INVESTIGATIONS OF PHENOLIC RESINS USED AS A MATRIX MATERIAL IN C-FIBER REINFORCED COMPOSITES

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INTRODUCTION

The phenol formaldehyde resins (PF resins, Phenolplasts) are frequently used as a matrix material for CFRPs (Carbon Fiber Reinforced Plastics), C/Cs (Carbon /Carbon Composites) and C/C-SiC composites. Phenol can be derived as well from crude oil as from charcoal. The supply situation of PF resins is stable and the costs allow a widespread application.

Charcoal and phenolic resins are polymers that have similarities in their structural changes which take place under thermal treatments like pyrolyses /1/. As a matter of fact some basic structural properties of the charcoal and PF resin state are transferred to the carbonized state whereas other resin properties affect the gained macroscopical appearance /2/. The occurrence of macroscopical pores in the carbonised state of charcoal and phenolic resins are such examples. This shows the need to tailor the PF resins chemically and to realise adequate curing processes.

A phenomenological model of the processes leading to microstructural changes of carbon materials is applied and used as a guide to modify raw materials and the carbonisation process. Subsequently the generated structures of the carbon materials derived from modified resins are monitored using standard techniques for carbon materials /3/. Parameters that influence the resin structure obtained after the carbonization are changed. Phenol formaldehyde resin synthesis is performed to realize these variations. The produced resin is used for the subsequent composite fabrication. A correlation of experimental results gained from macroscopical-mechanical investigations (DMTA) and microstructural investigations (optical microscopy; REM; TEM) is searched for. Chemical resin modifications are performed and CFRPs and C/Cs are produced under well controlled conditions. DMTA-experiments are performed to reveal the macroscopical effects of the performed microscopical chemical changes. A powerful source for experimental mechanical results is the Dynamical Mechanical Thermal Analysis (DMTA) /4/ and the Transmission Electron Microscopy (TEM) when combined with the pinpointed and reliable Foused Ion Beam FIB/TEM preparation /5/.

EXPERIMENTAL

CFRP samples of approximately 100 mm x 25 mm x 3 mm are produced by infiltrating stacks of carbon fiber cloth which precisely cut out of well defined textiles (bundle size, weight per unit area, roughness etc.). The raw phenolic resins are made by Dyneea GmbH, Erkner, Germany. They were characterised and chemically modified at the TU Chemnitz and infiltrated by gravity infiltration into preloaded moulds. Curing takes place under pressure (50 bar) and controlled time temperature profiles in an autoclave (PARR). The finished samples are sectioned into bending bars of 45 mm x 8 mm x 3 mm by a Struers Accutom saw. DMTA uses a 3 point bending geometry with 30 mm supporting length. The applied Dynamical Mechanical Analyser is the Model EPLEXOR 150N and made by GABO GmbH, Ahlden, Germany. A high temperature furnace (temperature range RT – 1000°C) securing an inert gas atmosphere (Nitrogen stream of 250 liters per minute) is used. The EPLEXOR 150N allows the acquisition of the temperature dependence of the elastic modulus and of the internal damping tanδ. Complementary to the mechanical measurements thermogravimetric and microstructural investigations (optical microscope, REM, TEM) are performed.

RESULTS

The selection of solvents in order to change the resin`s viscosity is an important factor to control the macroscopical porosity of the CFRP material and determines the suitability for a subsequent carbonisation, as the porosity existing in the CFRP state is not redeveloping. The porosity on microscopic scale, the kind of network which is forming and the thermal stability can be influenced by controlling the polymerisation conditions via raw materials and by adequate thermal processing. Thermal mechanical loading under Ar inert gas atmosphere shows that structural changes efficient to the mechanical properties finalize at temperatures below 550°C. The Dynamical Mechanical Thermal Analysis (DMTA) reflects changes in the polymeric state at temperatures below150°C.

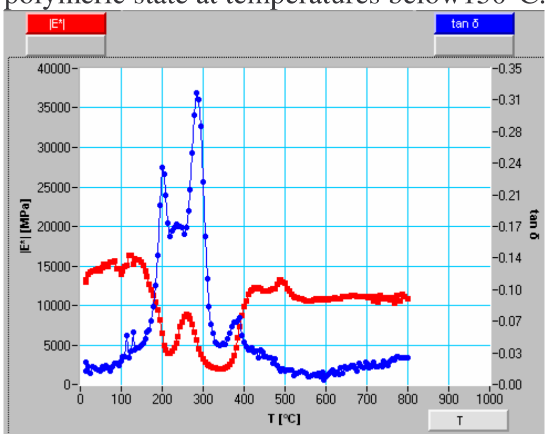

Fig 1: CFRP, Resin E97783 + Diethylenetriamine, THF

Resin matrices which are effective to the mechanical properties (E-Modulus, damping tanδ) take place only within the temperature range from 150°C to 600°C. The constancy of the Elastic Modulus and damping at temperatures above 600°C is obtained by supplying an intensive inert gas stream. The presence of oxygen above 600°C would decrease the stiffness steadily. The DMTA analysis is able to discriminate chemical changes of the resins on macroscopical mechanical level. All experimental resins are based on resin made by Dyneea, Erkner (type E 97783). The microstrutural analysis reveals a matrix porosity in nano scale by applying the Focused Ion Beam thin film preparation technique and by accessing closed porosity for imaging.

LITERATURE

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