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FRACTURE MECHANICS CHARACTERISATION OF EPOXY RESINS BY MEANS OF MINI-COMPACT-TENSION-SPECIMENS

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ABSTRACT

Epoxy resins typically show a high modulus of elasticity and nearly elastic stress-strain behaviour, but poor resistance to fracture. In case of micro-electronic applications such as chips a complex material behaviour occurs due to different mechanical and thermal properties throughout the sandwich, which can influence the mechanical and thermal reliability as well as the life time. The produced residual stress and material inhomogeneities as well as the high thermal gradients cause local defects such as cracks in the sandwich structure. As a result of this failure behaviour fracture mechanics methods are increasingly used for characterising and optimising the toughness of epoxy resins. Applying fracture mechanics concepts makes it possible to gain much information to characterise fracture toughness of new-developed or modified materials using miniaturised specimens.

In this study the influence of chemical structural parameters, filler content and filler geometry, and test conditions on the crack resistance behaviour and fracture mechanics parameters is examined. The investigation of fracture using small specimens is based upon analysing crack resistance against stable and unstable crack growth behaviour under quasi-static loading of standard specimens by means of experimental fracture mechanics. Furthermore, in this paper fracture properties and toughness behaviour of differently filled and modified epoxy resins for microelectronics applications are investigated.

KEYWORDS

Fracture Behaviour, Toughness, Compact-Tension-Specimen, Crack Initiation and Propagation, Epoxy Resin, Laser double scanner

INTRODUCTION

Epoxy resins are used in a wide range for microelectronics application and electronic packaging [1]. At room temperature crosslinked epoxies typically exhibit a high modulus and a nearly elastic stress-strain behaviour, but they have poor resistance to fracture. A complex material behaviour can often be observed due to different mechanical and thermal properties of filler and bonded components which can influence the total mechanical-thermal reliability strongly. In these systems local defects such as cracks can appear as a result of inhomogeneities, residual stress of material and high thermal and mechanical loading gradients between the different materials. Consequently the methods of fracture mechanics have an increasing significance regarding the assessment of failure behaviour. The aim is the optimisation of toughness for epoxies under extreme loading conditions of practice. The industrial application of fracture mechanics values for this purpose and the evaluation of structures basically depends on the geometry independence of these parameters. Only under the condition of geometry-independent parameters the structures developed have an optimum of reliability and operation safety in practice. Compact-tension (CT)-specimens normally used require much material and are often unsuitable for experimental investigations. In the future, the application of miniature specimens for determination of fracture mechanics parameters should be enabled so that an efficient toughness evaluation of new-developed materials can be performed [2].

EXPERIMENTAL

Material

The test material chosen for all investigations was epoxy resin Epilox A-1701 from Leuna-Harze GmbH. In accordance to the manufacturer's instructions the curing was carried out at 160 °C. The resin was used both in the unfilled state and reinforced with silica particles (table 1).
The fracture mechanics values of the cured resins were measured by mini-compact-tension-specimens with the dimensions length L = 25.4 mm, width W = 20.4 mm, and thickness B = 1 - 10 mm. For all specimens the ratio of the initial crack length to the width a/W was 0.55. Additionally, to realize a sharpened crack tip, the specimens were notched with an industrial razor blade.

TABLE 1
INVESTIGATED MATERIALS

Filler	Producer	Type of filler	Diameter
Teco Sil 44 L	C-E-Minerals	Glass powder	9 - 13 µm
Sheriglass 5000	Potters Ballotini	Glass balls	3.5 - 7 µm
Sheriglass 2024	Potters Ballotini	Glass balls	100 - 200 µm

