



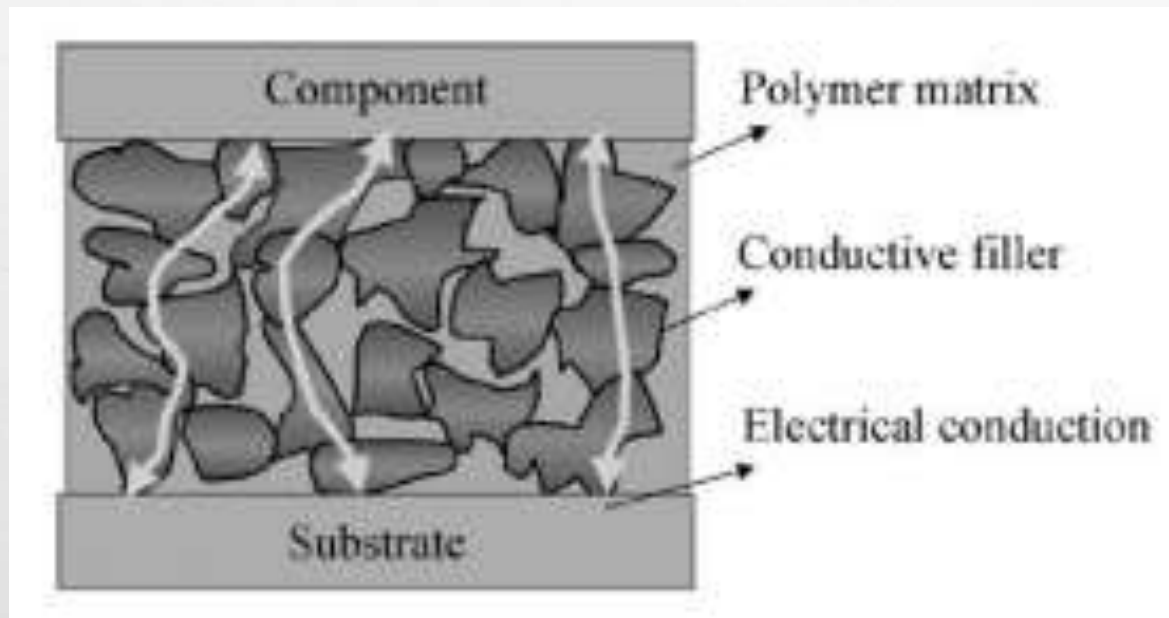
ELECTRICAL AND MECHANICAL PROPERTIES OF SILICONE ELECTRICAL CONDUCTIVE ADHESIVES (ECAS) FILLED CARBON BLACK AT ELEVATED TEMPERATURE

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PROBLEM STATEMENTS

- In electrical and electronic field, soldering process is critical due to its high temperature, difficult application and the usage of lead which is hazardous.
- Hence, ECA is introduced to compensate the setback of soldering using silicone.
- However, silicone has limitations that hinder the function of ECA.
- Therefore, Carbon black (CB) treated with APTES was utilized to improve interaction of the constituents materials and subsequently enhanced the conductivity of ECA.

