Optimization of tubular heating elements

Victor Almblad

Division of materials engineering Department of Mechanical Engineering Company: Backer AB, Sösdala, Sweden

Abstract

Tubular heating elements are used for intelligent heating applications in different industries. Today, moisture is a major problem, which causes bad electrical insualtion of the heating elements. To prevent this a sealing is a necessity. Today, the sealing process takes place in latter stages of production, increasing the risk of mosiutre penetration. Studying the effect of mositure on electrical insualtion properties of heating elements could improve the understadning of such, which is crucial in order to optimize the production. Investigation and evaluation of new sealing materials is another aspect that can be useful for optimizing tubular heating elements. The studies made in this project were able to provide a better understanding of how mosiutre affect electrical insulation of heating elements while also contributing useful knowledge regarding sealing materials.

Keywords: Heating element, sealing, moisture, MgO, Mg(OH)₂, hydration, electrical conductivity, electrical resistance.

1. Introduction

Tubular heating elements produced are used for intelligent heating in automotive, medical and household industries. When active, the heating element transfers heat from an electric current without the risk of current leakge due to electrical insualtion from magnesium oxide (MgO). MgO is used as an electric insulator due to its low electric conductivity (10⁻¹⁶-10⁻¹⁷ S/m) [1-3]. MgO is hygroscopic and forms $Mg(OH)_2$ in the presence of water [3-7]. $Mg(OH)_2$, also hygroscopic, can bind water along particle surfaces and in grain boundaries. This enables ion exchange between the surface and the interior of the Mg(OH)₂ particles, which increases the electrical conductivity of the $Mg(OH)_2$ [2, 3, 7, 8]. This makes moisture penetration a major problem and necessitates insulation from humid environments. To prevent moisture penetration a sealing is used. Today, the sealing process takes place in late stages of production as several production steps may harm the sealing. This increases the risk of moisture penetration in production and drying operations are necessary to keep the heating elements free from moisture.

Sealing materials used vary depending on the operating environment of the heating element. Many sealing materials require curing, which takes both time and sometimes also the access of heat [9, 10]. Commonly used sealing materials today are silicone, polyurethane (PU) and epoxy. The latter two possess great adhesion to metal while also being impermeable to moisture. Silicone is often used in hot environments where the high temperature can often be a problem for other sealing materials. [10-13]. A problem when working PU and Epoxy is toxicity as both can cause allergic reactions [14, 15].

There is a major interest in improving production and the sealing of tubular heating elements. UV curing and film spraying are methods with potential to improve todays production [14-16], while glass ceramics and different polymers could work as suitable sealing materials [17-20]. This paper reports the study of electrical insulation of tubular heating elements when exposed to humid environments, as well as studies on curable adhesives as potential sealing materials. The main objective is to evaluate how electrical insulation of heating elements is affected by the presence of moisture while also investigating different adhesives as suitable sealing materials.

2. Materials and methods

2.1 Preparation of samples

Samples for testing were tubular heating elements (600x8.5 mm), provided by Backer AB. The heating elements consisted of a resistance wire and a tube (mantle) in stainless steel, which was filled with MgO powder. After filling, the heating elements were rolled, which compacted the MgO powder into a porous MgO solid, which became the electrical insulator of the finished heating element samples. The samples were annealed at 1080 °C in inert atmosphere (no oxygen). This completely removed any moisture from the samples. After annealing, samples were placed inside a dry stock at 25 ° in 30 % relative humidity (6 g H_2O / m^3 air) or inside a climate chamber. If sealed, the samples where bent (U shaped) and sealing material was applied, in both ends, in an amount where it covered the MgO surface. Curing was done by thermal curing or UV-curing. If thermally cured, an insulating pearl was placed above the sealing material to increase electrical creeping distance. A schematic of a heating element end (with insulating pearl) can be seen in Figure 1.



Figure 1: Schematic of a tubular heating element end.

2.2 Adhesives for testing

Different sealing materials were used to investigate different properties such as sealing ability, adhesion to metal and curing time. Three sealing materials were provided by Backer AB, 1 PU and 2 Epoxy. Also three adhesives were procured from APM Technica, 1 Acrylate and 2 Epoxy. The material properties of the adhesives are summarized in **Table 1**.

Table 1: Critical parameters for adhesives selected for this project.