Test techniques

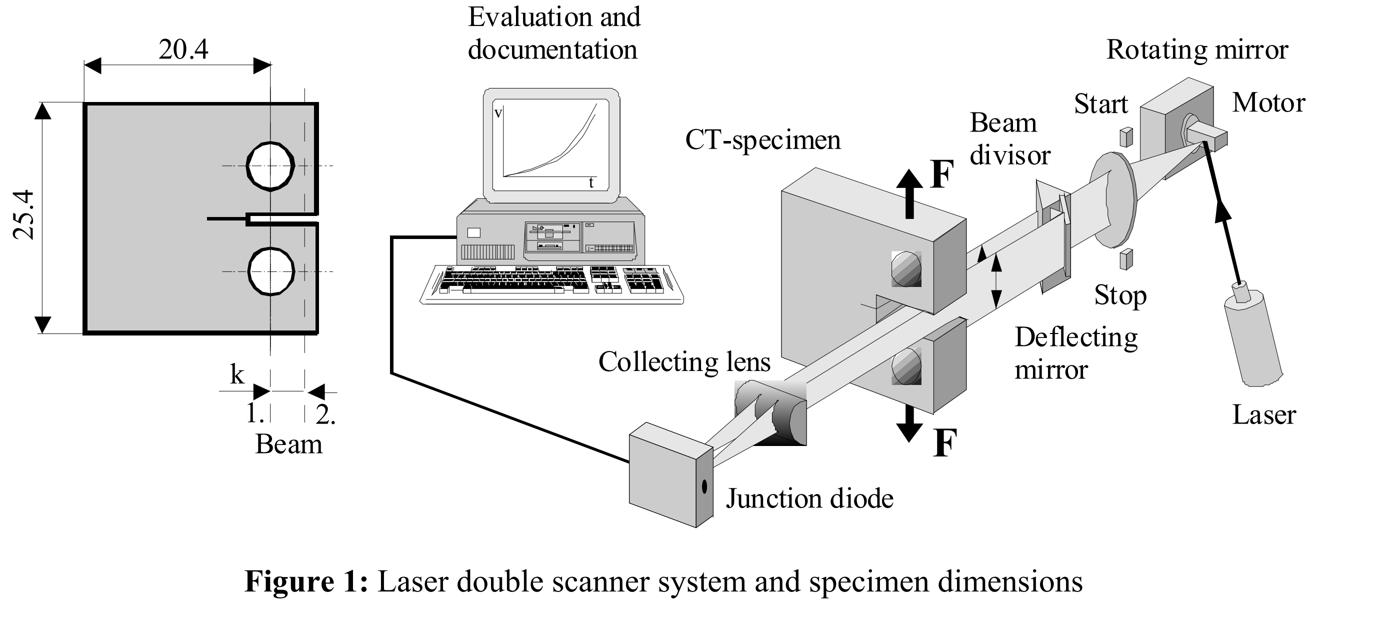

Figure 1: Laser double scanner system and specimen dimensions

All tests were executed on an INSTRON universal testing machine. The load-line displacement was measured by means of a laser scanner system. The laser double scanner used is favoured for the experimental fracture mechanics examination and works in transmission mode with two parallel laser beams. With these two beams the load-line displacement and crack mouth opening displacement can determined by contactless measurements for the shown specimens (fig. 1) [3].

RESULTS AND DISCUSSION

Unfilled and unmodified epoxy resins are brittle and exhibit unstable crack propagation. The estimation of fracture mechanics parameters (stress intensity factor K and J-Integral) by using miniature specimens bases on the analysis of the crack resistance behaviour under quasi-static loading.
Toughness values of epoxy resin depend on specimen thickness. Because the experimental estimation of the minimum thickness B_{min} is very expensive due to the required great number of specimens, of alternative geometry criteria should be used. The empirical geometry criterion (Eq.1) is valid and independent of loading conditions and the nature of materials failure.

$$B_{\min} = \beta\left(\frac{K_{Ic}}{\sigma_Y}\right)^2 \quad (1)$$

The geometry factor β depends on material and on the loading conditions, but it is often considered to be constant. In case of double-logarithmic plotting of β in dependence on the stress-intensity factor K the value β decrease with increasing toughness independent on the used standard or testing protocol. The experimentally obtained fracture mechanics values will be geometry-independent, if it fulfil this geometry criterion of minimal specimen thickness $B_{min.}$ With increasing thickness of mini-compact-tension specimen geometry independent fracture mechanics values can be found for the epoxy resins. The critical minimum specimen thickness was determined to be 4 mm (fig. 2-filled symbols).Thereby, the fracture mechanics parameters can express the influence of material structure, loading rate, and the test temperature on the toughness by using (CT-) specimens.

