SEM and EDX analysis of The Electrode System for Partial Discharge Inception Voltage Measurement and Arcing Test of The Mineral Oil

Norasage Pattanadech[1], Fari Pratomosiwi [1], Martin A. Baur[2], Michael Muhris[1]

[1]Institute of High Voltage Engineering and System Management.
Inffeldgasse 18, A - 8010 Graz, [2]Baur Prüf- und Messtechnik GmbH
Raiffeisenstr. 8, A - 6832 Sulz, Austria

Abstract: The aim of this paper is to describe the Scanning Electron Microscope (SEM) and the Energy Dispersive X - Ray (EDX) test results of the original and tested electrodes used for Partial Discharge Inception Voltage (PDIV) measurement, as well as for the arcing test of mineral oil. The experimental investigations were performed with the mineral oil, Nynas 4000x, with the water content not more than 10 ppm under room temperature. For PDIV test, the tungsten needle electrodes with the tip radius of 10µm, 20µm, and 40µm respectively were used as the high voltage electrode while the brass plane electrode with 75 mm diameter was used as the grounded electrode with a gap distance of 50 mm. The test circuit was set up according to IEC 60270. The test procedure was performed in accordance with IEC 61294. For the arcing test, the tungsten rod electrodes with the tip diameter of 1 mm, and 2 mm with the curvature of 0.2 mm were used as the high voltage electrode while the brass plane electrode of 75 mm diameter as the grounded electrode with the gap distance of 0.3 mm and 0.8 mm respectively. The test experiment was modified from IEC 60156. The erosion of electrodes used for the mineral oil testing was examined by SEM techniques. SE images, BSE images and EDX spectrum of the original and tested electrodes were produced. The topography, the morphology, and the EDX spectra of the examined electrodes are analyzed. From the test results, there was no evidence to show the erosion of the electrodes after they were used for PDIV and arcing test. It can also be argued that the investigated tungsten needles, rods and brass plane electrodes can be used for PDIV testing and for arcing test without the problem of erosion. In addition, carbon was the main contamination created at the surface of the tested electrodes. The development of carbon was highly possible from the degradation of mineral oil.

Keywords: Scanning Electron Microscope; Partial Discharge Inception Voltage; Arcing test; Mineral oil

1. Introduction

Mineral oil is predominantly used as liquid-insulating material for high voltage equipment, especially power transformers. The mineral oil provides not only an excellent electrical insulation but an outstanding cooling property also. The quality of such insulation oil plays an important role in the performance of the transformers which are expected to function reliably and efficiently for many years [1]. To assure that the mineral oil operates in good condition, the oil quality has to be examined regularly. Simultaneously, the good maintenance of the transformer needs to be continuously performed. Many test standards for verifying the mineral oil characteristics have been proposed such as IEC 60156[2], ASTM D 877[3] or ASTM D 1816[4]. Partial Discharge Inception Voltage (PDIV) is one of the test techniques used to investigate the mineral oil characteristics. Currently only the standard IEC 61294 is available for PDIV measurement of liquid insulation. IEC 61294 recommends needle - sphere electrode for such PDIV measurement [5].However, there are some questions about the validity and sensitivity of this PDIV measurement technique[6]. Recently, the effort to improve the PDIV

Received: February 26th, 2012. Accepted: December 7th, 2012

measurement of liquid insulation has been discussed and carried out continually. Additionally, new concepts for PDIV measurement of the insulation liquids have been proposed as in [7-12]. Focusing on the electrode system for PDIV measurement of the mineral oil, a needle – plane electrode is also used by some research groups [9-15].This electrode type shows the good performance for PDIV testing of the mineral oil.

Unfortunately, there is a lack of study for the erosion or damaging of the needle – plane electrode system used for PDIV testing. Moreover, the erosion study of the arcing rod is very useful for evaluating the deformation of the electrode system used for arcing phenomena research.

