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Degradation of silicone-based sealing materials used in microelectronics

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Abstract

1. Introduction

Adhesive bonding is a key joining technology in many industrial applications, including automotive, aerospace industries, biomedical devices, and microelectronic components. Adhesive bonding is gaining more and more attention due to the increasing demand for joining similar or dissimilar components, mostly within the framework of designing lightweight structures. Silicone sealant is widely used in engineering application due to its thermal stability, excellent energy absorption, and good damping characteristics. In those applications, sealant usually exposed to various environment stress, such as, high temperature, mechanical stress, humidity, light radiation, and chemical attack. Long-term stability and durability of sealant is crucial to the performance of the associated application. The main degrading factors for silicone in microelectronic applications are temperature, humidity, alkali, and mechanical loading. The focus in the present paper is to understand different failure mechanisms in silicone sealants and adhesives and to study how different environmental, mechanical, and service-related stresses attribute to the kinetics and extent of degradation in silicone sealants and adhesives. The impact of different failure mechanisms on the lifetime and reliability of microelectronic devices will be methodically investigated.

2. Materials and Method

The sealant was synthesized based on the instruction with mixing 50 g part A with 50 g part B of sealant. After having a homogenous sealant mixture, the sealant was put in the oven at 115 °C for 15 min. Aluminium was used as the substrate, given that it is commonly used in different applications such as gaskets and PCBs. Aluminium plates were polished and washed with acetone in an ultrasonic set-up for 30 min, before applying the sealant layer. Sealants with thickness of 0.25 mm (Fig.1a) were applied mechanically to make sure that the thickness is even and homogeneous.

2.1.2 Nanoparticle-Containing Samples

To improve the mechanical properties of sealants, different Nanoparticles such as TiO2 P25 and Al_2O_3 at different weight percentages of 1, 3, and 5 % were added mechanically to the mixture.

Results showed that nanoparticles TiO2 P25 and 5% Al_2O_3 have negative impact on the sealant as they adversely influence the flowability/viscosity of the composite mixture (Figure 1).

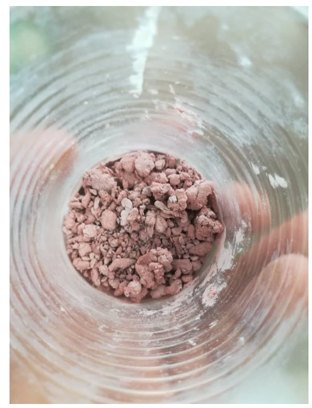

Figure 1: Agglomerated sealant with 5% Al2O3

3 Ageing tests

3.1. Thermal ageing

The effects of exposing sealants to high temperatures were studied at 150 and 175 °C. After curing at room temperature, one set of samples were exposed to a steady high temperature of 150 °C for 1 week. The samples were then subjected to tensile and shear tests at room temperature to determine the changes in ductility, strength, stiffness, and bonding characteristics of the sealant.

3.2. Alkali ageing

A set of samples were aged at 40 °C in a salt spray test setup with 100% of humidity in a salty atmosphere. The salt spray set-up simulates the harsh environment condition with aggressive ionic contaminations. In such set-up both temperature and oxidizing ionic species are employed to accelerate the ageing kinetics. Samples were aged for five days for all four testing conditions described above. Once the experiment was finished, the mechanical and chemical properties of aged samples were studied.

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3. Mechanical testing

3.1 Tensile test

The tensile tests which were conducted with the universal tensile testing machine to determine the strength of the sealant.

3.2 Shear test

To determine the shear strength of the joint, samples of 10 ×10 mm were cut and placed in a fixture, as schematically shown in Figure 2. The lap shear test is surely the most used experimental procedure to characterize adhesive behaviours, due to its simplicity. It is an interesting method, when it comes to comparative results, where it is possible to verify among many materials, which can offer the highest strength under shear loading. A failure mechanism can also be detected through single lap shear tests, which allows for determining the failure mode: whether it is adhesive or substrate failure. Figure 3 shows the shear test fixture and samples, used for this test.

