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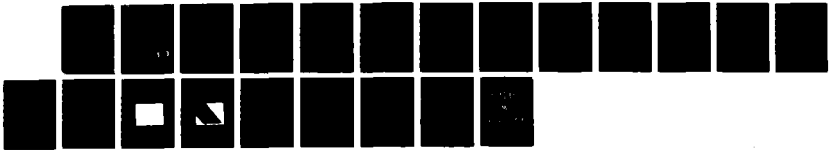

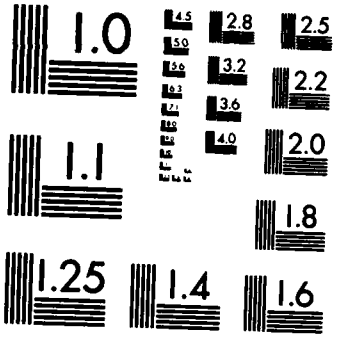

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HIGH YIELD SYNTHESIS OF B_4C/BN CERAMIC MATERIALS BY PYROLYSIS OF POLYMERIC LEWIS BASE ADDUCTS OF DECABORANE(14)

by

William S. Rees, Jr. and Dietmar Seyferth

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Polymers of type $[B_{10}H_{12}\cdot\text{diamine}]_x$ (diamine = $H_2NCH_2CH_2NH_2$, $(CH_3)_2NCH_2CH_2NH_2$, $(CH_3)_2NCH_2CH_2N(CH_3)_2$, etc.) have been found to be useful ceramic precursors. In a stream of argon their pyrolysis gives B_4C/BN, in a stream of ammonia, BN.

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High Yield Synthesis of B_4C/BN Ceramic Materials by Pyrolysis of Polymeric Lewis Base Adducts of Decaborane (14)

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In previous work, we have developed useful polymeric precursors whose pyrolysis provides high yields of silicon nitride or silicon nitride/silicon carbide blends.[1] The main applications of such "preceramic polymers" (preparation of ceramic fibers and coatings and their use as binders for ceramic powders) require that the polymeric precursor be processable, i.e., soluble in organic solvents and/or fusible.

Although the major efforts of workers in the preceramic polymer area have, to date, been directed toward the development of useful precursors for silicon carbide, silicon nitride, "silicon carbonitride" and silicon oxynitride, boron-containing ceramics (the carbide, nitride, phosphides, silicides and others) are a class of ceramic materials whose properties are very attractive in terms of high technology applications.[2] Thus boron carbide has exceptional thermal stability (mp 2450°C), a microhardness of 4.05 GPa (vs 2.53 GPa for SiC), high compressive strength and radiation stability.[3] Various routes exist for the preparation of boron carbide and boron nitride. For boron carbide, however, none of these proceed by way of a processable intermediate. For boron

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nitride, preparative routes based on the pyrolysis of silicon-substituted, oligomeric borazines[4a,b] and of a boric acid/1,2,3-propanetriol condensation product[4c] have been reported.

We report the results of our initial efforts to develop processable polymeric precursors whose pyrolysis gives boron-containing ceramic materials in high yield. The initial objective of our research was the preparation and evaluation of polymers which would serve as precursors for boron carbide or blends of boron carbide and boron nitride.

Various polyhedral borane systems were considered as candidates for the boron-containing component in the design of the preceramic polymer. Since the pyrolysis of the polymer should give a high ceramic yield (to minimize shrinkage and the destructive effect of evolved gases), the pyrolysis chemistry should involve extensive thermal crosslinking so that retention of the pyrolysis charge is maximized. Thermal "cracking", the evolution of volatile molecules containing the elements of interest (B, C, and N in the present case), should be avoided as much as possible. On the basis of these considerations, we directed our efforts to an investigation of the applicable chemistry of decaborane (14) which has a reactive, open nido structure (Fig .1) rather than to the more stable closo borane derivatives. The known reactivity of $B_{10}H_{14}$ is well suited to the preparation of polymeric derivatives. A well-studied reaction of $B_{10}H_{14}$ is the Lewis base substitution process shown in eq. 1. Electron

