this research also, only the basic dielectric properties of the enamel filled with nano composites are discussed. Researches on nano are emerging in the few decades only. Hence many research works are under progress. This paper will show a way for doing research in the field of nano polymeric insulation. There is no hiding of truths in this research. All the results are given as data for the easy understanding of the concepts. The readers will definitely get some innovative ideas to conduct research in the field of nano electrical apparatuses and nano dielectric materials. Some of the few important properties of the enamel filled with micro and nano composites of silica and alumina are analyzed and the results are compared with that of the standard enamel. Although there is an improvement in the properties of the nano filler mixed enamel is shown clearly, the basic mechanisms responsible for the improvement in the dielectric properties of the nano filler mixed enamel were still under research. Many researches should be conducted to study the basic mechanisms responsible for the improvement of the dielectric properties of the nano and micro filler added enamel, varnishes and dielectric materials.

5. Measurement of Capacitance as the Function of Temperature and Frequency

It is observed that when the frequency changes, the

values of the capacitance decreases. There is a deviation in the values of the capacitance of the enamel, enamel mixed with micro and nano composites of silica and alumina taken in different proportions. It has been recorded that the value of capacitance also changes with the addition of nano fillers to the enamel. The dielectric losses would also changes due to the change in the value of the capacitance of the sample. The value of the dielectric constant would also change with the variation in the capacitance of the sample. At 50°C, the value of the capacitance reaches the maximum value for the micro silica and alumina filler in 3:1 mixed enamel sample. Thermal energy has activated the increase in the value of the capacitance of the samples. Thermal energy is also responsible for the production of thermally initiated electrons. These electrons are responsible for thermal breakdown in the dielectric materials. When the temperature increases, the value of the capacitance will begin to decrease and the breakdown process will begin to start leading to thermal breakdown. The exact reason for the variation in the value of capacitance is not determined. But the addition of the nano composites of silica and alumina has shown an improvement in the value of the capacitance. The main reasons for the improvement in the values of the dielectric and thermal properties of the insulating materials with the addition of the nano fillers were under research. The variation of capacitance as the function of temperature and frequency for different samples is shown in the tables 1 to 5.

6. Conclusions

From these researches, it is observed the quality factor, capacitance of the enamel is improved by adding the micro and nano fillers of silica and alumina to it. The dissipation factor and dielectric losses are reduced by mixing micro and nano fillers to the enamel. Hence the life time of the insulation and the electrical apparatuses are improved by the addition of the nano fillers to the enamel. Similarly, various micro and nano fillers such as alumina, CNT, zirconia, titania, barium titanite, ZNO and silica can be added to the enamel to improve the performance of the dielectric materials. The suitable mechanisms responsible for the improvement of the physical, chemical, electrical, thermal, magnetic and optical properties of the nano filler

Table 1. Capacitance in Farad at 50°C

Sample	Frequency in Hz						
	50	100	1000	10000	100000	1000000	5000000
Micro Silica and Alumina (1:1) Mixed Enamel	7.45×10^{-11}	2.43×10^{-11}	1.91×10^{-11}	1.40×10^{-11}	1.22×10^{-11}	1.17×10^{-11}	1.28×10^{-11}
Micro Silica and Alumina (3:1) Mixed Enamel	9.41×10^{-11}	2.92×10^{-11}	2.41×10^{-11}	1.53×10^{-11}	4.31×10^{-11}	1.3×10^{-11}	2.42×10^{-11}
Nano Silica and Alumina (3:1) Mixed Enamel	4.86×10^{-11}	2.73×10^{-11}	2.96×10^{-11}	2.43×10^{-11}	2.25×10^{-11}	2.21×10^{-11}	1.36×10^{-11}
Nano Silica and Alumina (1:3) Mixed Enamel	3.24×10^{-11}	3.26×10^{-11}	2.29×10^{-11}	1.69×10^{-11}	1.481×10^{-11}	1.44×10^{-11}	1.66×10^{-11}
Nano Silica and Alumina (1:1) Mixed Enamel	9.36×10^{-11}	3.41×10^{-11}	2×10^{-11}	2.48×10^{-11}	2.27×10^{-11}	2.23×10^{-11}	2.29×10^{-11}
Micro Silica and Alumina (1:3) Mixed Enamel	2.425×10^{-11}	1.625×10^{-11}	4.25×10^{-11}	1.503×10^{-11}	1.608×10^{-11}	1.61×10^{-11}	2.09×10^{-11}
Enamel	5.674×10^{-11}	3.094×10^{-11}	2.40×10^{-11}	1.790×10^{-11}	1.568×10^{-11}	1.54×10^{-11}	1.79×10^{-11}