2. Erosion of the electrode used for electrical testing of mineral oil

The erosion of the electrodes may occur after the electrodes are used for insulation tests for a certain time. The erosion process is accelerated by the persistent discharges or arcing of the electrode system. The erosion of the electrodes is caused partly by the evaporation of the metal from electrode spots during the arcing [16]. Examples of the needle tip erosion used for three standard lightning impulse breakdown testings, 80kV negative polarity and 120 kV positive polarity, of perfluoropolyether (PFPE) and transformer oil (AGIP ITE 360) had been reported in [17]. The shape of the needles is dramatically distorted compared to the original one after the impulse breakdown test as shown in Figure 1.Generally, electrode materials, arc discharge voltage, arc current, arc temperature, and the insulating medium characteristics are the main factors of the electrodes´ erosion. The appearance of electrode spots relates to the electrode erosion behavior, higher temperature and smaller spot size leading to more erosion. Unfortunately, the erosion quantification is a difficult task. Weight loss is not a good measure of erosion because mass transport and redeposit ion in regions away from the arc attachment obscure the effect of the arc´s operation [18]. An example of arc erosion rates of different material tested by 12,000 ampere, 60 Hz, half cycle arc is illustrated in Figure 2[19]. Furthermore, arcing in the mineral oil generates bubbles, gases, carbonization and other consequences. The carbonization appears as some black deposits in the oil and finally degrades the oil characteristics.

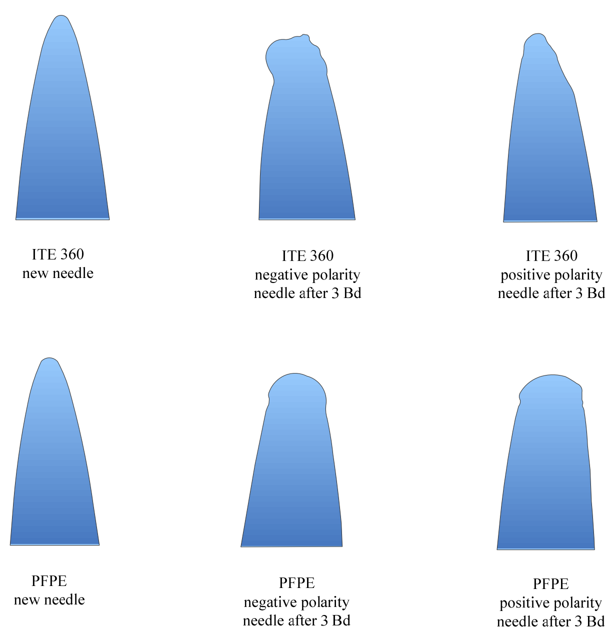


Figure 1. Needle tips before and after three standard lightning breakdown test of transformer oil (ITE 360) and PFPE for both polarities [adapted from 17]

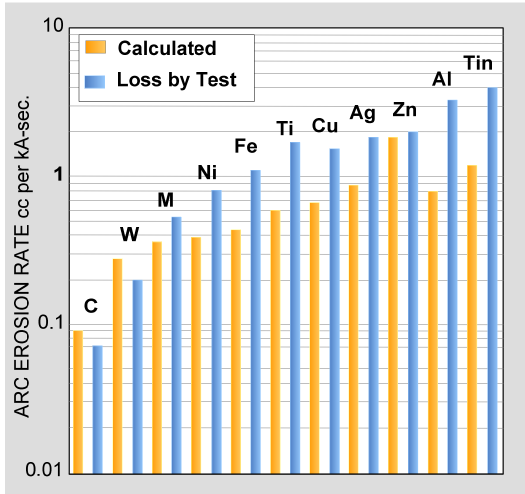


Figure 2. Comparison of measured arc erosion rates and calculated vaporization rates of different elements, based on half cycle arc of about 12,000 ampere, 60 Hz [adapted from 19]

3. SEM and EDX test techniques

To study electrode erosion, Scanning Electron Microscope (SEM) is a very powerful instrument. The general structure of SEM equipment is depicted in Figure 3[20]. With SEM, it permits the observation and characterization of materials on a nanometer to micrometer scale [21]. The topography of electrodes is acquired by reading the secondary electrons (SE images). Moreover, the image of the surface morphology of a specimen as well as the information of the surface composition can be identified by reading the backscattered electrons (BSE images) and the Energy Dispersive X – Ray (EDX) respectively. Figure 4 shows beam specimen interaction signals released by the tested specimen after it undergoes the electron beam. These beam specimen interactions are responsible for a multitude of signal types: secondary electrons, backscattered electrons, X- rays, auger electrons, cathadoluminescence. Details of SEM and EDX analyses can be found in [21-23].

