A Tentative Preparation of Glassy Carbon Monofilament from Thermosetting Resins.*

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Till now, there seems to have never been any literature except a patent[1] describing about the preparation of filament from any thermosetting resin, from which glassy carbon material can be derived. In order to prepare the glassy carbon monofilament tentatively, various mixed solutions, consisting of initial condensates of furfurylalcohol and furfural, and of phenollic resin, were examined in detail as its starting material. A field located in diagram of ternary system of (1)resol, (2) novolak and (3)initial condensate of furfurylalcohol and furfural, where one can carry out a spinning procedure with ease, could be decided. The optimum field was 7:1:2 or 8:1:1 (=(1):(2):(3)). It was evaluated by the most simple spinning apparatus, however an improved installation is being investigated. In practice, the filament could be formed chiefly by extrusion and spool-spinning method followed by quenching with ice-cooled aqueous solutions of acids such as hydrochloric. By determining the relationship between tensile strength of the carbonized filament and curing and carbonizing conditions, the optimum condition was searched.
An example obtained by such a procedure is given as it follows: 7 Parts of a resol(derived from phenol and formaldehyde, molar ratio=1:2, catalysed by ammonium hydroxide, viscosity≒100 centipoise at 70°C, mol.wt.=367), 1 part of a novolak(derived from phenol anf formaldehyde, molar ratio= $1:0.8_8$, catalysed by hyrochloric acid, mol.wt.=354)and 2 parts of initial condensate of furfurylalcohol and furfural(wt. ratio≒8:2, viscosity≒80 centipoise at 30°C, mol.wt.=185) were carefully mixed unto a homogeneous solution. It was then heated and kept at 70---75°C, followed by spinning through a nozzle having a diameter of 1 mm under a pressure of 1.05 ---1.1 kg/cm^2 of nitrogen, then by cooling with 2---5°C of 20%-aqueous solution of hydrochloric acid. The specimens thus spun were immersed in this solution at room temperature for 12 days, followed by washing and drying. 400---420°C/hr. was employed as its carbonization rate. Tensile strength and Young's modulus in relation with diameter of 1000°C heat-treated filaments are shown as following:

Diameter(μ)	Tensile Strength (x10^{3}psi)	Young's Modulus (x10^{6}psi)
8---10	180---300	15---30
10---12	60---300	12---25
12---14	35---130	8---15

By employing the above mentioned new installation for spinning, it is expected that the diameter could become thinner.

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1) S.Yamada and S.Nakamura (to Tokai Electrode Mfg.Co.); Japanese Patent, determined to be registered(Publ. No. Showa 41-15727, applied on 10 August, 1963).