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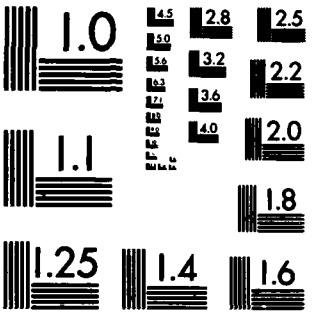

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

SiC-AlN ceramics are fabricated by hot pressing a powder derived from carbothermal reduction of silica and alumina in nitrogen atm. Densities were greater than 99% theoretical. At high temp. a complete solid solution exists but at a low temp. (below ~ 1800°C) phase separation occurs by (i) Nucleation and growth (ii) Cellular, (iii) Spinodal mechanism. This indicates that a miscibility gap exists Thermal conductivity, expansion coefficent, strength, fracture toughness, Youngs modulus were determined. Extensive electron microscopy was conducted.

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SILICON CARBIDE ALLOYS

The principal objectives of this project were to fabricate and characterize SiC-AlN ceramics. Extensive work over the past decade has shown that aluminum oxynitride can be dissolved in Si_3N_4 to form sialons. Little work, however, has been done to form ceramics that are SiC-based solid solutions. Work of Cutler et.al. (1) showed that it may be possible to form an extensive solid solution in the SiC-AlN-Al_2OC system. In the current work, techniques have been developed for fabricating SiC-AlN ceramics and several physical and mechanical properties have been characterized. We have further demonstrated that SiC(2H) and AlN form a complete solid solution above about 2100°C. However, upon heat treatment at a lower temperature (∿ 1700°C), the solid solution decomposes into two solid solutions; one rich in SiC while the other rich in AlN. Optical and scanning transmission electron microscopy was used to characterize the precipitate morphology and to conduct micro-micro diffraction & microchemical analyses.

Based on this work, tentative phase diagram in the SiC-AlN system was proposed. X-ray diffraction techniques were used to identify phases. The present work, however, showed that X-ray diffraction patterns appear single phase even though optical and electron microscopy clearly show the presence of two phases. This discrpancy was rationalized in light of the fact that the lattice parameters of SiC (2H) and AlN are very close. Young's modulus of elasticity, coefficient of thermal expansion, thermal conductivity, microhardness and fracture toughness were determined as a function of composition. Preliminary information was also obtained on the creep rate and strength at elevated temperatures. The procedure used for sample fabrication, the results of phase studies and property measurements are briefly described in the Following.

Carbothermal reduction of silica and alumina in nitrogen atmosphere was used to prepare an intimate mixture of SiC and AlN. Cabosil, $AlCl_3.6H_2O$ and starch were used as sources of Si, Al and C respectively. $AlCl_3.6H_2O$ was dissolved in water and cabosil & starch were dispersed in it. Then NH_4OH was used to precipitate $Al(OH)_3$. Subsequent drying, coking, calcining (to remove volatiles - expecially NH_4Cl) and reacting in N_2 led to the formation of SiC-AlN powder.

During the course of this work, a hot press capable of operating in vacuum or inert atmosphere to a temperature of 2400°C was designed and built. Using this hotpress, samples were hotpressed to near theoretical density using graphite dies. The temperature was generally in the range from 1900°C to 2300°C and the pressure was ∿ 5000 p.s.i.

Samples that were hotpressed or 2300°C were optically homogeneous and the corresponding XRD paterns showed the presence of 2H pattern as the only phase. Samples hotpressed at 2100°C as below showed regions of dark grey in a matrix of light grey. Microhardness measurements clearly revealed that their hardnesses were different. XRD patterns, however, indicated the presence of single phase 2H pattern. TEM foils of the samples were made by ion beam thinning and the foils were examined in JEM 100CX STEM unit. It was observed that samples hotpressed at 2100°C consisted of bimodal grainsize distribution - fine grained regions were rich in SiC while coarse grained (∿ 3-5 μm) regions were rich in AlN. On the other hand, samples that were hotpressed at 2300°C had uniform grain structure and microchemical analysis indicated the composition to be uniform. When samples containing 47 % AlN were heat-treated in the range from 1700°C to 1800°C, two types of precipitate morphologies were observed. In some samples, precipitates were found at 3- and 4-grain junctions indicating the process of phase separation by the normal nucleation and growth mechanism. Microchemical analysis indicated that the precipitates were rich in AlN (∿ 94% AlN-6% SiC). In some samples, modular pre-

cipitates which occurred throughout the grains were observed. Typical wavelength of these modulations was on the order of ∿100 A°. This type of phase separation appears to be of the spinodal decomposition type. Since lattice parameters of AlN & SiC(2H) are very similar, coherent spinodal should not be too far depressed from the chemical spinodal. When the samples are heated to a temperature above 2100°C, the precipitates redissolve and a homogeneous solid solution is formed. Based on this data, we have tentatively concluded that a miscibility gap exists between AlN & SiC (2H). Clearly, much more definitive work is needed to pin down the phase boundaries in this difficult system. When a sample containing 75% AlN was heat treated at 1600°C for several days, lamellar precipitates were formed which could be easily observed under an optical microscope. Microchemical analysis on the STEM unit indicated that the precipitates were rich in SiC (∿ 92% SiC-8% AlN). This precipitate morphology suggests that the phase separation had occurred by the mechanism of discontinuous phase separation. In this sample also, heat treatment at ∿ 1900°C leads to the dissolution of lamellar precipitates to form a single phase solid solution.