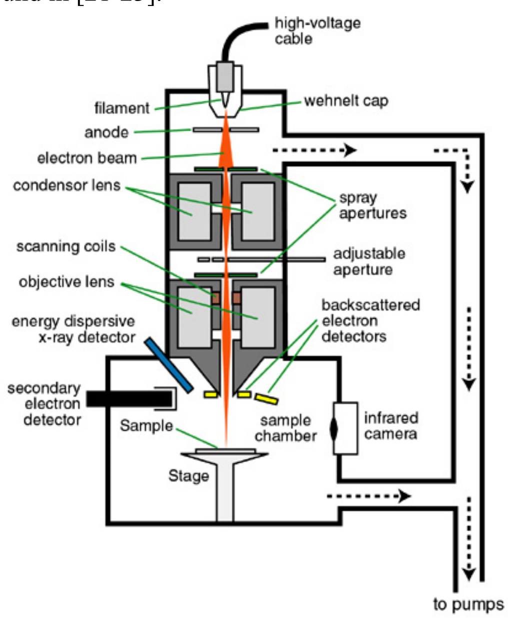


Figure 3. The structure details of the SEM [20]

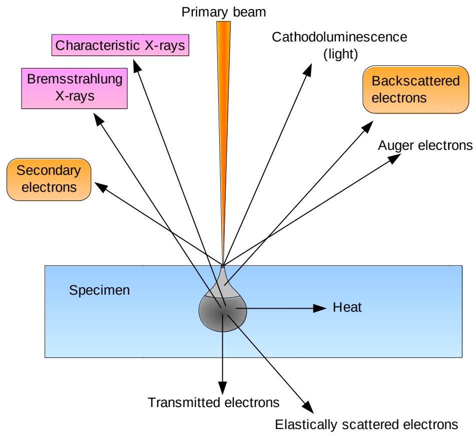


Figure 4. Beam specimen interaction signals caused by electron beam (primary beam) [24]

4. Test procedure

The experiment can be divided into 2 parts: The former was the mineral oil testing experiment, the PDIV experiment and arcing test; the latter was the SEM and EDX investigation of the original and tested electrodes.

A. Mineral oil testing

A.1. PDIV testing

The tungsten needle electrodes with the tip radius of 10µm, 20µm, and 40µm respectively were used as the high voltage electrode while the brass plane electrode with 75 mm diameter was used as the grounded electrode. The gap distance of the electrode system was set up at 50 mm. The test circuit was set up according to IEC 60270 [25]. The test procedure was performed in accordance with IEC 61294 [5]. The test voltage was increased with a rate of 1 kV/s from zero until PDIV occurred. Then, PDIV was recorded. Each needle was tested ten times and nine needles of each needle tip radius were investigated. After that, the PDIV mean value of each electrode configuration was computed. The PDIV test circuit is shown in Figure 5.

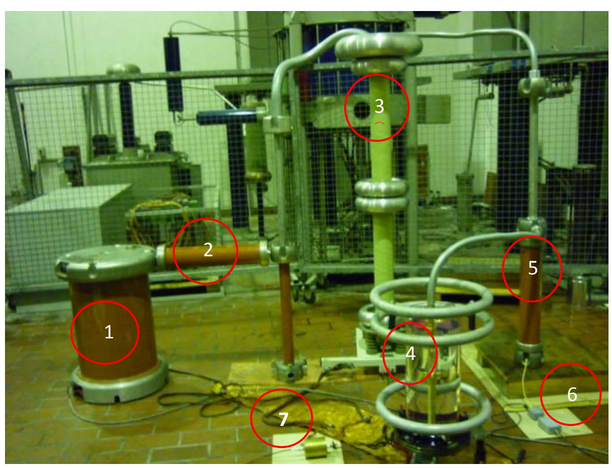


Figure 5. PDIV test circuit set up

Where 1. High-voltage supply 50 kV, 2. Current limiting resistor 50 kΩ, 3. Capacitive voltage divider 200 kV, ratio 2,000:1, 4. Test vessel: needle – plane electrode arrangement, 5. Coupling capacitor 100 kV,100 pF, 6. Coupling device and 7. Shunt resistor 50 Ω.

A.2. Arcing test

The tungsten rod electrodes with the tip diameter of 1 mm, and 2 mm with the curvature of 0.2 mm were used as the high voltage electrode, while the brass plane electrode of 75 mm diameter was used as the grounded electrode. The gap distance of the electrode system was set up at 0.3 mm and 0.8 mm respectively. The test experiment was modified from IEC 60156[2]. The test voltage was applied to the electrode system from zero until a complete arcing occurred. Then the test voltage was kept at that arcing voltage level for 10 seconds. The arcing current and arcing voltage were recorded. The oil sample was tested with the same procedure six times for each arcing rod. Then, the mean value of the arcing voltage and arcing current of each electrode configuration was computed.The arcing test circuit is illustrated in Figure 6.

