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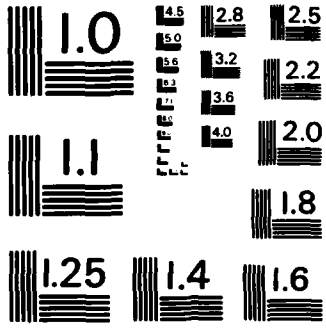

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The objective was to develop processible preceramic polymers amenable to transformation into pure boron nitride for potential applications as fibers and coatings. A series of alkyl, aryl, and trimethylsilyl-substituted borazines was synthesized and subjected to pyrolysis to determine the type of substituent leading to an optimum preceramic polymer. The feasibility of the process was established. From trimethylsilyl-substituted precursors, following stepwise pyrolysis, fibers were drawn and cured in an ammonia atmosphere giving pure boron nitride. Mechanisms responsible for borazine condensations were elucidated; processes involved in precursor synthesis were investigated.

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SUMMARY OF WORK ACCOMPLISHED

A series of borazines, namely B-trichloro-N-triphenylborazine, B-triamino-N-trimethylborazine, B-triamino-N-triphenylborazine, B-trianilinoborazine, B-trichloro-N-tris(trimethylsilyl)borazine, B-triamino-N-tris(trimethylsilyl)borazine, and B-tris[di(trimethylsilyl)amino]borazine was synthesized. All the materials were evaluated as potential candidate monomers for preceramic polymer formation. The halo-substituted materials yielded exclusively glassy products; the alkyl-substituted members of the series, due to their ease of sublimation and preferential ammonia elimination, did not appear to be promising precursors of boron-nitride systems; the pyrolysis products of the aromatic group containing borazines (both homo- and copolymers) exhibited high melting points even at low degree of polymerization. B-Tris[di(trimethylsilyl)amino]borazine was recovered unchanged following exposure to 400°C. Pyrolysis up to 1000°C of the phenyl-substituted borazines did not afford carbon-free boron nitride. The highest combined weight loss of 66.7% registered by the trianilinoborazine was significantly lower than the 79.0% required for boron nitride formation. In all the instances, the residues were black pointing to the presence of carbon. These results were contrary to the claims made in patent literature. Preceramic polymers amenable to fiber drawing were obtained from B-triamino-N-tris(trimethylsilyl)borazine on pyrolysis at 65-227°C. Further pyrolysis and cure in an ammonia atmosphere at 65-970°C resulted in pure boron-nitride fibers.

The early stages of the polymerization process, both in the trimethylsilyl and phenyl-substituted borazines, were found to occur in a stepwise fashion accompanied by ring opening and closing giving what appears to be fused alternating six- and four-membered ring systems. The operative mechanisms

were elucidated, based on the volatiles evolved (nature and quantities) and telomers formed (molecular weight, composition, and spectral characteristics).

In addition, efforts were expended at synthesis of other trimethylsilyl-substituted borazines and precursors. Types of products formed were found to depend strongly on relative quantities of reactants, nature of solvents used, temperature, and the presence of acid acceptors.

PUBLICATIONS/PATENTS

1) K. J. L. Paciorek, R. H. Kratzer, D. H. Harris, and M. E. Smythe, American Chemical Society meeting, April 8-13, 1984 (Polymer Preprints, 25 (1), 15-16 (1984)).

2) K. J. L. Paciorek, D. H. Harris, and R. H. Kratzer, "Boron-Nitrogen Polymers. I. Mechanistic Studies of Borazine Pyrolyses", J. Polym. Sci., in press.

3) K. J. L. Paciorek, R. H. Kratzer, D. H. Harris, M. E. Smythe, and P. F. Kimble, "Boron Nitride Preceramic Polymers", patent application No. 733,457, filed May 13, 1985.

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