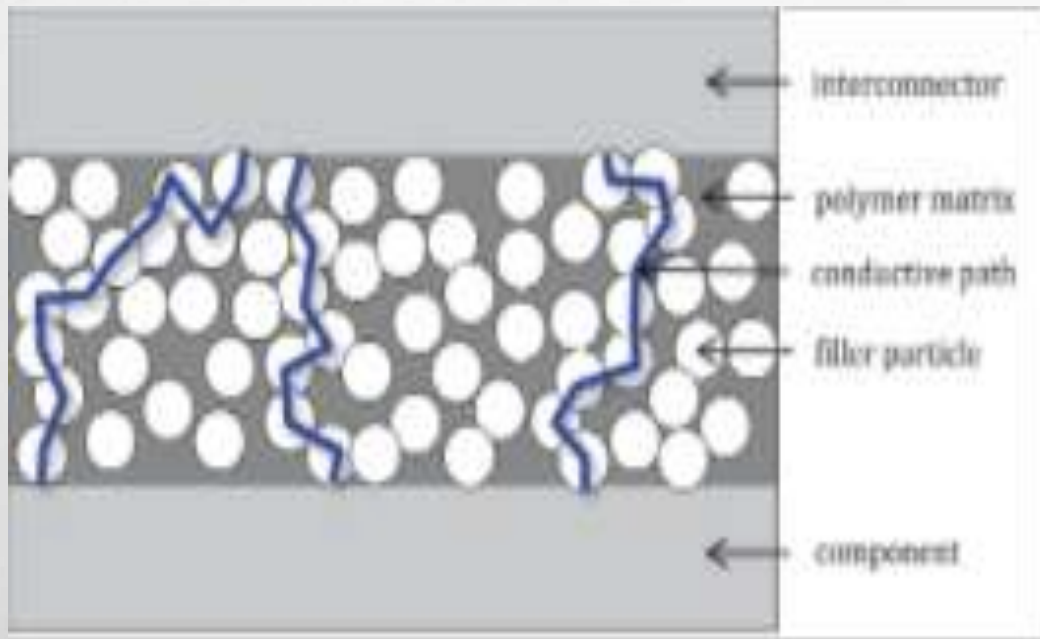
RESEARCH BACKGROUND



Saipeng Li, Xinyue He, Jian Hao, Jian Zhou*, Feng Xue 2017

- Electrical conductive adhesive (ECA) is glue that is primarily used for electronic application.
- ECA advantages as compared to soldering technology such as environmentally friendly, lower processing temperature, less steps in processing that reduce the processing cost and increase the capacity of fine performance due to small sized conductive filler.

RESEARCH BACKGROUND



- ECAs based on silicone has great potential in rapid development of flexible and stretchable electronic because of its unique elasticity combination, moisture resistance, thermal stability and flexibility.
- Although, silicone have excellent mechanical characteristics, the electrical efficiency of silicone-based ECAs are limited thus prevents their widespread usage in electronic industry.
- Therefore, incorporation of conductive filler Carbon black (CB) is introduced as an alternative.

RESEARCH BACKGROUND

- In order to improve the electrical conductivity of CB, surface treatments was performed using 3-Aminopropytriethoxysilane (APTES).
- Surface modifiers containing functional groups of amine can improve the electrical conductivity.
- Increasing the surface properties will increase the filler and polymer affinity to achieving a well-integrated structure.
- CB surface is altered by addition of amide functional groups to improve the electrical conductivity, wetting property and filler dispersion.

OBJECTIVES

To prepare and characterize silicone based ECAs filled with various loading of CB untreated and treated with 3-aminopropyltriethoxysilane on its electrical and mechanical properties at elevated temperature

To determine the effect of 3-aminopropyltriethoxysilane treatment of various CB loadings in silicone based ECAs on its electrical and mechanical properties at elevated temperature

METHODOLOGY

Surface treatment
of CB with APTES

0.25g of APTES solution was added into (0%, 5%, 10% and 15%) of CB with 90ml of distilled water and stir by using mechanical stirrer at 350rpm for 10min at room temperature and cured at 100°C in oven for 24hour

Preparation of
silicone filled CB
ECAs

- Different CB loading (0%, 5%, 10% and 15%) was added to 6.24 g of silicone in a beaker and were stirred by using mechanical stirrer at 350 rpm at room temperature for 10 min. The solution was cast on glass slide mold (75 mm x 26 mm), then cured for 1hour at 100°C

Testing

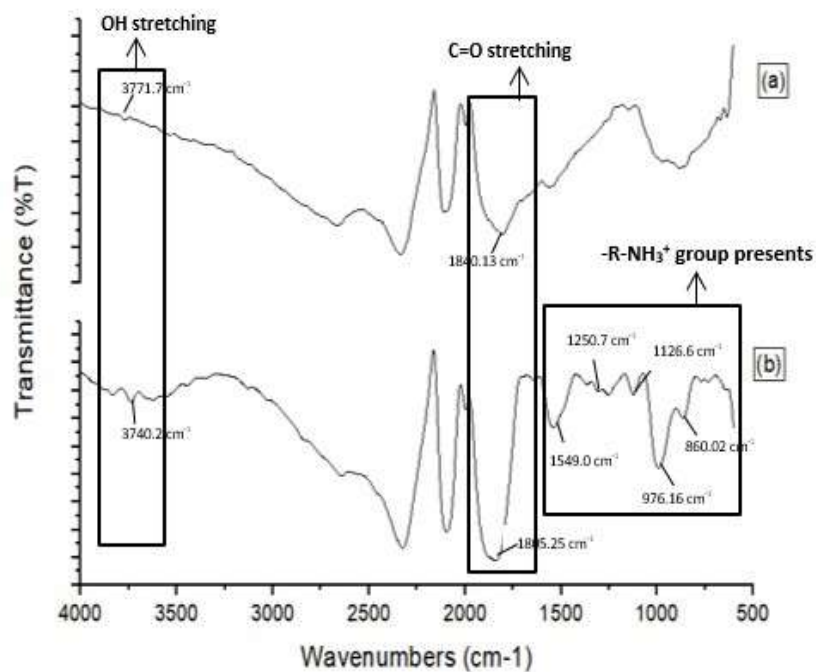
FTIR – film was observed at wavenumber transmission of 4000 cm^{-1} to 500 cm^{-1}