Figure 6. Arcing test circuit set-up

Where 1. High-voltage supply 8/16/32 kV, 5.5 kVA, 2. Capacitive voltage divider 200 kV, ratio 2,000:1, 3. Test vessel, rod – plane electrode arrangement, 4. Shunt resistor 50 Ω, 5. Multimeter

B. SEM and EDX Investigation

The original and tested electrodes, six needles, six rods, eight plane electrodes, were randomly selected to investigate by SEM and EDX test techniques. SEM and EDX investigation was performed by FELMI-ZFE, The Austrian Centre for Electron Microscopy and Nano Analysis. SE images, BSE images and EDX spectrum of the electrodes were created and analyzed.

V. Test results

A.PDIV and Arcing test results

According to the experiment, the PDIV of the mineral oil (U_{PDIV}) tested with the needle – plane electrode system strongly depends on the needle tip radius. The PDIV test results of 10µm, 20µm, and 40µm tip radius needles are shown in Table 1. For acing test, the mean value of the arcing voltage (Uarc) and the arcing current (Iarc) of the mineral oil are illustrated in Table 2.

Table 1. Mean value of U_{PDIV} of the electrode system as a function of needle tip radius with σ as standard deviation

Needle tip radius	U_{PDIV} (kV)	σ (kV)
10 µm	31.2	1.9
20 µm	35.8	2.0
40 µm	43.2	2.6

Table 2. Mean value of Uarc and Iarc of the rod – plane arrangements with σ_u as voltage standard deviation and σ_i as current standard deviation

Rod diameter (mm)	**Gap distance (mm)**	**Arcing parameters**			
		Uarc(kV)	**σ_U (kV)**	**Iarc (mA)**	**σ_I (mA)**
1	0.3	9.9	0.9	372	32
	0.8	17.1	1.4	141	17
2	0.3	14.5	0.9	125	19
	0.8	20.5	1.2	163	12

B. SEM and EDX test results for original and PDIV tested needles

SEM and EDX test results of the needles, 10µm, 20µm, and 40µm tip radius, before and after they were used for PDIV testing of the mineral oil, were relatively similar. The examples of the 20 µm tip radius needle topography obtained from SE images and the needle morphology acquired from BSE images are illustrated in Figure 7.

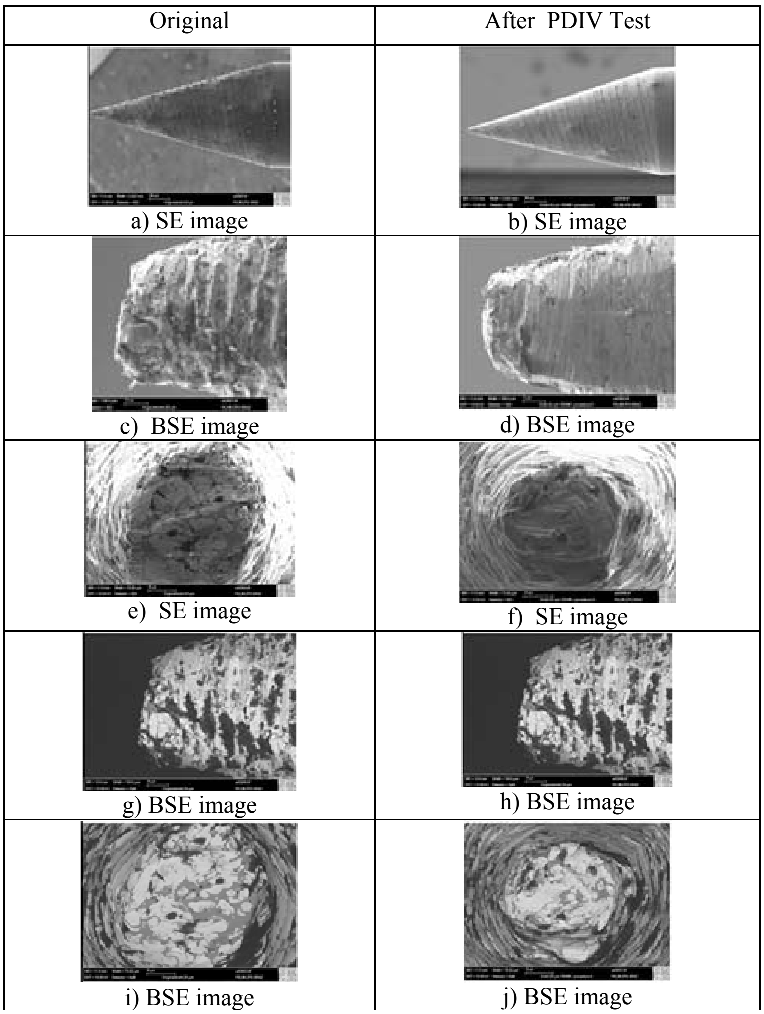
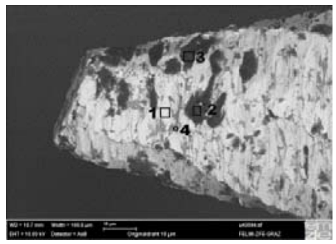
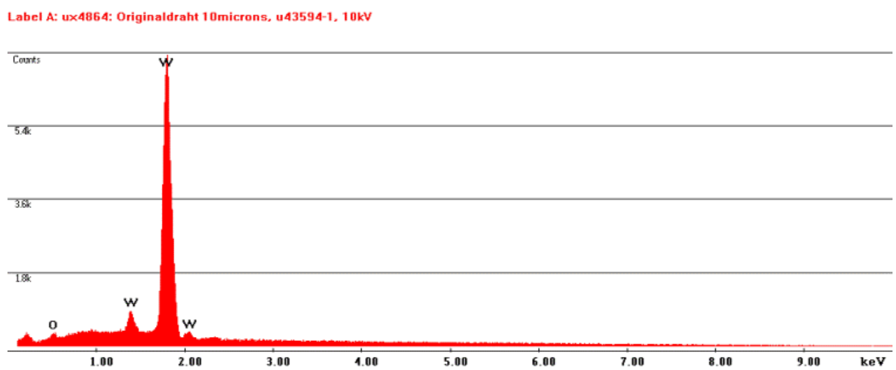