$$B_{10}H_{14} + 2\,{:}L \longrightarrow L\text{-}B_{10}H_{12}\text{-}L + H_2 \quad (1)$$

donors of diverse type ($:L$) undergo this reaction with $B_{10}H_{14}$ in an

organic solvent at ambient temperature with evolution of one mole of H_2 and formation of $L{\cdot}B_{10}H_{12}{\cdot}L$ compounds whose structure is shown in Fig. 2. There is no polyhedral rearrangement during the reaction, the only structural difference being the relocation of the B-H-B 3-center, 2-electron bridge bonds upon going from one nido structure to the other.[5] It will be appreciated that if the Lewis base molecule used in eq. 1 has two electron pair donor sites, then a polymer should result (eq. 2).

$$x\ B_{10}H_{14} + x\ :L\sim\sim\sim\sim L: \longrightarrow x\ H_2 + [B_{10}H_{12}{\cdot}L\sim\sim\sim\sim L]_x \quad (2)$$

Some examples of such polymers already were reported 25 years ago, in which the difunctional Lewis base molecules ($:L\sim\sim\sim\sim L:$) were $Et_2PCH_2CH_2PEt_2$,[6] Ph_2POPPh_2 and $Ph_2PN=PPh_2\,CH_2CH_2PPh_2=NPPh_2$.[7] These and other phosphorus-containing polymers which we prepared ($:L\sim\sim\sim\sim L:$ = $Ph_2PCH_2CH_2PPh_2$, $Ph_2PC{\equiv}CPPh_2$, $Ph_2PNHNHPPh_2$) proved to be largely unsuitable for our purposes for two main reasons: (1) There was a high retention of excess carbon and phosphorus when they were pyrolyzed to 1000°C under argon (for instance, $[B_{10}H_{12}{\cdot}Ph_2POPPh_2]_x$ gave a 93% ceramic yield on pyrolysis, leaving a residue which contained 52.01%C, 25.30%B, 8.69%P and 12.05%O). (2) Fibers could not be prepared from them, although some of them, e.g., the $[B_{10}H_{12}{\cdot}Ph_2POPPh_2]_x$ polymer served well as binders for B_4C powder in the preparation of shaped bodies and as such in the preparation of ceramic monoliths by pyrolysis of shaped polymer bodies.

Such problems were not encountered with the new $B_{10}H_{12}$·ethylenediamine polymers[8] which were prepared by the reaction of $B_{10}H_{14}$ with the appropriate diamine in a suitable organic solvent. In diethyl ether or tetrahydrofuran medium solvated products, e.g., $\{[B_{10}H_{12}\cdot H_2NCH_2CH_2NH_2]\ (Et_20)_{0.15}\}_n$ in the case of the ethylenediamine product, were obtained. Unsolvated products may be obtained by employing hexane or toluene as the reaction medium. These are soluble in polar organic solvents such as dimethylformamide, dimethyl sulfoxide, hexamethylphosphoric triamide and acetone, but not in hydrocarbon solvents such as benzene, toluene or hexane. The inapplicability of vapor *pressure osmometry* to the determination of their molecular weight suggests that their molecular weights exceed 50,000. Such $B_{10}H_{12}$·diamine polymers were prepared using $H_2NCH_2CH_2NH_2$, $(CH_3)_2NCH_2CH_2N(CH_3)_2$, $(CH_3)_2NCH_2CH_2NH_2$, a commercial 85/15 $CH_3NHCH_2CH_2NHCH_3$/ $CH_3NHCH_2CH_2NH_2$ mixture, $H_2N(CH_2)_3NH_2$, and other diamines. Heating these polymers above 120° C gives materials of reduced solubility in solvents of medium polarity: thermal crosslinking processes appear to be operative. In the case of the $[B_{10}H_{12}\cdot H_2NCH_2CH_2NH_2]_x$ polymer, pyrolysis under argon to 1000° C (10°C/min) left a gray-black amorphous residue in 83% yield. Its composition (analysis for C, B, N) could be rationalized in terms of a constitution $(B_4C)_1\ (BN)_1\ (C)_{0.19}$. Further heating to 1500° C under argon resulted in another 6.8% weight loss and left a ruddy-brown colored ceramic residue which now contained a slight excess of boron. This material, on examination by powder X-ray diffraction, showed the presence of B_4C. Examination of both the amorphous and crystalline pyrolysis products by diffuse reflection