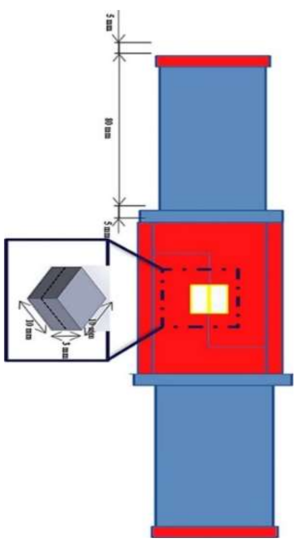

Figure 2: Schematics of the holder for the shear test.

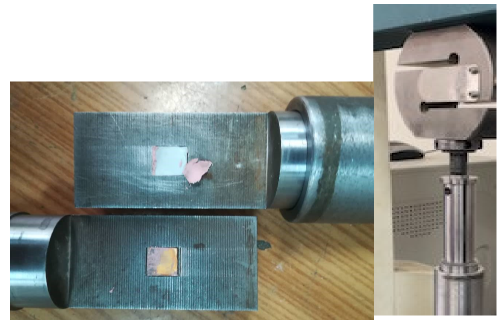

Figure 3: a) the fixture and b) set up, used for shear tests.

2.4 Characterizations

Scanning electron microscope (SEM) was used to do the fractography on the fracture surface. Surface morphology of the aged samples were analysed with scanning electron microscopy (SEM) and stereomicroscope. Infrared spectra were recorded using a Perkin–Elmer Spectrum 100 series spectrometer in the attenuated total reflection (ATR) mode for 200 scans at a resolution of 4 cm−1.

3 Results and discussion

3.1 Mechanical result

The mechanical properties were firstly measured because tensile strength and displacement at breaking point are the most immediate evidences to evaluate the ageing effects [1-5]. Thermal ageing revealed obvious decrease in tensile strength. Figure 4 shows the tensile behaviour of thermally-aged Al specimens at 175 °C. All ageing conditions have the same trend as 175 °C, Increasing the ageing time is always associated with a decrease in the maximum force.

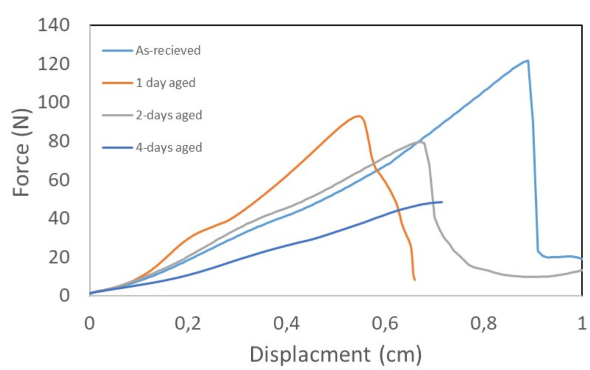

Figure 4: Tensile strength of different conditions at 175°C

Figure 5 depicts the tensile strength of samples at different ageing conditions. It is shown that by increasing the temperature, tensile strength decreases. Interestingly, even though the temperature during the salt spray test is not so high (40 °C), the salt spraying is associated with a dramatic decrease of maximum tensile force right from the early stage of ageing. This infers that ionic contaminations can dramatically deteriorate mechanical properties of sealants and that their negative influences are much more than thermal stresses. On the other hand, it is shown that tensile strength of samples with 1% Al2O3 increased, but 3% Al2O3 mixture has almost same behaviour of the 175 °C for pure sealant, showing that 3% Al2O3 does not improve the mechanical properties of the sealant.

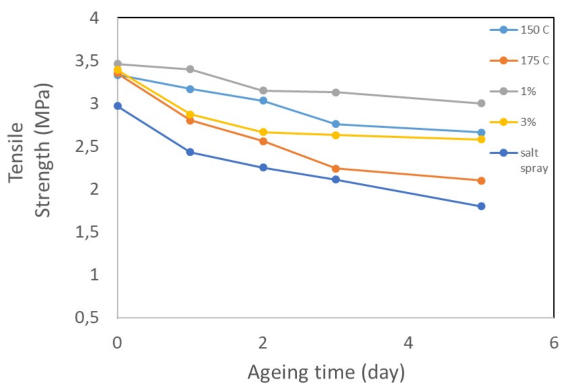

Figure 5: Tensile Strength of Different conditions

Figures 6 exhibits shear tests, aged at 150. The same arguments as tensile tests can be applied from shear strength graph (Figure 9): with increasing the ageing time, the maximum shear stress in most cases decreases, the effects of salt spraying on the shear tests of specimens, again confirming how deleterious ionic contaminations are, as compared with thermal stresses , Figure 7. The same as Tensile strength 1% Al2O3 mixture have best strength among all the conditions, and alkali environment has shown the decreasing trend of ageing.