Adhesive	Material	Curing type	Temperature range (°C)
PU 403	Polyurethane	Thermal	-50, +120
Araldite D	Epoxy	Thermal	-50, +120
Ecobond 144A	Epoxy	Thermal	-50, +200
Unocol 818	Acrylate	UV	-40, +110
NOA 81	Epoxy	UV	-40, +110
NEA 121	Epoxy	UV	-40, +110

2.3 Conditions of exposure to moist air and resistivity measurements

Testing was made through measurement of electrical resistance inside a dry stock (25 °, 30 % relative humidity). After annealing, samples were placed inside the dry stock or inside a climate chamber (CC), in both cases no sealing was used. The time and conditions inside the climate chamber can be seen in **Table 2** and after exposure to moist air inside the climate chamber, samples were placed in the dry stock. Electrical resistance was measured using a FLUKE 1507 insulation tester. The measurement started when samples were removed from the climate chamber or immediately after annealing (if placed directly in the dry stock). The time of the experiment was during a 10 week period and electrical resistance was measured on a daily basis.

 Table 2: Conditions of exposure to moist air for the samples of tubular heating elements.

Storage in CC (h)	Temperature (°C)	Humidity (%)	Water content (g / m ³ air)
24		90	18
72	25		
24	70		297

2.4 Testing of curable adhesives

The different adhesives were used to seal samples, followed by exposure to humid air inside the climate chamber. Before sealing, samples were dried inside a furnace at 200 °C for 12 hours. Samples were only sealed if resistance was above a threshold of 10 G Ω , a standard used by Backer AB. When sealed, time and temperature of curing was noted for comparison between the different sealing materials. After being sealed, samples were exposed to humid air inside the climate chamber. Conditions used were the same as for previous testing, see **Table 2**. Resistance was measured before and after every storage inside the climate chamber. If electrical resistance went below 10 G Ω the adhesive would be excluded from future measurements. After measurement of electrical resistance, current leakage and durability at 120 °C was tested. The resistance is decreasing over time for all samples. Immediately after annealing the electrical resistance is very high (3 x $10^7 \text{ M}\Omega$) and when studying samples with no storage inside the climate chamber (Sample 1) it can be seen that resistance is decreasing very rapidly the first few hours. This is followed by a continuous decrease in electrical resistance. All samples stored inside the climate chamber (Sample 2-4) show a low electrical resistance when removed from the climate chamber, see **Figure 2**. When placed in the dry stock samples stored at 25 °C in the climate chamber (Sample 2 & 3) show increasing electrical resistance in early stages of storage followed by decreasing electrical resistance for the rest of the measurement. Samples stored at 70 °C show very little change in electrical resistance, which remains low throughout the entire measurement.

3. Results

3.1 Dependence of electrical insulation on ambient environment

Changes in electrical resistance for samples stored in the dry stock (25 $^{\circ}$, 30 % relative humidity) can be seen in **Figure 2**.



Figure 2: Evolution of electrical resistance of insulation material in annealed tubular heating element samples during exposure to moist air in dry stock (25 °C, 30 % humidity). Sample 1 was stored in the dry stock immediately after annealing and samples 2-4 was stored inside a climate chamber before placed in the dry stock.

3.2 Results from testing of curable adhesives

All curable adhesives were successful in preventing moisture penetration of the heating elements except for the Unocol 818. Due to insufficient curing it was therefore excluded from further testing. Samples sealed with other adhesives show good electrical resistance ($\geq 11G\Omega$), which is above the threshold of 10 G Ω .

The time of curing did vary and the UV-curable adhesives NOA 81 and NEA 121 cured in a matter of seconds once exposed to UV light from a UV light source. Curing of PU 403 took 1 hour at a temperature of 70 °C while the curing of both Araldite D and Ecobond 144A took 24 hours at 120 °C. No samples show signs of current leakage, proving that all adhesives are good in preventing moisture penetration after curing. When placed in a furnace at 120 °C the thermally cured adhesives remain durable and maintain their adhesion to the sample. This is not the case with the UV-curable adhesives, which did not stick to the samples at a temperature of 120 °C.

