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**Physical and adhesive properties of some materials made by 'Click' chemistry**

Nicolas Le Baut  
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# **Physical and Adhesive Properties of Some Materials Made by “Click” Chemistry**

A thesis submitted in fulfillment of the requirements for the award of the degree

**Honours Master of Engineering by Research**

From

**UNIVERSITY OF WOLLONGONG**

By

Nicolas Le Baut, BEng (Mat)

Materials Engineering Discipline

2005

## **Certification**

I, Nicolas Le Baut, declare that this thesis, submitted in fulfillment of the requirements for the award of Honours Master of Engineering by Research, in the Materials Engineering Discipline, University of Wollongong, is wholly my own work unless otherwise referenced or acknowledged. The document has not been submitted for qualifications at any other academic institution.

Nicolas Le Baut

May 2005.

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## **Abstract**

The aim of this work is to examine the physical and adhesive properties of a number of crosslinked polymers made by “click” chemistry, a technique, that has been explored thus far only in the context of drug discovery. The polymers were synthesised between copper and brass plates. Differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) were used to measure the glass transition temperature ( $T_g$ ) of these materials. The polymers were found to have unusually high  $T_g$  values, sometimes up to 60°C higher than the curing temperature, depending on the cure time. The adhesives properties of these materials on brass substrates have also been examined using a fracture mechanics test, the double cantilever beam test (DCB). The adhesion was found to be very similar (sometimes higher) than that of some commercial epoxy systems. Finally, the copper-polymer interface was characterized using the surface enhanced Raman scattering (SERS) technique. SERS showed the presence of a triazole-based compound adsorbed on copper. The adhesion strength of these “click” polymers on copper substrates is believed to be function of the formation of the triazole-copper complex.

## Abbreviations

DSC	Differential scanning calorimetry
DMA	Dynamic mechanical analysis
T <sub>g</sub>	Glass transition temperature
DCB	Double cantilever beam test
SERS	Surface enhanced Raman scattering
Cu	Copper
Cu(I)	Copper ion (primary)
Cu(0)	Copper metal
Cu(II)	Copper ion (secondary)
CO <sub>2</sub>	Carbon dioxide
Kcalmol <sup>-1</sup>	Kilocalory per mol
H <sub>2</sub> O	Water
O <sub>2</sub>	Dioxide
CPMV	Cowpea mosaic virus
Fuc-T	Fucosyltransferases
CuI	Copper iodide
η	Viscosity
G <sub>e</sub>	Equilibrium modulus
T <sub>cure</sub>	Curing temperature
T <sub>g∞</sub>	Glass transition temperature for a fully cure system
TTT	Time-temperature-transformation
T <sub>g0</sub>	Glass transition temperature of the prepolymer
<sub>gel</sub> T <sub>g</sub>	Temperature at which gelation and vitrification coincide
THF	Tetrahydrofuran
DMAP	4-Dimethylaminopyridine
TLC	Thin layer chromatography
Et <sub>3</sub> N	Triethylammoniac
Et <sub>3</sub> N·HCl	Drochloride
EtOAc	Ethyl Acetate
Na <sub>2</sub> SO <sub>4</sub>	Sodium Sulphate

## Abbreviations

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Mmol	Millimole
ML	Millilitre
NaN <sub>3</sub>	Sodium azide
G	Gram
EtOH	Ethanol
°C	Degree celcius
NMR	Nuclear magnetic resonance
<sup>1</sup> H-NMR	Nuclear magnetic resonance of Hydrogen
<sup>13</sup> C-NMR	Nuclear magnetic resonance of carbon 13
CDCl <sub>3</sub>	Deuterated Chloroform
<i>R<sub>f</sub></i>	Resolution factor for chromatography
Mp	Melting point
δ	Chemical shift (in “Monomers synthesis” section, Chapter 3 and 5)
s	Single peak
d	Doublet peak
t	Triplet peak
H	Hydrogen
<i>J</i>	Coupling constant
Hz	Hertz
IR	Infrared
MS	Mass spectroscopy
<i>m/z</i>	Relative intensity
M	Molecular ion peak
Na	Sodium
HRMS	High resolution mass spectroscopy
Calcd	Calculated



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