Figure 7. SE and BSE images of the 20 µm tip radius needle; SE images; longitudinal view of a)original needle, b)PDIV tested needle; BSE images; large scale image of c)original needle, d)PDIV tested needle; SE images; cross view of e)original needle, f)PDIV tested needle, BSE images; large scale image of g)original needle, h)PDIV tested needle; cross views of i)original needle, j)PDIV tested needle

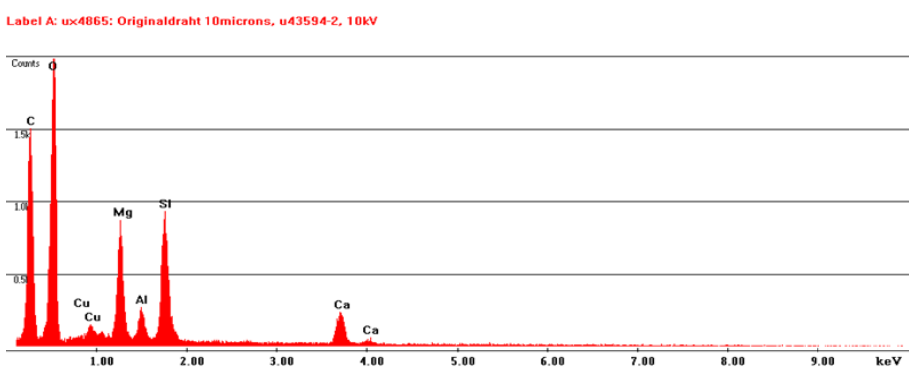
BSE images revealed that both original and PDIV tested needles comprised of at least 2 types of material. To analyze the composition elements, the EDX examination was conducted. EDX spectra generated by the areas at the tip of the original needle revealed that tungsten was the major element and carbon was the minor element. The example of EDX analysis at the tip of the 10 µm tip radius needle is shown in Figure 8. Furthermore, oxygen, sometimes with high count rate, copper, calcium and aluminum were found, as well as silicon and magnesium.



a) Interested areas,1-4, for EDX analysisof the 10 µm tip radius needle



b) EDX analysis of interested area 1



c)EDX analysis of the interested area 2

Figure 8. EDX analyzes of the interested areas of the original 10 µm tip radius needle

C. SEM and EDX test results fororiginal and arcing tested rods

The SE images and BSE images of the original and arcing tested rods with a diameter of 1 mm, gap spacing of 0.8 mm, arcing current 132.9 mA, arcing current density 16.93 A/cm^2 are illustrated in Figure 9 a) –f) and for gap spacing of 0.3 mm, arcing current 389 mA, arcing current density 49.53 mA/cm^2 as shown in Figure 9 g) –j) respectively.