Youngs' modulus of elasticity was measured using straingauge method. It was found that the Young's modulus varied linearly with composition. Coefficient of thermal expansion was measured up to 900°C using a dilatometer and was found to vary linearly with composition. A thermal comparator was designed and built in the present study for the determination of thermal conductivity. Thermal conductivity was determined as a function of composition at various temperatures up to 300°C. It was observed that small additions of AlN to SiC or small additions of SiC to AlN led to rapid drops in thermal conductivity and the thermal conductivity exhibited a minimum at about 50% AlN. This behavior is to be expected in the case of a binary solid solution provided the major contribution to the thermal conductivity is the lattice contribution.

Then, small additions of solute leads to a reduction in the phonon mean free path and thereby reduces thermal conductivity. This aspect is expected to be of particular significance for application as a ceramic liner in the adiabatic disel engine where low thermal conductivity is desirable.

Fracture toughness, K_{IC} was determined using the indentation technique. Small amounts of AlN additions to SiC led to an increase in K_{IC} (from 2.8 MPa$\sqrt{m}$ to 4.7 MPa$\sqrt{m}$). Of particular interest would be the fracture properties of SiC-AlN composites formed by discontinuous phase separation. This would be subject of a future study.

Strength of SiC-AlN samples (containing 20% AlN) was measured up to 1400°C and was found to be ∿80 ksi. (∿ 520 MPA). Preliminary creep studies were conducted on fine-grained SiC + 35% AlN and SiC hotpressed with boron. It was observed that creep rates were comparable and above 1450°C, the creep rate of SiC-AlN sample was infact lower than SiC.

The present work has indicated that the properties can be engineered by properly alloying SiC with AlN. Clearly, much more work remains to be done The SiC-Al_2OC system is also likely to present similar versatility.

Based on the present work, two papers have been published, one has been accepted for publication (to appear in April 1983 issue of the J. Am. Ceram. Soc.), One has been submitted for publication and one more is currently under preparation. Details of these are given below.

1) W. Rafaniello and I.B. Cutler "Preparation of Sinterable Cubic Aluminum Oxynitride by the Carbothermal Reduction of Aluminum Oxide" J. Am. Ceram. Soc. 64, [10], C128 (1981)

2) W. Rafaniello, K. Cho and A.V. Virkar "Fabrication and Characterization of SiC-AlN Alloys" J. Mat. Sci. 16, 3479-3488 (1981)

3) W. Rafaniello, M.R. Plichta and A. V. Virkar "Investigation of Phase Stability in the SiC-AlN System" To appear in April (1983) issue of the J. Am. Ceram. Soc.

4) K. Cho and A.V. Virakr "Thermal Conductivity of SiC-AlN Solid Solutions using Thermal Comparator" submitted to the Am.Ceram. Soc. (1983)

5) W. Rafaniello, M. R. Plichta and A. V. Virkar "Phase Transformation in the SiC-AlN System". Under preparation.

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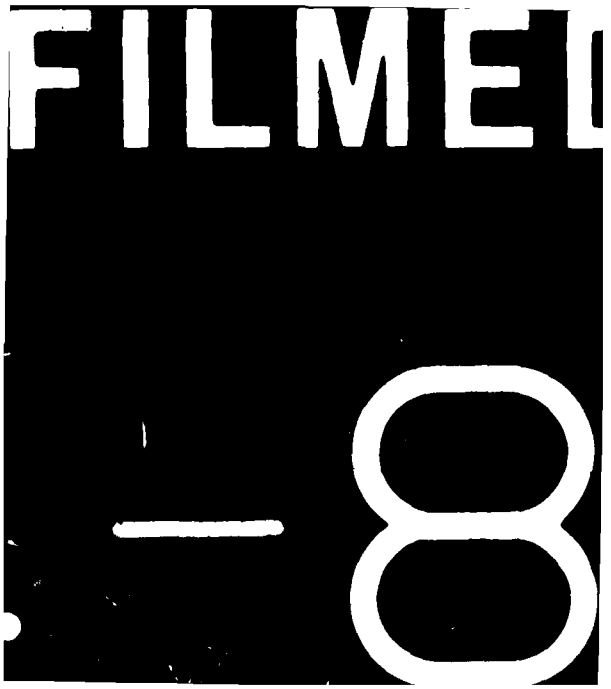
(1) I.B. Cutler and P.D. Miller, U.S. Patent No. 4,141,740 Febr. 27 (1979)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