4. Discussion

The rapid decrease in electrical resistance is due to the presence of moisture inside the heating elements. The high conductivity of water molecules (10⁻³-10⁻⁴ S/m) [2, 7, 21] results in bad electrical insulation and electrical resistance drops at a rapid rate, see Sample 1 in Figure 2. During annealing the high temperature forces hot air out of the heating elements, which causes a vacuum inside the heating element. This vacuum sucks ambient air into the element when leaving the annealing furnace and moisture enters the element at a rapid rate. This explains the large drop in electrical resistance when samples are placed immediately in the dry stock after annealing, see sample 1 in Figure 2. When samples are exposed to humid environments after annealing (inside climate chamber) more moisture is present. This explains why resistance is very low when samples are removed from the climate chamber and placed inside the dry stock, see sample 2-4 in Figure 2. When hydrated, MgO absorbs water [1, 2, 7], which has a drying effect on the heating elements by removing conductive water molecules inside the heating element. This explains the increase in electrical resistance, observed for sample 2 & 3 (see Figure 2).

The hydration of MgO introduces $Mg(OH)_2$ inside the samples. When hydrated $Mg(OH)_2$ binds water along particle surfaces and in grain boundaries. The binding of water to $Mg(OH)_2$ particles is what causes bad electrical insulation [5, 7]. The hydration of $Mg(OH)_2$ has a negative effect on electrical resistance and explains why the resistance decreases over time for all samples. The hydration of MgO and Mg(OH)₂ continues when moisture is present and explains why constant changes in electrical resistance can be observed for all samples, see **Figure 2**. This proves the importance of a sealing and shows the sensitivity towards moisture for tubular heating elements.

Results from testing of curable adhesives show the importance of efficient curing for a sealing material. After curing, the sealing prevents moisture penetration of the heating element. Results show variations in curing time, where the UV curable adhesives demonstrated a significantly shorter time of curing compared to thermally cured adhesives. The Unocol 818 did not prevent moisture penetration and a reason for the insufficient curing can be high viscosity. The viscosities of the UV curable adhesives are summarized in **Table 3**.

Table 3: Viscosities of the UV curable adhesives.

Adhesive		Viscosity (kg m ⁻¹ s ⁻¹)
Unocol 818	3	0,7
NOA 81		0,3
NEA 121		0,3

Higher viscosity can make it more difficult for the adhesive to fill out deficiencies in the mantle, which is critical for obtaining good adhesion. The formation of an interface between the mantle and the sealing makes the sealing stick while also making the heating element more impermeable to moisture [22, 23]. The high viscosity of Unocol 818 may have prevented formation of such an interface, which can be the reason for the bad sealing ability of the adhesive. An issue with the UV curable adhesives is the temperature range, limiting them to be used in environments where operating temperature is low. Applying the insulating pearl is another issue, as it limits the access to UV light during curing. These problems has not been investigated further in this project.

5. Conclusions

The importance of a sealing was evident when studying the evolution of electrical resistance of insulating material in annealed tubular heating elements during exposure to moisture. In presence of moisture the hydration of MgO will initially increase electrical resistance but will over time lead to an increased amount of $Mg(OH)_2$ in the heating element. When hydrated, the binding of water increases electrical conductivity of $Mg(OH)_2$ and the electrical resistance of heating elements decreases. This can lead to current leakage or short circuit and a sealing is necessary in preventing such. Curable sealing materials can efficiently be used to prevent moisture penetration of heating elements. UV curing is a method with promise for reducing the curing time but there are still practical issues that need to be solved before they can be used in large scale production.

Acknowledgements

Acknowledgements goes to Hans Göran Kunkel and Backer AB for providing samples equipment and knowledge in the matter. Acknowledgement also goes to Professor Dmytro Orlov and Professor Srinivasan Iyengar for their support in the project.