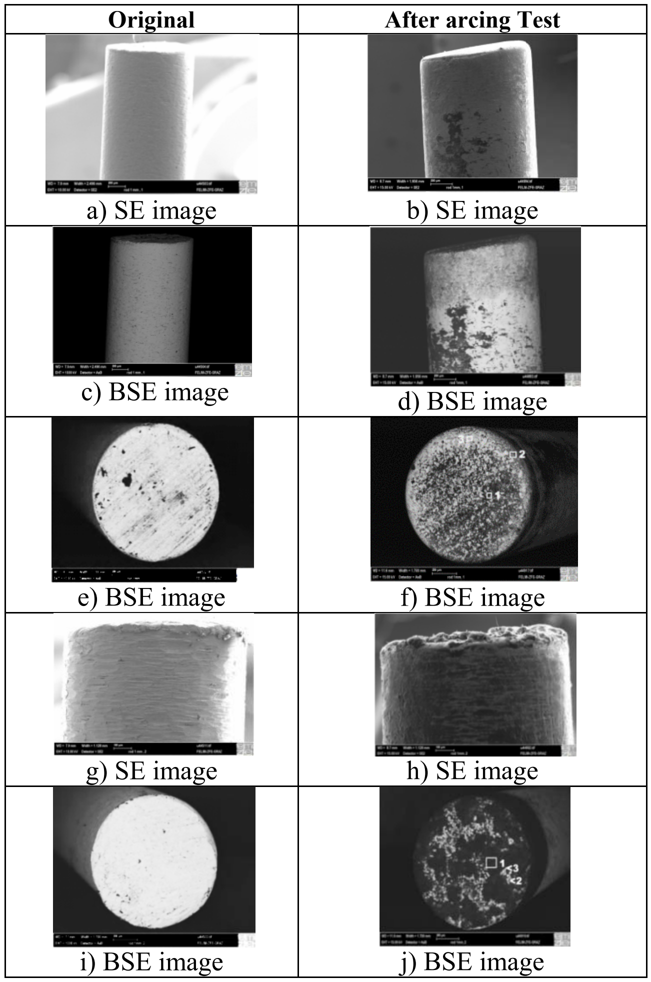


Figure 9. SE image and BSE image comparison of the 1 mm diameter rod before and after arcing test with different current densities a) - f) with current density of 16.93 A/cm^2 and g) - j) with current density of 49.53 mA/cm^2; SE images; longitudinal view of a)original rod, b)arcing tested rod; BSE images; longitudinal view of c)original rod, d)arcing tested rod; cross view of e)original rod, f)arcing tested rod; SE images; longitudinal view of g)original rod, h)arcing tested rod; BSE images; cross view of i)original rod, j)arcing tested rod

The topography at the tip of the arcing tested rod as shown in Figure 9 h)has clearly changed from the original, Figure 9 g), because it was covered by the contamination layer generated from the arcing phenomena. However, there was no evidence to show that any part of the rod melted or eroded. The new composition was investigated by the EDX technique. Figure 10 represents the example of the composition elements of the area of interest, area 2 of Figure 9 j), of the tested rod. The main component was comprised of tungsten and carbon.

The SE images and BSE images of the original and arcing tested rods with a diameter of 2 mm with the curvature of 0.2 mm, gap spacing of 0.8 mm, arcing current 163.2 mA, arcing current density 5.20 mA/cm^2 are illustrated in Fig. 11a) – f) respectively.