Hardness test – using hand-held durometer

Tensile test – using Instron Universal testing machine

EIS – using Hioki 3532-50 LCR Hitester

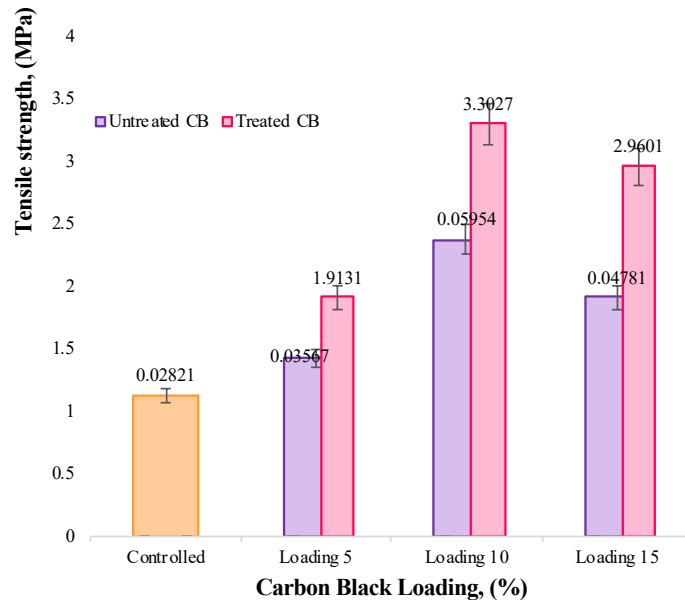
FTIR



- Figure 1 shows the presence of peak at 3771.7 cm⁻¹ and 1805.25 cm⁻¹ attributed to the presence of hydroxyl groups (-OH) and (C=O) respectively, on the surface of the untreated CB.
- Amide functional groups that appeared on the treated CB surfaces. Hence, the new bands appeared at 1549 cm⁻¹, 1250.7 cm⁻¹, 1126.6 cm⁻¹, 976.16 cm⁻¹ and 860.02 cm⁻¹ corresponding to the N-H, SiO-H, Si-O-Si, C-N and C=C stretching vibrations of the amino groups (-R-NH₃⁺), respectively. These spectrums indicate the reaction of 3-aminopropyltriethoxysilane on the surface of CB.



