OFFICE OF NAVAL RESEARCH

FINAL REPORT

for

Contract N00014-87-C-0713

R&T Code s400029srh

BORON NITRIDE AND ITS PRECURSORS

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The objective was to develop processible preceramic polymers amenable to transformation into pure boron nitride for applications as fibers and coatings. Pyrolysis of B-tri(amine)-N-tris(trimethylsilyl)-borazine gave a fusible BN precursor amenable to continuous filament fiber (10-20μ) drawing. These were transformed into BN ceramic fibers. From B,B’,B”-trichloroborazine and hexamethyldisilazane was obtained a soluble/nonfusible BN precursor utilized for coatings applications on carbon and alumina fibers. Linear 2-, 3-, and 4-BN systems were synthesized and evaluated. Functional linear BN compounds were prepared. Reactions of the materials synthesized were studied and operative mechanisms postulated.
DESCRIPTION OF PROJECT

The objective of this project was to develop processible precursors to pure BN ceramic. The program involved (a) synthesis of novel monomer systems, (b) preparation of boron-nitrogen polymers, (c) manufacture of preceramic fibers and development of procedures for their transformation into BN fibers. and (d) development of coating technology and application of BN coatings to non-BN fibers and substrates. Concurrent with the above, basic research into boron nitrogen chemistry was to be pursued. This was to include synthesis of novel compositions, mechanistic studies, structure/property determinations, and structure establishment.

SUMMARY OF WORK ACCOMPLISHED

A novel BN preceramic polymer system amenable to melt fiber production was developed. It is based on controlled pyrolysis of B-tri(amino)-N-tris(trimethylsilyl)borazine. The attendant mechanisms have been elucidated. Using the above process, continuous preceramic boron nitride fibers, 10-20μ, were successfully spun and cured to BN ceramic. To better control the formation of the processible BN precursor, mono- and bis(trimethylsilylamino)-substituted B-trichloro-N-tris(trimethylsilyl)borazine were prepared and transformed into the corresponding amino analogues. An improved preceramic polymer resulted. The above undertaking required, among others, the
development of an optimized procedure for the synthesis of B-trichloro-N-tris(trimethylsilyl)borazine.

For BN coatings production, a novel nonfusible but organic solvent soluble precursor was developed by controlled reaction of B,B',B''-trichloroborazine with hexamethyldisilazane. This coating was successfully applied to carbon and alumina fibers and metal surfaces. Transformation into BN ceramic proceeded readily in nitrogen atmosphere. Transparent, continuous films (~0.1μ) resulted. BN sheath could be formed by burning away (air, 750°C) carbon from the BN coated carbon fibers; when the carbon fiber ends were BN sealed, protection against oxidation (air, 750°C) of carbon fibers was achieved.

Novel 2-, 3-, and 4-boron nonfunctional, BN linear chains were synthesized and characterized. For the 2-boron system, X-ray crystal structure was obtained. For this series of compounds, thermal stability and reactivity with ammonia was investigated. Functional (diamino) mono- and diborylamines were synthesized and their reactivity evaluated.

Reactions of trimethylsilylamino-substituted borazines and related systems with hydrogen chloride and boron trichloride were studied and the operative mechanisms elucidated. Utilization of simple boranes such as chlorobis(trimethylsilyl)-aminotrtrimethylsilylaminoborane and aminobis(trimethylsilyl)aminotrimethylsilylaminoborane as potential precursors to precramic polymers was investigated and the processes occurring identified.