infrared Fourier transform (DRIFT) spectroscopy showed absorptions due to B-C and B-N bonds. Similarly, pyrolysis of $\{B_{10}H_{12}{\cdot}(CH_3)_2NCH_2CH_2N(CH_3)_2\}_x$ gave $(B_4C)_1(BN)_1(C)_{0.53}$ (80%yield) at 1000° C and $(B_4C)_1(BN)_1(C)_{0.17}$ at 1500° C. High ceramic yields were observed in the pyrolysis to 1000° C under argon of other systems: $[B_{10}H_{12}{\cdot}(CH_3)_2NCH_2CH_2NH_2]_x$, 85%; $[B_{10}H_{12}{\cdot}H_2N(CH_2)_3NH_2]_x$, 89%; $[B_{10}H_{12}{\cdot}H_2NC_6H_4NH_2(para)]_x$, 88%. (TGA-derived yields; yields of pyrolysis of larger quantities in a tube furnace usually gave ceramic yields 3 - 10% lower).

Ceramic monoliths may be produced by pyrolysis (under argon) of a rectangular polymer bar. The resulting cerramic bar, uniformly shrunken by ~10%, was found to be of excellent strength. These $B_{10}H_{12}$·diamine polymers can serve as good to excellent binders for commercial boron carbide powder, (0.5g polymer/2.5g B_4C) in that pyrolysis (under argon) of a rectangular B_4C powder/polymer binder bar gave a ceramic bar that had retained its shape without undergoing any discernible shrinkage or bloating. Fibers could be pulled from a syrup of the $[B_{10}H_{12}{\cdot}H_2NCH_2CH_2NH_2]_x$ polymer and DMSO/acetone. The green fibers maintained their form and could be pyrolyzed (to 1000°C under argon) to give black ceramic fibers 3 - 5 μ in diameter. Scanning electron microscopy (SEM) showed them to have a circular cross-section, a smooth surface and no obvious major flaws (Fig. 3). Others of the $B_{10}H_{12}$· diamine polymers noted above were capable of forming fibers. The polymers derived from $(CH_3)_2NCH_2CH_2N(CH_3)_2$ and from the 85/15 $CH_3NHCH_2CH_2NHCH_3/CH_3NHCH_2CH_2NH_2$ mixture melt when heated (mp 246

-250°C and 222 - 225°C, respectively) and may be suitable for melt-spinning.

The $B_{10}H_{12}$·diamine polymers also serve as boron nitride precursors. Their pyrolysis to 1000° C in a stream of ammonia (rather than argon) leaves a white ceramic residue. These samples were, within our limits of determination, spectroscopically indistinguishable from authentic boron nitride. Analytical data supported this. For instance, the pyrolysis of $[B_{10}H_{12}{\cdot}H_2NCH_2CH_2NH_2]_x$ in a stream of ammonia gave a powdery ceramic residue in 62.4% yield which contained B and N in 1.02 : 1 ratio and only a slight amount (0.08g atom/g atom N) of carbon. In a manner like that described above, a ceramic bar was produced by pyrolysis (to 1000°C under ammonia) of a rectangular BN powder/polymer binder (2.7g BN/0.3g polymer) bar. The resulting white bar was of excellent strength and exhibited shape retention in all dimensions. White ceramic fibers, with solid circular cross-sections, could be obtained by pyrolysis of green fibers (produced as outlined above) under an atmosphere of NH_3 (to 1000°C).