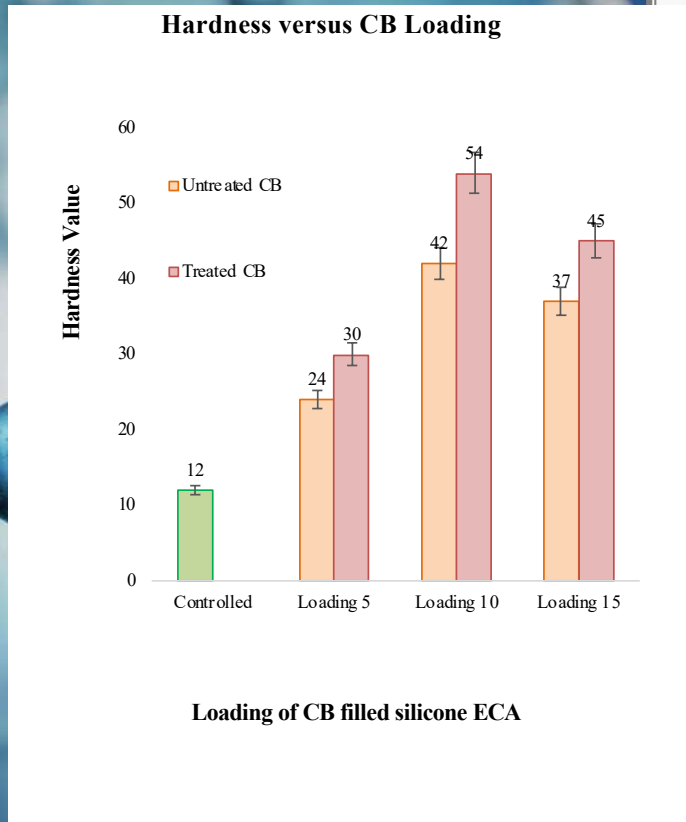
Tensile strength versus CB Loading



TENSILE

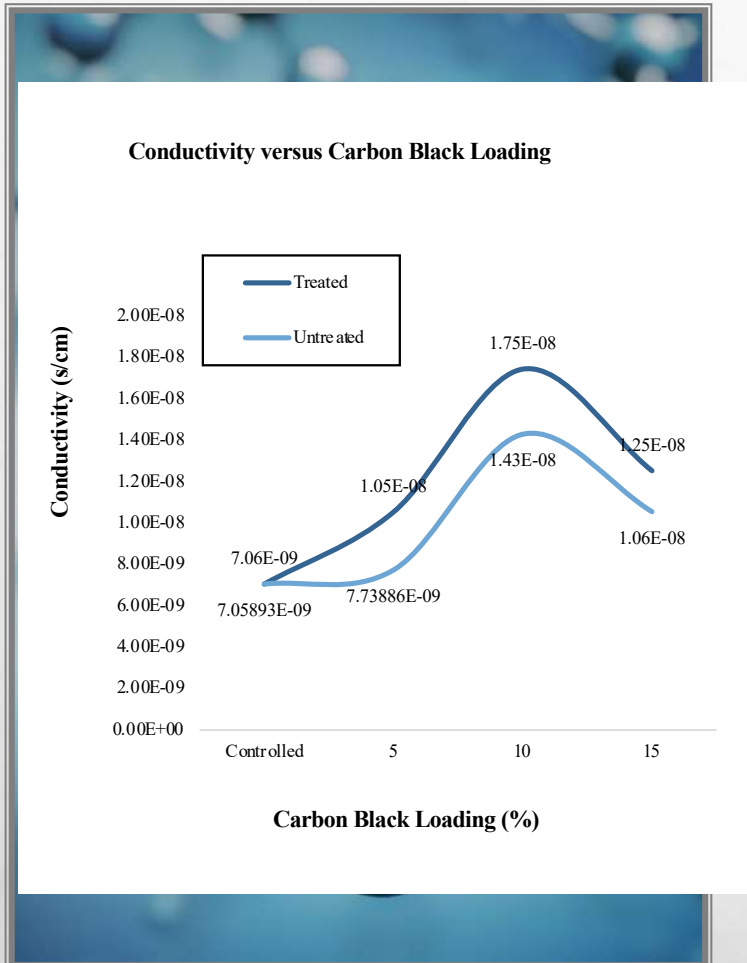
- Untreated samples display increment in tensile strength with increasing in the CB content.
- The modification of CB with 3-aminopropyltriethoxysilane increment in tensile strength up to 80% as compared to untreated CB due to the present of the amide functional groups to the CB surface making more interaction can be formed.
- Increment of TS with increasing CB content until 10% for both untreated and treated carbon black at 0.05954 MPa and 3.3027 MPa respectively.
- In addition, as similar reported by Siti *et al.* (2014) increasing of carbon black loading increased the surface interaction of carbon black, which was decreased the chain mobility of silicone filled treated carbon black.
- At 15% CB loading for treated/untreated, it is found that its presence hindered the mobility of macromolecule chain segments in silicone blends at 0.04781 MPa and 2.9601 MPa for untreated and treated CB respectively.

