The development of cellulose nanofiber / alkoxysilane hybrid material

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INTRODUCTION

The sol-gel process is a technique to hydrolyze and polycondense alkoxysilanes like tetraethoxysilane (TEOS) into polysiloxane (Si). By using this technique, it is possible to make organic-inorganic hybrid compounds at low temperature. In this research, transparent alkoxysilane, which is resistant to high temperatures but is extremely fragile (brittle), was combined to cellulose nanofibers (CNF), and the effects of this reinforcement in the hybrid material were evaluated at various fiber contents.

MATERIALS and METHODS

The starting material was microfibrillated cellulose (MFC) produced by Daicel Chemical Industries, Ltd., a mechanically fibrillated kraft pulp. CNF was obtained by further fibrillating a 1wt% MFC water suspension through a grinder (MKCA6-3, MASUKO SANGYOU Co., Ltd.).

A 0.2wt% CNF water suspension was filtered using a membrane filter and CNF sheets were obtained after drying. CNF sheets were immersed in a solution of silanol derived from TEOS or methyltrimethoxysilane (MTMS). Thereafter, the impregnated chemical compounds were hydrolyzed by drying for 15 minutes at room temperature and polycondensed by drying for additional 15 minutes at 120℃ or 140℃. In this way, about 85wt% fiber content hybrid materials (CNF/Si hybrid and CNF/SiMe hybrid) were obtained.

Alternatively, CNF was added to a solution of silanol derived from MTMS, and a given amount of the stirred solution was put into petri dishes. Subsequently, those were hydrolyzed by drying for 2 days at room temperature. After drying for 2 days at 50℃, those samples were drawn from the petri dishes and were polycondensed by heating for 1 day at 140℃. Thus, hybrid materials with fiber contents of 0wt%, 1wt%, 3wt%, 5wt%, and 10wt% were obtained.

RESULTS and DISCUSSION

By combining CNF sheets and transparent alkoxysilane, the regular transmittance of hybrid materials at 589nm was over 80% (Fig.1). The fracture strain of CNF/Si hybrid and CNF/SiMe hybrid increased over 2.5 times (Fig.2). These results suggest that hydroxyl groups of cellulose were bound covalently to Si or SiMe. The fracture strains of low fiber content hybrid materials increased from 0.7% to 1.3% at an addition of only 1wt% CNF, then tensile strength and fracture strain increased accordingly to the increase of CNF content (Fig.3). This is attributed to the fact that CNF formed a complex with SiMe at a nano scale level as CNF was evenly dispersed in SiMe.

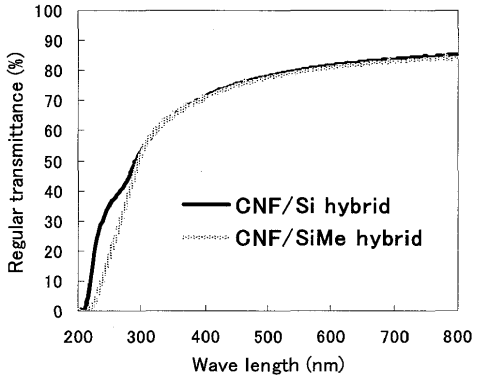

Fig.1 Regular light transmittance of high fiber content hybrid materials

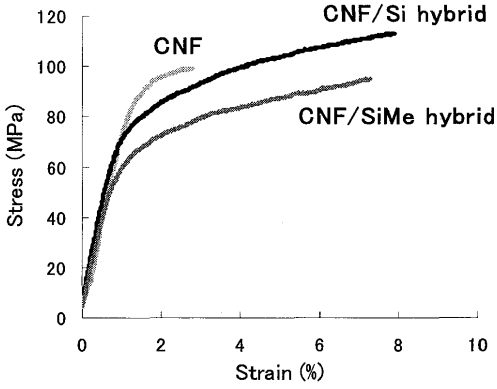

Fig.2 Stress-strain curves of high fiber content hybrid materials

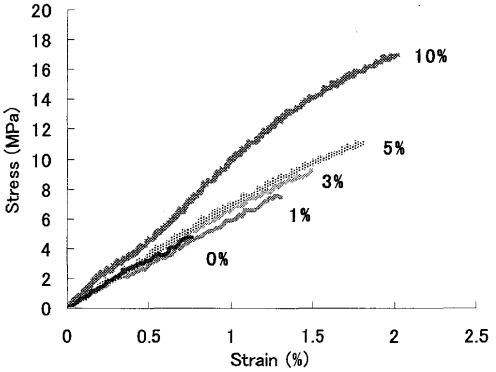

Fig.3 Stress-strain curves of low fiber content hybrid materials