The syntheses of the $B_{10}H_{12}$ diamine polymers are easily effected; the polymers are stable at room temperature and their pyrolysis gives a high yield of the desired ceramic product without producing large amounts of excess free carbon or boron. They are soluble in polar organic solvents and so the desired applications may be realizable. It is clear that potentially useful preceramic polymers which serve as precursors for boron nitride and for boron carbide/boron nitride blends are in hand. Detailed studies of the ceramic materials formed in their pyrolysis are in progress.

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8. Materials claimed to be linear polymers, $[B_{10}H_{12} \cdot R_2NCH_2CH_2NR_2]_x$ ($R = CH_3$ and C_2H_5) had been claimed earlier, but their reported complete insolubility in organic solvents indicates that extensive crosslinking (i.e., chemical modification) had occurred during their preparation in benzene at 80°C and thermally effected solvent removal: R. H. Cragg, M. S. Fortuin and N. N. Greenwood, "Complexes of Decaborane. Part I. Ultraviolet Spectra of Some Bis(Ligand) Complexes containing Phosphorus and Sulfur", J. Chem. Soc. A., 1617-1621 (1970).

Figure 1. Structure of Decaborane (14), $B_{10}H_{14}$

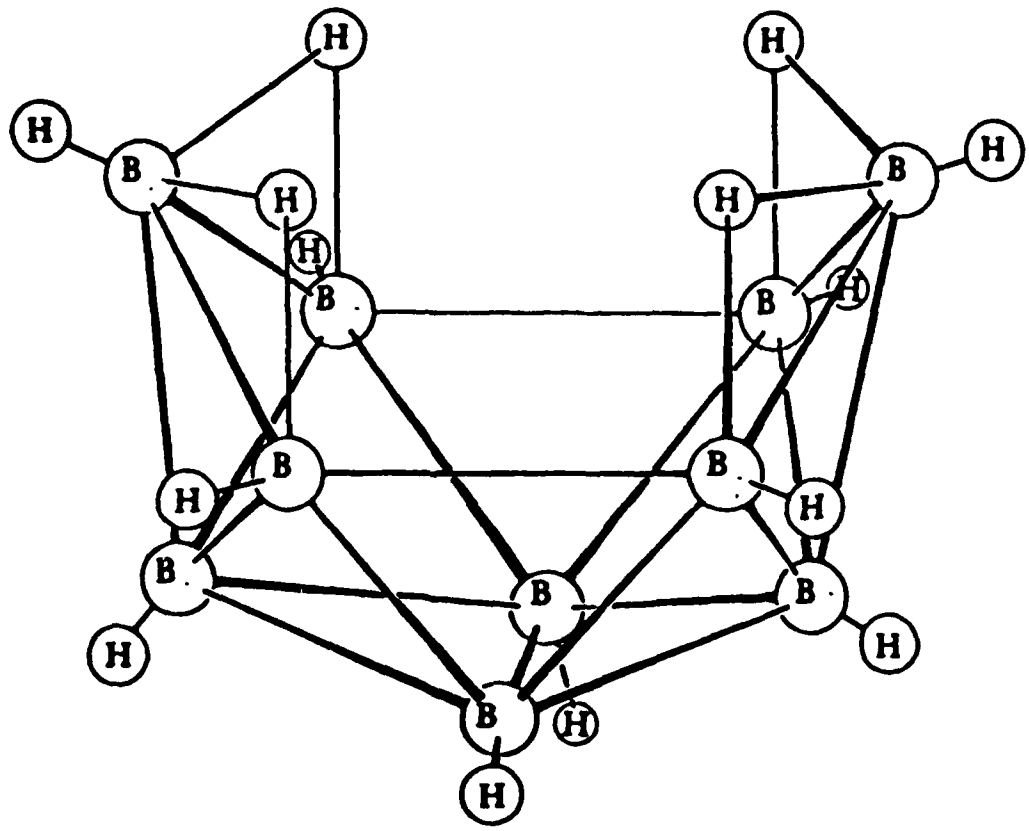


Figure 2. Structure of $B_{10}H_{12} \cdot 2$ Ligand Complexes

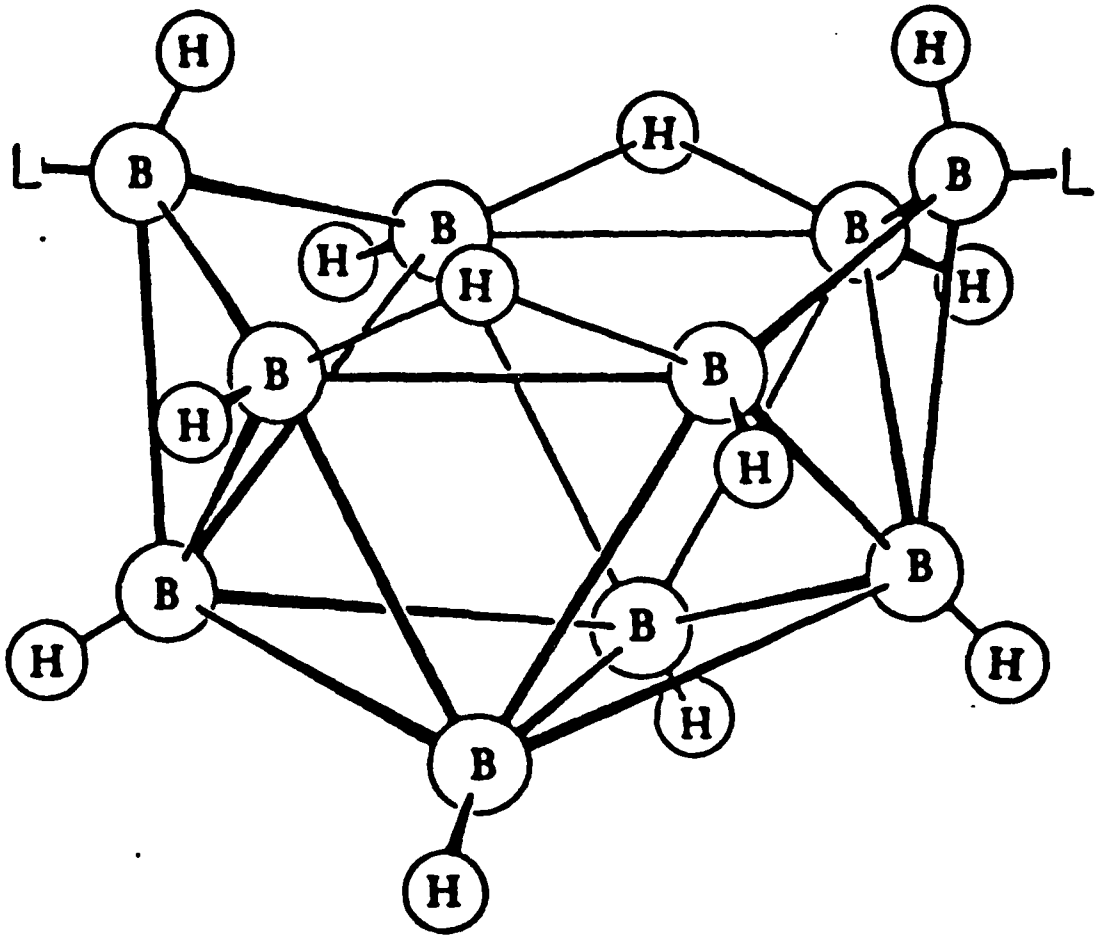
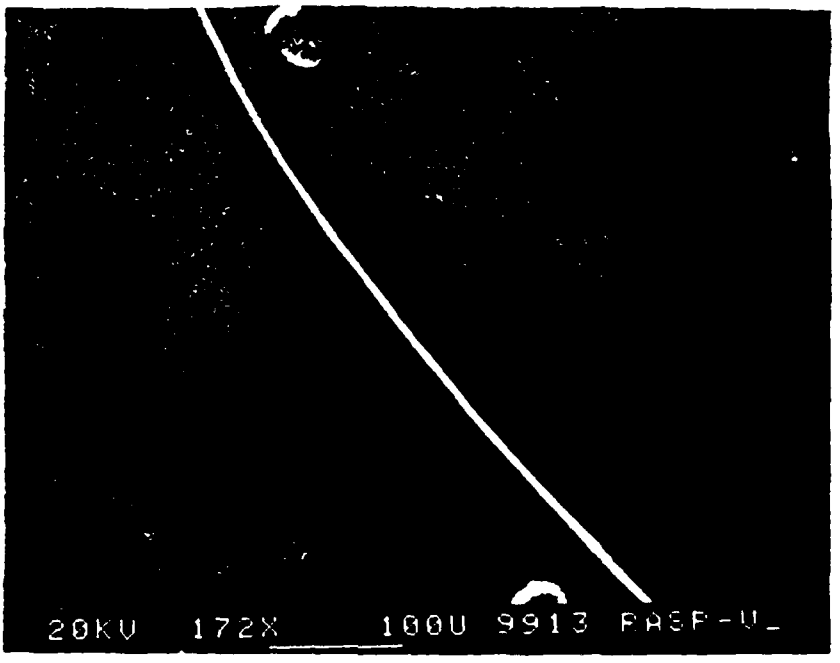