HARDNESS



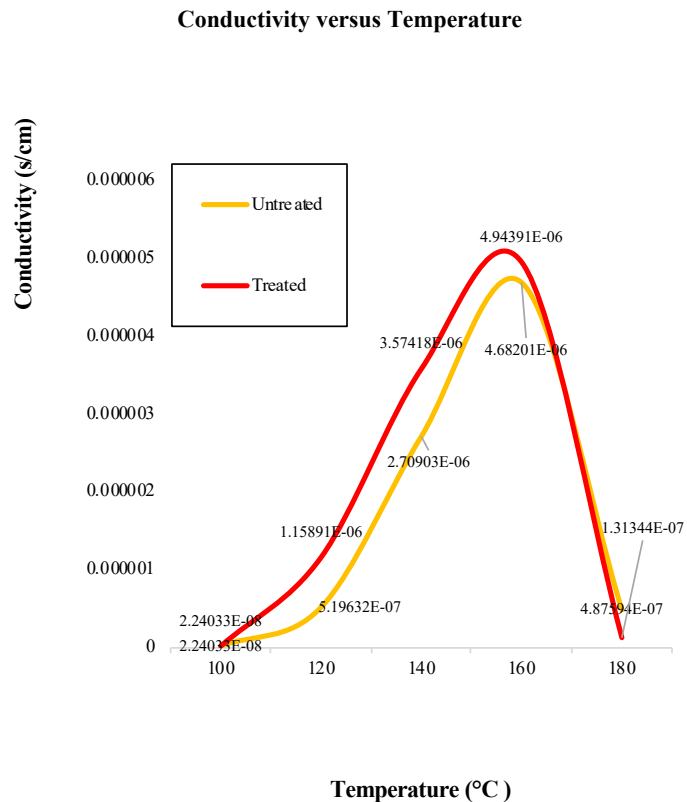
- There is an increment in hardness of silicone ECAs with increasing of CB loadings. Similar finding reported that as expected hardness increases continuously with increasing filler content [9].
- The treated CB filled ECAs display greater in hardness in comparison with untreated CB at 52 and 42 respectively. However, at 15% of CB loading, the hardness value decreased because of poor surface interaction of CB, which decreased the chain mobility of silicone filled treated carbon black [10].

CONDUCTIVITY



- The higher the CB loading, the higher the conductivity of the silicone film and the optimum found at 10% carbon black loading where $1.75\text{E}-08$ s/cm while the resistivity of the silicone film was decreased at 10% carbon black loading of $3.78\text{E}-04$ s/cm.
- The conductivity of the treated CB at $1.75\text{E}-08$ s/cm shows the greater values when compared to the untreated CB at $1.43\text{E}-08$ s/cm due to presence of APTES as surface treatments. This was supported by Li et al., al that the higher structure of APTES having high surface area, easy formation of conductive networks at lower loading of CB and exhibit a lower resistivity [11].
- However, after the optimum CB loading, the conductivity of the film decreased due to the interparticle distance are smaller hence increased the resistivity.

CONDUCTIVITY



- The optimum temperature obtained at 160 °C regardless with or without the treatment. According to Li et al., it is believed that with increasing the temperature, the interparticle average distance increase due to the difference in the thermal expansion of silicone and CB [11].
- The interparticle of untreated CB which are van der Waals bonding have the lower bonding when compared to interparticle of treated of carbon black. The higher the interparticle average distance, the higher the gap of silicone and carbon black therefore, the thermal expansion is larger which means that the silicone film can conduct the electrical at optimum value of 160 °C.
- However, after the optimum temperature, at 180 °C, the conductivity of the silicone film decreased due to the thermal expansion are smaller, hence reduce the conductivity.

CONCLUSION

- As conclusion, the modification of CB with APTES has been successfully improved the mechanical and electrical properties of the silicone cas.
- The FTIR spectrums show the appearance of amino groups on the surface of the CB. The intensity of N-H, SiO-H, Si-O-Si, C-N and C=C peaks also shifted upwards as the loading of treated CB increased.
- Whilst the incorporation of CB treated and untreated up to 10% increase the electrical conductivity of the film but further increment produces low electrical conductivity. The highest conductivity was achieved when carbon black loading at 10% with $1.75E-08$ s/cm but when carbon black loading at 15%, the conductivity decreased at $1.25E-08$ s/cm. This is due to increasing of CB loading in silicone, it will increase the conductivity of the film. Furthermore, when the surface treatment of carbon black was done, the conductivity of the film increased then the untreated CB due to amide functional groups that presents on the surface of treated CB.
- The same trend is obtained in mechanical properties such as tensile and hardness at 10% CB loading regardless treated on untreated at. This is due to the larger surface interaction of carbon black, which decreased the chain mobility of silicone filled treated CB.

FUTURE WORK

**Comparison
study on different
conductive filler.**

**Self healing
properties of ECA**

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THANK YOU