Table 2. Capacitance in Farad at 75 °C

Sample	Frequency in Hz						
	50	100	1000	10000	100000	1000000	5000000
Micro Silica and Alumina (1:1) Mixed Enamel	6.146×10^{-11}	2.671×10^{-11}	5.92×10^{-11}	2.4×10^{-11}	2.21×10^{-11}	2.1×10^{-11}	2.2×10^{-11}
Micro Silica and Alumina (3:1) Mixed Enamel	7.392×10^{-11}	3.019×10^{-11}	3.14×10^{-11}	2.57×10^{-11}	2.36×10^{-11}	2.3×10^{-11}	2.4×10^{-11}
Nano Silica and Alumina (3:1) Mixed Enamel	5.99×10^{-11}	2.788×10^{-11}	2.99×10^{-11}	2.45×10^{-11}	2.27×10^{-11}	2.23×10^{-11}	2.238×10^{-11}
Nano Silica and Alumina (1:3) Mixed Enamel	6.174×10^{-11}	3.448×10^{-11}	2.34×10^{-11}	2.73×10^{-11}	2.5×10^{-11}	2.46×10^{-11}	2.62×10^{-11}
Nano Silica and Alumina (1:1) Mixed Enamel	7.238×10^{-11}	3.457×10^{-11}	6.05×10^{-11}	2.49×10^{-11}	2.31×10^{-11}	2.26×10^{-11}	2.32×10^{-11}
Micro Silica and Alumina (1:3) Mixed Enamel	5.413×10^{-11}	2.882×10^{-11}	6.06×10^{-11}	2.27×10^{-11}	2.6×10^{-11}	2.6×10^{-11}	2.1×10^{-11}
Enamel	5.47×10^{-11}	3.082×10^{-11}	2.42×10^{-11}	1.79×10^{-11}	1.57×10^{-11}	1.55×10^{-11}	1.82×10^{-11}

Table 3. Capacitance in Farad at 100 °C

Sample	Frequency in Hz						
	50	100	1000	10000	100000	1000000	5000000
Micro Silica and Alumina (1:1) Mixed Enamel	5.9×10^{-11}	6.51×10^{-11}	3.92×10^{-11}	2.38×10^{-11}	2.21×10^{-11}	2.17×10^{-11}	2.2×10^{-11}
Micro Silica and Alumina (3:1) Mixed Enamel	6.9×10^{-11}	5.10×10^{-11}	3.19×10^{-11}	2.6×10^{-11}	2.38×10^{-11}	2.34×10^{-11}	2.5×10^{-11}
Nano Silica and Alumina (3:1) Mixed Enamel	5.0×10^{-11}	6.70×10^{-11}	3.02×10^{-11}	2.46×10^{-11}	2.28×10^{-11}	2.23×10^{-11}	2.37×10^{-11}
Nano Silica and Alumina (1:3) Mixed Enamel	6.12×10^{-11}	5.57×10^{-11}	3.41×10^{-11}	2.76×10^{-11}	2.52×10^{-11}	2.46×10^{-11}	2.71×10^{-11}
Nano Silica and Alumina (1:1) Mixed Enamel	7.69×10^{-11}	5.52×10^{-11}	3.09×10^{-11}	2.52×10^{-11}	2.32×10^{-11}	2.28×10^{-11}	2.35×10^{-11}
Micro Silica and Alumina (1:3) Mixed Enamel	5.92×10^{-11}	6.71×10^{-11}	3.06×10^{-11}	2.48×10^{-11}	2.61×10^{-11}	2.61×10^{-11}	2.1×10^{-11}
Enamel	6.13×10^{-11}	3.26×10^{-11}	2.44×10^{-11}	1.77×10^{-11}	1.51×10^{-11}	1.46×10^{-11}	1.69×10^{-11}