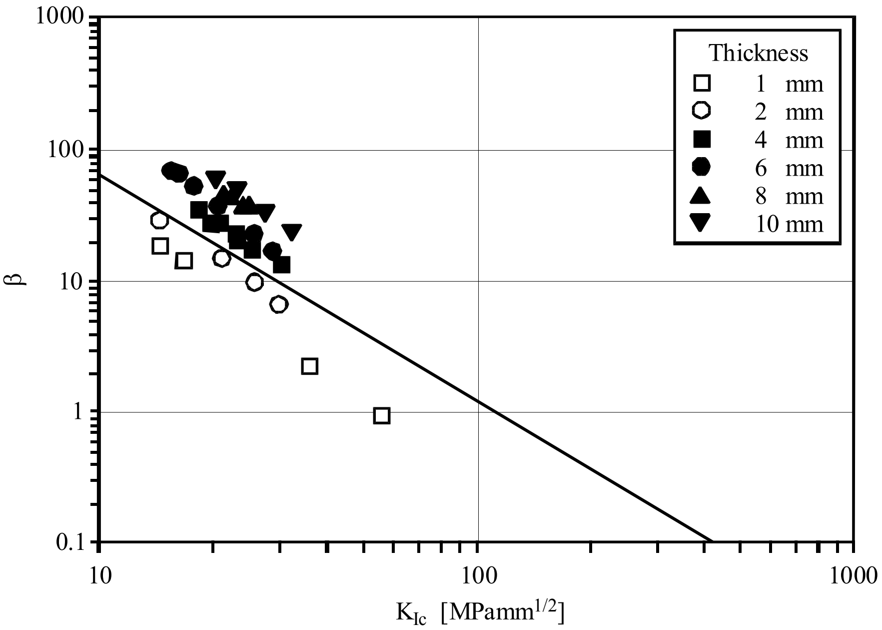

Figure 2: Requirements on specimen geometry for the determination of critical fracture toughness values K_{Ic} with miniature specimens

Under the view of practice, the fixing of employment limits for epoxies is of a special significance and requires the description of the mechanical properties in dependence on temperature. Up to a temperature of 100°C the toughness behaviour changes insignificantly, but if the temperature is further rising, the fracture toughness increases rapidly (fig. 3). The maximum load reaches the highest value at 140°C just below the glass transition temperature (T_g). This increase of fracture toughness at higher temperatures is caused by an increasing molecular mobility of the network chains. On the other hand, the temperature-induced decrease in the yield stress of the polymers leads to a larger plastic zones in front of the crack tip connected with a crack blunting [4].

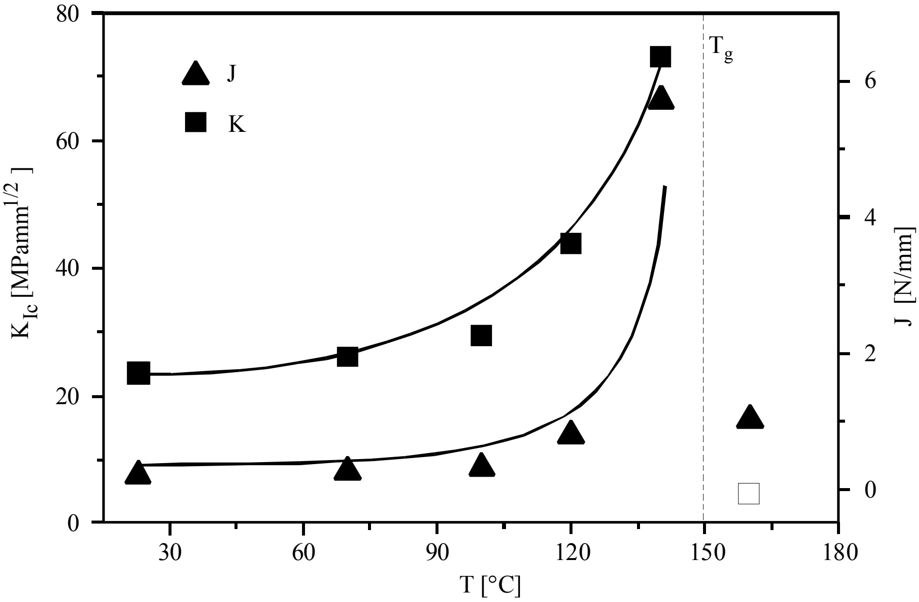

Figure 3: Effect of temperature on fracture toughness for unfilled epoxy resin

The toughness improvement of polymers can be achieved by an enhancement of energy dissipation capability. The efficiency of toughness increase depends on filler geometry, content and chemical bonding. Generally for the materials investigated the coupling to the matrix is insufficient. Because there exist no interaction between matrix and filler, especially the modification with glass balls show a very small effect in toughness behaviour (fig. 4). In opposition to this result the toughness of glass powder filled resin increases essentially, which is caused by the irregular geometry of the powder.

