# *Optimisation of the Ceramic Phase for Ceramizable Silicone Rubber Based Composites*

Z Pędzich<sup>1</sup>, D M Bieliński<sup>2,3</sup>, J Dul<sup>2</sup>, M Zarzecka-Napierała<sup>1</sup>

 <sup>1</sup> - AGH-University of Science and Technology, Faculty of Materials Science, Advanced Ceramics Department, 30 Mickiewicza Av., 30-059 Kraków, Poland
<sup>2</sup> - Institute for Polymers & Dyes Technology, Division of Elastomers & Rubber Technology, 30 Harcerska St., 05-820 Piastów, Poland
<sup>3</sup> Technical University of Lódé Institute of Polymers, 12/18 Stafeneverlines St.

<sup>3</sup> - , Technical University of Łódź, Institute of Polymers, 12/18 Stefanowskiego St., 90-924 Łódź, Poland

E-mail: pedzich@agh.edu.pl

**Abstract.** The paper presents results of investigation on composition of mineral components used for ceramizable silicone rubber-basing composites. Using different crystalline phase additives it is possible to assure the proper course of the composite degradation and its transformation during ceramization process. Three crystalline components were tested as an additive to silicone rubber – bentonite, kaolinite and wollastonite. They were added simultaneously with a glassy phase. The process of silicone part of composites degradation during heating up to  $1050^{\circ}$ C were examined by DSC/TG technique. Characteristic of ceramized composite body created during heating was investigated by mercury porosimetry. The porosity evolution was described in the range of  $600 - 1050^{\circ}$ C.

#### 1. Introduction

Flame resistance of electrical cable covering insulation is very important from the safety point of view of people using means of transport, visiting shopping centres, sport halls, theatres or cinemas. Metal rods in protective covering make the main part of electrical circuits, responsible for distribution of electricity in cars and planes, providing power supply for elevators or fire-fighting systems. Material used nowadays, based on silicone rubber or PVC is incombustible, but the problem concerns its mechanical durability under flame, maintaining circuit integrity and energy supply during fire, indispensable to secure time for evacuation or emergency landing. Integrity of electrical cables can be obtained by application of covering insulation made of polymer composites ceramizing at high temperature arising during fire [1]. The paper presents results of investigation on optimization of ceramic phase using different composition of the mineral part of composites. As the main crystalline component bentonite, kaolinite or wollastonite were used. Investigation of composite transformations (polimer degradation and material ceramization) during heating up to 1050°C showed the influence of a type of crystalline phase on the composite microstructure after ceramization at the whole interesting temperature range ( $600 - 1050^{\circ}$ C).

### 2. Experimental

Materials used for investigation were silicone rubber-basing composites. They were composed of silicon rubber containing silanized Silbond binder (45 phr) and the mixture of glassy phase, borax

and as the crystalline component one of three different crystalline phases – bentonite, kaolinite or wollastonite (100 or 200 phr). Glassy phase, borax and the crystalline component ratio was (3:2:7). Additives necessary for the proper crosslinking of the silicone base were also added.

Vulcanized composites were examined by DSC/TG (Differential Scanning Calorymetry/ Thermogravimetry) up to 1050°C (Netzsch STA449F3 Jupiter).

Samples were also fired using controlled temperatures of 600 and 1050°C. Microstructure of ceramized samples was investigated using mercury porosimetry method (Poremaster 60 Quantachrome).

#### 3. Results

Figures 1 and 2 shows that despite of the kind of crystalline phase and its content the process of degradation of silicone proceeded at comparable range of temperatures in each composite type. The DSC analyses of degradation in composites containing the lower amount of "ceramic mixture" (glass-borax-crystalline component) – 100 phr, showed many steps of this process, which could be distinctly separated. It is commonly known, typical behaviour [2, 3]. The way of silicone degradation in composites containing the higher amount of "ceramic mixture" process was different. The identification of separate degradation mechanisms temperature ranges was rather difficult.





**Figure 1.** DSC curves of composites containing 100phr of crystalline phase.

**Figure 2.** DSC curves of composites containing 200phr of crystalline phase.

Thermogravimetric measurements showed that there were essential differences in weight losses for composites containing 100 phr of "ceramic mixture". The difference between composite containing wollastonite and composite containing bentonite was about 8%. The weight loss for kaolinite containing composite was on intermediate level.

The composites containing 200 phr of "ceramic mixture" showed lower weight losses. It is worth to notice that these weight losses were much more uniform for all composites (the difference not exceeded 2%) than observed at lower temperature.



**Figure 3.** TG curves of composites containing 100phr of the main ceramic phase.

**Figure 4.** TG curves of composites containing 200phr of the main ceramic phase.

The pore size distributions of materials subjected to high temperature ("ceramized ") were collected in the figures 3 and 4. Figure 3 compares pore size distribution for composites containing 100 phr of "ceramic mixture". After firing at 600°C porosity of each composite is rather high - the highest in bentonite containing material, the lowest in wollastonite containing one. Firing at 1050°C decreased porosity in all materials, but this effect was observed for bentonite and kaolinite containing materials mainly. Composite with wollastonite showed relatively stable porosity, however the structure of porosity was changed, the smallest pores were eliminated.



Figure 5. Cumulative curves of pore size distribution for composites containing 100phr of crystalline phase ceramized at 600 and 1050°C.



**Figure 6.** Cumulative curves of pore size distribution for composites containing 200phr of crystalline phase ceramized at 600 and 1050°C.

Figure 4 compares pore size distribution for composites containing 200 phr of "ceramic mixture". Generally, these composites showed less porosity than described above materials containing 100 phr of "ceramic mixture". It is worth to notice that after firing at 1050°C porosity in composites containing bentonite and kaolinite practically disappeared due to huge amount of glassy phase created while bentonite and kaolinite transformed during heating.

# 4. Summary

Performed investigations allowed us to evaluate the differences in microstructure of silicone rubberbasing composites containing the additive of different crystalline phase.

It was established that composite with wollastonite additive showed the most stable properties. Its weight losses during firing were the lowest, the porous structure which was created during firing changed slowly and small amount of porosity is present up to the highest temperatures of firing. It should be profitable because of potential increase of fracture resistance of ceramized composite and should decrease its thermal conductivity.

## Acknowledgments

Authors are greatly indebt to Dr. M. Szumera and Dr G. Grabowski from Department of Advanced Ceramics of AGH for their thermogravimetrical analyses.

We would also to express our thanks to M.Sc. R. Lach for assistance in porosimetry measurements. The work was financially supported by the European Union within a framework of National Coherence Strategy under Innovative Economy Operating Programme; grant no. POIG.01.03.01-00-067/08-00.

#### References

- [1] Processable silicone composite materials having high temperature resistance. US Patent 5552466 (03.08.1996)
- [2] Hamdani S, Longuet C, Perrin D, Lopez-Cuesta J-M, Ganachaud F, *Polymer Degradation and Stability* **94** 465
- [3] Hayashida K, Tsuge S, Othani H, Polymer 44 5611