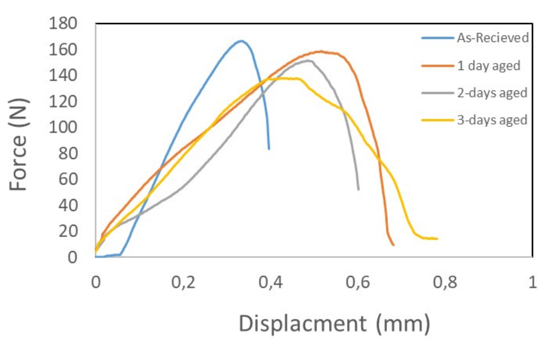

Figure 6: Shear Force of Different ageing time at 150 C

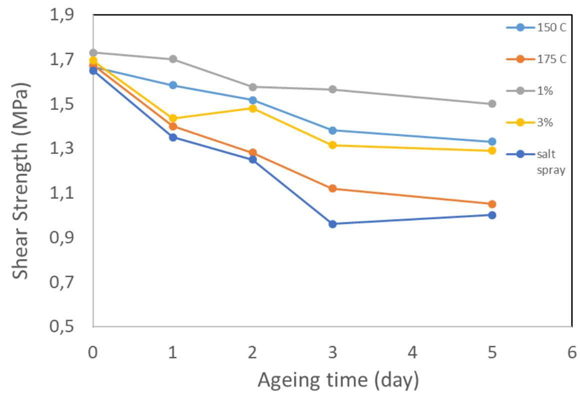

Figure 7: Shear Strength of Different conditions

3.2 FTIR

To get a better understanding of the ageing mechanism and more reasonable evaluation, the FTIR analysis was also implemented. Fig.8 gives the FTIR spectra of silicone sealant under different ageing conditions.

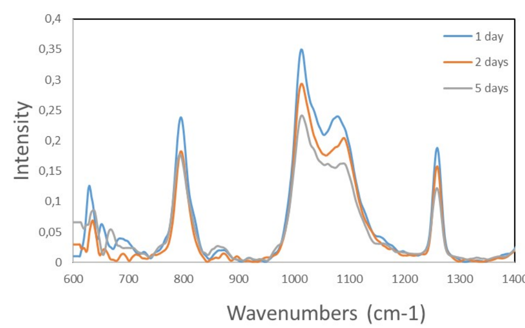

Figure 8: FTIR of sample at 175 C

The five characteristic peaks for silicone rubber are observed at 2964, 1256, 1015–1085, and 795 cm−1, which correspond to the vibrations of methyl (–CH3), Si–CH3, Si–O–Si, and Si–(CH3)2 bonds, respectively [6-7]. 1015–1085 vibrations of Si–O–Si, and There is reduction changes in the FTIR spectra of aged samples as compared with that as-received sample.

The 1256 cm−1 wavelength, indicates the presence of silicon organic compounds with a methyl group. Around 1260 cm−1, methyl groups attached to silicon atoms produced a distinctive deformation absorption [37]. As the material was thermal-aged, a certain decrease in the intensity at this wavelength was found, which indicates that polymer breakages and/or oxidation of C–H bond and Si–C bond, which could promote the oxidation reactions accompanying by the formation of free radicals and oxidation products. The breakage of Si–C or/and oxidation of methyl group in Si–CH3 are further identified by the significant decrease in the intensity of the absorption peak at 785 cm−1. The peak at the 2962 cm−1 wavelength was analyzed, and it shows that the CH3 and CH2 bonds decrease with thermal aging, which is a clear indication of polymer chain scission.