FIGURE 3.

SEM photographs of ceramic fibers derived from $[B_{10}H_{12} \cdot H_2NCH_2CH_2NH_2]_x$

a) note long, regular shape

b) note small size and smooth surface



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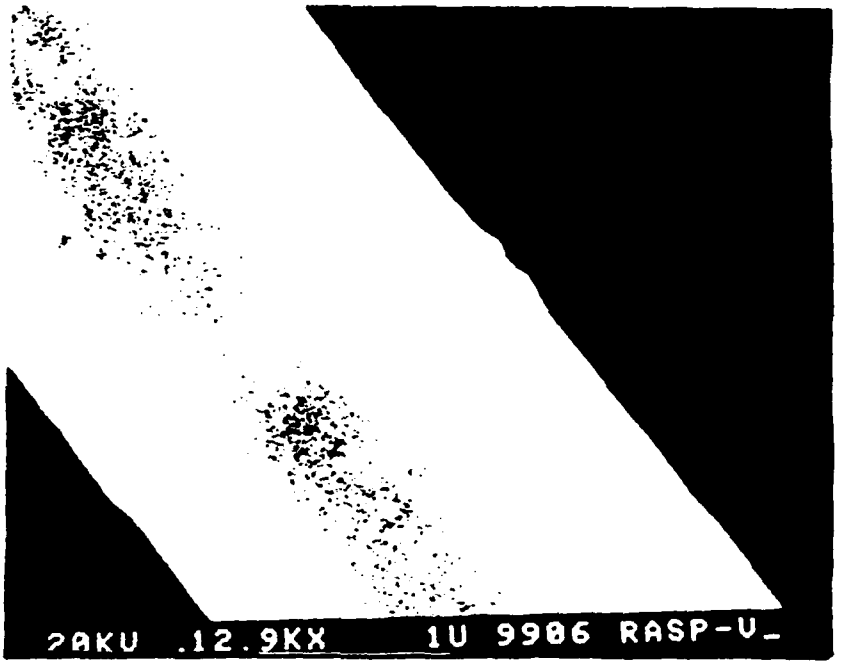

(b)

High Yield Synthesis of B_4C/BN Ceramic Materials of Pyrolysis of Polymeric Lewis Base Adducts of Decaborane(14)

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ABSTRACT

Polymers of type $[B_{10}H_{12} \cdot \text{diamine}]_x$ (diamine = $H_2NCH_2CH_2NH_2$, $(CH_3)_2NCH_2CH_2NH_2$, $(CH_3)_2NCH_2CH_2N(CH_3)_2$, etc.) have been found to be useful ceramic precursors. In a stream of argon, their pyrolysis gives B_4C/BN, in a stream of ammonia, BN.

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