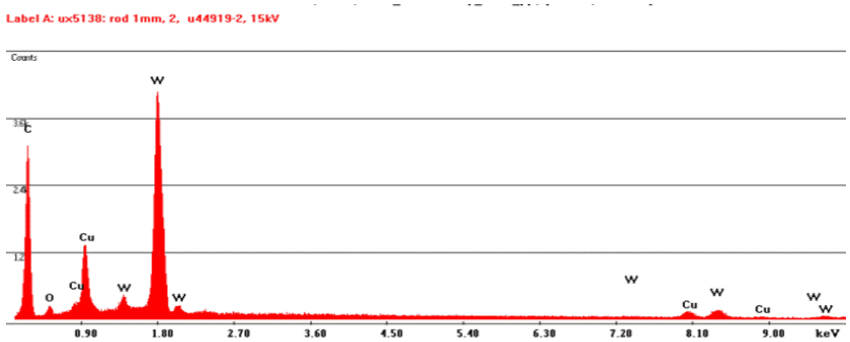


Figure 10. EDX-analysis of the interested area 2 of figure 9j

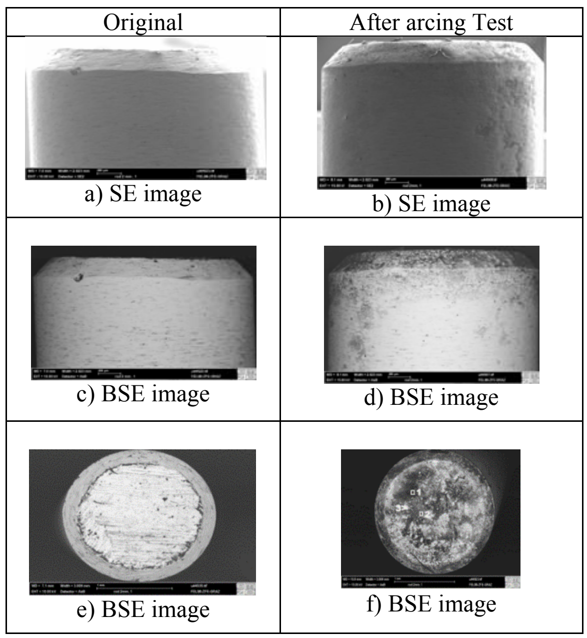


Figure 11. SE image and BSE image comparison of the 2 mm diameter rod with the curvature of 0.2 mm before and after arcing test; SE images; longitudinal view of a)original rod, b)arcing tested rod; BSE images; longitudinal view of c)original rod, d)arcing tested rod; cross view of e)original rod, f)arcing tested rod

The main component of other arcing tested rods was also tungsten and the main component of the contamination was carbon as well.

D. SEM and EDX test results for original and tested plane electrodes

SEM and EDX techniques were employed for analysis the plane electrodes as well. The main component of the original plane electrode was copper and zinc. For the PDIV tested plane electrodes, the surface morphology was a bit changed. BSE images showed the clearly changing of the surface morphology of the arcing tested plane electrodes especially at the arcing point. However, there was no evidence to show that any part of the tested plane electrodes melted or eroded. Figure 12 and Figure 13 represent the BSE images of the PDIV and the arcing tested plane electrodes respectively.

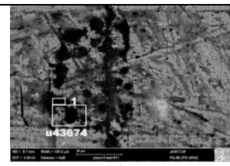
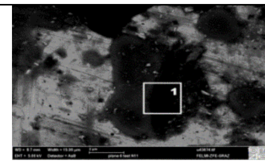
Big picture	Zoom in
 a)	 b)

Figure 12. BSE images of the PDIV tested plane electrode after 10 time tests with 20µm needle tip radius, at 36 kV PDIV level

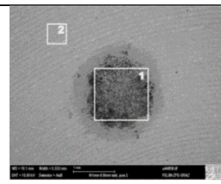
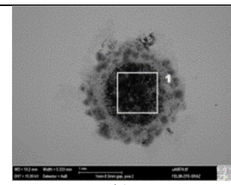
Arcing position of the arcing test plane electrode	
 a)	 b)

Figure 13. BSE images of the arcing tested plane electrode after testing with the 1 mm diameterrod; a)gap distance 0.8 mm, arcing current 132.9 mA, arcing current density 16.93 A/ cm^2 b)gap distance 0.3 mm, arcing current 389 mA; arcing current density 49.53 A/ cm^2 .

BSE images revealed that the surfaces of PDIV and arcing tested plane electrodes comprised of at least 3 types of materials. EDX spectrum as depicted in Figure 14 shows that the major component consists of carbon, zinc and copper. Carbon was generated from the degradation of the mineral oil during the existence of partial discharge and the arcing process. Zinc and copper were the major elements for manufacturing the plane electrodes.