The obvious decreases in the intensity of the absorption peaks at 1085 cm−1 and 1015 cm−1 are mostly due to the bond breakage of the Si–O–Si main chain. There is no absorption peak at 1408 cm−1 which indicates an integral part of rubber crosslinking network. In addition, there is no indication of the intensity of the absorption peak at 1729 cm−1 shows non-monotonic variations with the increasing temperature. Furthermore, there is no peak at 3200 cm−1 - 3400 cm−1 attributed to the O–H symmetric stretching vibration was observed, and slightly strengthened with the increasing aging time and temperature, which implies the formation of hydroxyl groups. Figure 9 illustrates the intensity peak at 1600 cm-1 which is showing the oxidation of the aged sample. The same an tensile strength the higher the temperature the more oxidation of the sample. The oxidation of the composite sample with 1% Al2O3 is less than other samples which is in an agreement with mechanical properties.1% Al2O3 enhanced the chemical and mechanical properties of the sealant.

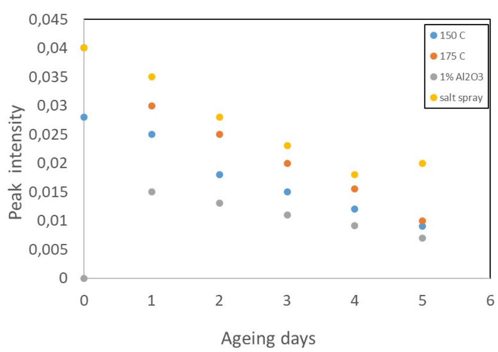

Figure 9 Intensity at 1600 Cm-1 for all ageing condition

4- conclusions:

The design of the experiment was used to investigate the effects of three ageing factors and investigate their interactions on the physical properties of silicone rubber used in a particular industrial process. There is a detectable change in the FTIR spectra of aged samples as compared with that as-received sample. In all accelerated testing conditions, peak intensities decreases with ageing time. It is an indication that the surface characteristics of the sealant is affected by thermal ageing and oxidized. In the shear and tensile test it is shown that with increasing the ageing time, the maximum strength stress in all cases decreases at all ageing temperatures. One of the observations in the mechanical test is that shear and tensile strength of 1% Al2O3 mixture have best strength among all the conditions, and alkali environment has shown the decreasing trend of ageing. Results showed that nanoparticles with 3% Al2O3 have negative impact on the sealant as they adversely influence the flowability/viscosity of the composite mixture. Salt spraying on the shear tests of specimens, shows how deleterious ionic contaminations affects the mechanical properties of the sealant.

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References

1. Goudie, J.; Owen, M.; Orbeck, T. A review of possible degradation mechanisms of silicone elastomers in high voltage insulation applications. In Proceedings of the 1998 Annual Report Conference on Electrical Insulation and Dielectric Phenomena, Atlanta, GA, USA, 25–28 October 1998.
2. Kashi, S.; Varley, R.; De Souza, M.; Al-Assafi, S.; Di Pietro, A.; De Lavigne, C.; Fox, B. Mechanical, Thermal, and Morphological Behavior of Silicone Rubber during Accelerated Aging. *Polym.-Plast. Technol. Eng.* **2018**, *57*, 1687–1696. [CrossRef]
3. Characteristic Properties of Silicone Rubber Compounds. 2012. Available online: https://www.shinetsusiliconeglobal.com/catalog/pdf/rubber_e.pdf (accessed on 25 February 2018).
4. Mark, J.E. Some Interesting Things about Polysiloxanes. *Acc. Chem. Res.* **2004**, *37*, 946–953. [CrossRef] [PubMed]
5. Mark, J.E.; Lin, G.; Schaefer, D.W. *The Polysiloxanes*; Oxford University Press: New York, NY, USA, 2015.
6. Andriot, M.; Chao, S.H.; Colas, A.R.; Cray, S.E.; DeBuyl, F.; DeGroot, J.V.; Dupont, A.; Easton, T.; Garaud, J.L.; Gerlach, E.; et al. Silicones in industrial applications. *Inorg. Polym.* **2007**, 61–161.
7. Jerschow, P. *Silicone Elastomers*; Rapra Technology Limited: Wolverhampton, UK, 2001.