References

- 1. Fuji-Ta, K., et al., *Electrical conductivity* measurements of brucite under crustal pressure and temperature conditions. Earth, planets and space, 2007. **59**(6): p. 645-648.
- 2. Gasc, J., et al., *Electrical conductivity of polycrystalline Mg (OH) 2 at 2 GPa: effect of grain boundary hydration–dehydration.* Physics and Chemistry of Minerals, 2011. **38**(7): p. 543-556.
- Wu, X., et al., *Electrical conductivity measurements* of periclase under high pressure and high temperature. Physica B: Condensed Matter, 2010. 405(1): p. 53-56.
- 4. Hansen, K.K., et al. Magnesium-oxide boards cause moisture damage inside facades in new Danish buildings. in International RILEM Conference on Materials, Systems and Structures in Civil Engineering. 2016. Rilem publications.
- 5. Kuenzel, C., et al., *The mechanism of hydration of MgO-hydromagnesite blends*. Cement and Concrete Research, 2018. **103**: p. 123-129.
- Hermann, A. and M. Mookherjee, *High-pressure* phase of brucite stable at Earth's mantle transition zone and lower mantle conditions. Proceedings of the National Academy of Sciences, 2016. 113(49): p. 13971-13976.
- Gieseke, W. and F. Freund, The influence of molecular hydrogen on the conductivity of magnesium oxide containing paramagnetic surface defects. Journal of Physics and Chemistry of Solids, 1977. 38(2): p. 183-186.
- 8. Karato, S.-i. and D. Wang, *Electrical conductivity of minerals and rocks*. Physics and Chemistry of the Deep Earth, 2013: p. 145-182.
- Saenz-Dominguez, I., et al., *Effect of ultraviolet* curing kinetics on the mechanical properties of out of die pultruded vinyl ester composites. Composites Part A: Applied Science and Manufacturing, 2018. 109: p. 280-289.
- 10. Zhou, J., et al., *Fast curing of thick components of epoxy via modified UV-triggered frontal polymerization propagating horizontally.* Materials Letters, 2016. **176**: p. 228-231.
- 11. Liu, F., et al., *Green fabrication of ultraviolet curable epoxy acrylate-silica hybrid coatings.* Progress in Organic Coatings, 2017. **109**: p. 38-44.

- Montarnal, D., et al., Silica-like malleable materials from permanent organic networks. Science, 2011. 334(6058): p. 965-968.
- 13. Brachaczek, W., The modelling technology of protective silicone coatings in terms of selected physical properties: Hydrophobicity, scrub resistance and water vapour diffusion. Progress in Organic Coatings, 2014. **77**(4): p. 859-867.
- 14. Chattopadhyay, D., S.S. Panda, and K. Raju, *Thermal and mechanical properties of epoxy acrylate/methacrylates UV cured coatings*. Progress in Organic Coatings, 2005. **54**(1): p. 10-19.
- 15. Firdous, H. and M. Bajpai, UV curable heat resistant epoxy acrylate coatings. 2010.
- 16. Masson, F., et al., *UV-Radiation curing of waterbased urethane–acrylate coatings*. Progress in Organic Coatings, 2000. **39**(2-4): p. 115-126.
- Borhan, A., et al., Influence of (CoO, CaO, B2O3) additives on thermal and dielectric properties of BaO–Al2O3–SiO2 glass–ceramic sealant for OTM applications. Ceramics International, 2016. 42(8): p. 10459-10468.
- Donald, I., et al., Recent developments in the preparation, characterization and applications of glass-and glass-ceramic-to-metal seals and coatings. Journal of Materials Science, 2011. 46(7): p. 1975-2000.
- Jaiswal, P., et al., Comparative evaluation of sealing ability of light cure glass ionomer cement and light cure composite as coronal sealing material: An in vitro study. Journal of the International Clinical Dental Research Organization, 2017. 9(1): p. 12.
- 20. Fridrichovsky, M., F. Steiner, and M. Hirman. Comparison of the characteristics of PCB protective coatings. in Electronics Technology (ISSE), 2017 40th International Spring Seminar on. 2017. IEEE.
- 21. Santos Jr, T., et al., Mg (OH) 2 Nucleation and Growth Parameters Applicable for the Development of MgO-Based Refractory Castables. Journal of the American Ceramic Society, 2016. **99**(2): p. 461-469.
- People, E.T.S. Conformal Coatings Vs Potting Compounds Which is better to protect my PCB, a coating or a resin? 2018; Available from: https://www.electrolube.com/technicalarticles/conformal-coatings-vs-potting-compoundswhich-is-better-to-protect-my-pcb-a-coating-or-aresin/.
 people, E.T.s. Thermal Management Solutions For
 - 5. people, E.1.s. *Thermal Management Solutions For Long Term Protection Of Military And Aerospace Electronics.* 2018; Available from: <u>https://www.electrolube.com/technical-</u> <u>articles/thermal-management-solutions-for-long-</u> <u>term-protection-of-military-and-aerospace-</u> <u>electronics/</u>.