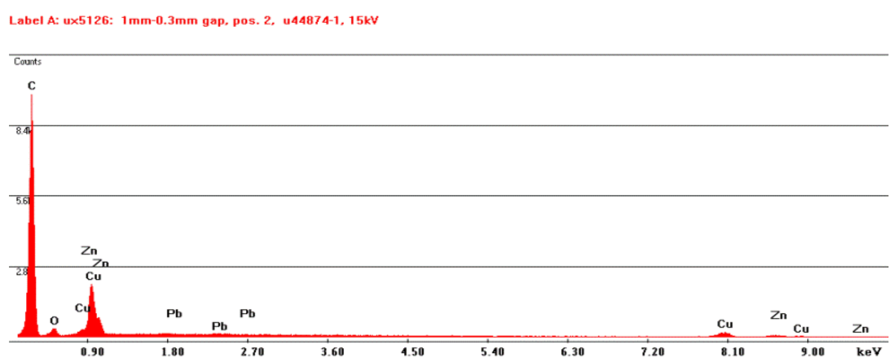


Figure 14. EDX-analysis of the interested area 1 of Figure 13b

6. Conclusion

The SEM and EDX test results of the needle – plane and the rod – plane electrodes for the PDIV and the arcing test of the mineral oil may be concluded as the following:

1. There was no evidence to show the erosion or melting of the needles, rods and brass plane electrodes after they were used for PDIV and arcing test. It can be stated that the tungsten

needles with tip radius of 10 µm, 20 µm, and 40 µm can be used for PDIV testing without the problem of erosion. Rods with 1 mm or 2 mm diameter including the brass plane electrode scan be used for arcing test also without the problem of erosion.
2. Carbon, very likely generated from the degradation of mineral oil, was found at the surface of electrodes; this was especially present at the tip of the arcing tested rods and at the arcing point of the tested plane electrodes.
3. The intensity of carbon depended on the arcing current density. The collected carbon on the tip of the arcing tested rod changed the topography and the surface morphology of the original rod which may affect the scattering of the arcing voltage and the arcing current of the mineral oil. At low current density, most carbon was found at the plane electrodes. With higher current density, carbon was found at the plane electrode and also the rod.

Acknowledgment

The authors would like to express their thanks to Dipl.-Ing. Dr.techn. Univ.-Doz. Peter Pölt and Dipl.-Ing. Dr.techn. Stefan Mitsche for SEM investigation. The financial support for this research work is from BaurPrüf- und Messtechnik GmbH.

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Norasage Pattanadech received B.Eng and M.Eng degree in electrical engineering from King Mongkut's Institute of Technology Ladkrabang (KMITL) in 1997 and Chulalongkom University in 2001 respectively. He joined Mahanakom University of Technology in 200 - 2003 before working in King Mongkut Institute of Technology Ladkrabang, Bangkok, Thailand until now. He is currently also studying for PhD in the Institute of High Voltage Engineering and System Management, Graz University of Technology, Austria. His research activities have been mainly involved Partial discharge in insulating liquid, solid insulator characteristics, high voltage testing and equipment, and Electromagnetic Compatibility.

Fari Pratomosiwi was born in Indonesia in 1985. He received B.Eng. and M.Eng. degrees in electrical engineering from Bandung Institute of Technology (ITB), Indonesia in 2007 and 2009, respectively. Now, he is currently PhD student in the Institute of High Voltage Engineering and System Management, Graz University of Technology, Austria. His major research interests are high voltage insulating materials for substitutes of insulating mineral oils and partial discharge.

Martin A. Baur, born 1944 in Feldkirch, Austria. He received his electrical engineering degree at the TGM, Institute of Technology, Vienna, Austria in 1969. 1978 until 2009 he was active in R&D, general manager of BAUR Prüf-und Messtechnik GmbH, Sulz, Austria, specialized in the field of high voltage cable testing, fault location and oil testing devices. Today, he is active as consultant in BAUR. He is a Member of IEEE since 1981 and IEEE SA since 1999. He has been active within the IEEE Power Engineering Society and Insulator Conductor Committee, 1998 appointed as Austrian Government official engineering expert witness, he is a member Swiss and Austrian Electro technical Associations SEV and ÖVE and a member since 1985 of ASTM subcommittee D 027.05 and since 2007 an expert member in the IEC technical committee TC 10 for electrical testing of insulating liquids and gases.

Michael Muhris an emeritus professor at the High Voltage Engineering and System Management of Graz University of Technology (TU Graz), Austria. Since 1990, he has been the Head of the Institute and Test Institute of High Voltage Engineering and System Management of TU Graz. He is a member ÖVE, ÖGE, DKE, IEEE, IEC and CIGRE (convenor of 5 working groups). He has published more than 170 publications and reports and also more than 160 lectures. He received Honorary Doctoral „Dr.h.c.“ of the West Bohemian University of Plzen.