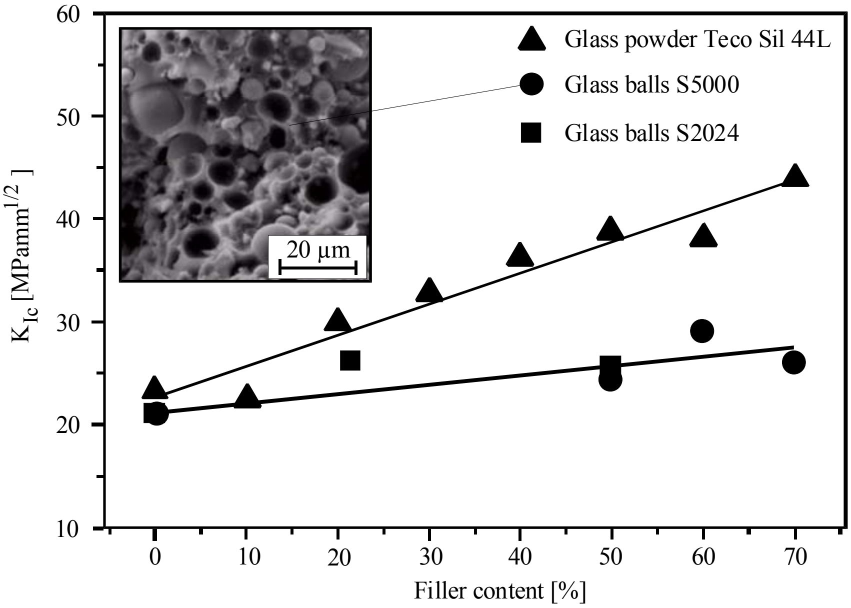

Figure 4: Fracture toughness of differently filled epoxy resin as a function of filler content

If the glass powder filled material is modified with the plasticizer M670 additionally, the chemical activity of this flexibilizier leads to a higher fracture toughness in comparison with the unmodified ma-

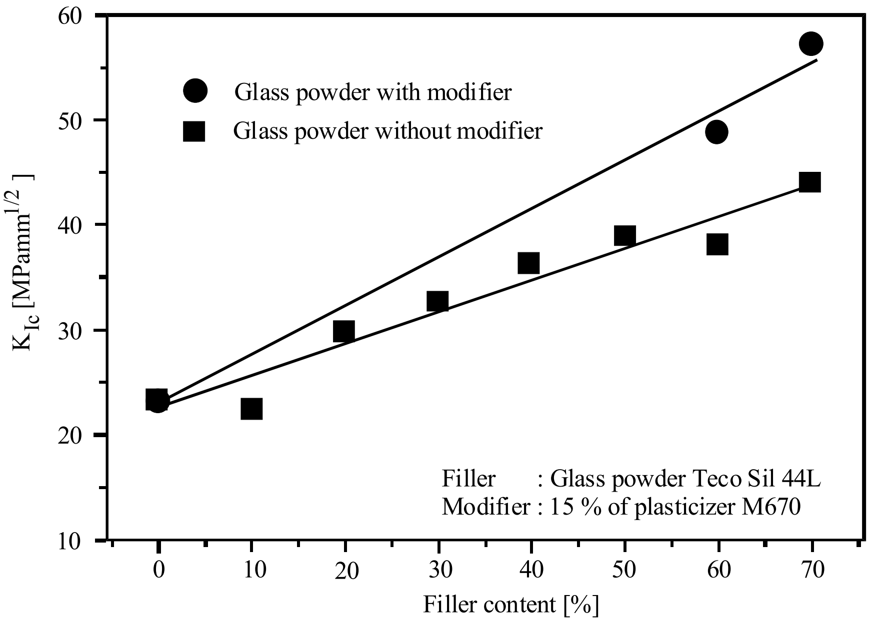

Figure 5: Fracture toughness of differently filled epoxy resin as a function of filler content

terial (fig. 5). Because of the appearing stress distribution the flexibilizier causes an increase of the ductility of the resin matrix and the adhesion between matrix and filler particles is improved simultaneously.

CONCLUSION

The miniature specimens used allow the evaluation of the fracture toughness and the assessment of deformation behaviour. The dependencies of stress intensity factor K on specimens thickness determined using miniature specimens lead to geometry values β those can be introduced into the general dependencies β = f(K). That means, the experimentally determined fracture mechanics values fulfil the geometry criterion and are independent of loading conditions and nature of material failure. At higher temperatures the fracture toughness increases and reaches the maximum near the glass-transition temperature. In the case of filled materials, the fracture toughness is always better if irregular particles such as glass powder are used. Because of the effect of chemical flexibilizier on matrix behaviour and bonding mechanisms the best results were measured for filled and modified epoxy resins. The laserextensometry, used for these investigations, is a successful method for determining the specific material behaviour of epoxy resins by means of miniature test specimens.

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