Table 4. Capacitance in Farad at 125 °C

Sample	Frequency in Hz						
	50	100	1000	10000	100000	1000000	5000000
Micro Silica and Alumina (1:1) Mixed Enamel	4.9×10^{-11}	2.78×10^{-11}	1.98×10^{-11}	1.4×10^{-11}	1.21×10^{-11}	1.16×10^{-11}	1.28×10^{-11}
Micro Silica and Alumina (3:1) Mixed Enamel	5.8×10^{-11}	2.98×10^{-11}	2.22×10^{-11}	1.61×10^{-11}	1.38×10^{-11}	1.35×10^{-11}	1.51×10^{-11}
Nano Silica and Alumina (3:1) Mixed Enamel	6×10^{-11}	2.98×10^{-11}	2.07×10^{-11}	1.47×10^{-11}	1.29×10^{-11}	1.23×10^{-11}	1.4×10^{-11}
Nano Silica and Alumina (1:3) Mixed Enamel	6.5×10^{-11}	3.54×10^{-11}	2.5×10^{-11}	1.78×10^{-11}	1.54×10^{-11}	1.47×10^{-11}	1.7×10^{-11}
Nano Silica and Alumina (1:1) Mixed Enamel	8.1×10^{-11}	2.55×10^{-11}	2.08×10^{-11}	1.53×10^{-11}	1.33×10^{-11}	1.3×10^{-11}	1.6×10^{-11}
Micro Silica and Alumina (1:3) Mixed Enamel	6.1×10^{-11}	1.21×10^{-11}	4.39×10^{-11}	1.52×10^{-11}	1.6×10^{-11}	1.61×10^{-11}	2.0×10^{-11}
Enamel	5.01×10^{-11}	3.61×10^{-11}	2.42×10^{-11}	1.74×10^{-11}	1.49×10^{-11}	1.44×10^{-11}	1.67×10^{-11}

Table 5. Capacitance in Farad at 150 °C

Sample	Frequency in Hz						
	50	100	1000	10000	100000	1000000	5000000
Micro Silica and Alumina (1:1) Mixed Enamel	4.96×10^{-11}	2.64×10^{-11}	1.99×10^{-11}	1.41×10^{-11}	1.21×10^{-11}	1.17×10^{-11}	1.3×10^{-11}
Micro Silica and Alumina (3:1) Mixed Enamel	5.45×10^{-11}	3.24×10^{-11}	2.28×10^{-11}	1.63×10^{-11}	1.39×10^{-11}	1.5×10^{-11}	1.5×10^{-11}
Nano Silica and Alumina (3:1) Mixed Enamel	5.9×10^{-11}	2.86×10^{-11}	2.13×10^{-11}	1.91×10^{-11}	1.31×10^{-11}	1.6×10^{-11}	1.4×10^{-11}
Nano Silica and Alumina (1:3) Mixed Enamel	6.8×10^{-11}	3.47×10^{-11}	2.42×10^{-11}	1.73×10^{-11}	1.49×10^{-11}	1.62×10^{-11}	1.72×10^{-11}
Nano Silica and Alumina (1:1) Mixed Enamel	6.79×10^{-11}	3.83×10^{-11}	2.2×10^{-11}	1.94×10^{-11}	1.4×10^{-11}	1.5×10^{-11}	1.77×10^{-11}
Micro Silica and Alumina (1:3) Mixed Enamel	5.69×10^{-11}	2.23×10^{-11}	4.12×10^{-11}	1.39×10^{-11}	1.6×10^{-11}	1.61×10^{-11}	1.09×10^{-11}
Enamel	5.83×10^{-11}	3.09×10^{-11}	2.44×10^{-11}	1.73×10^{-11}	1.46×10^{-11}	1.41×10^{-11}	1.63×10^{-11}

added enamel are under research.

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