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13. ABSTRACT *(Maximum 200 words)*

Synthesis of nonoxide powders in a nonthermal (microwave) plasma was demonstrated, using SiC, Si_3N_4 and Si as model materials. Particle sizes are ultrafine, 2-5 nm. Compositions and structures have been characterized. Equipment has been developed and characterized.

Creep of polycrystalline AlN has been investigated for the first time. Mechanisms are identified.

(25)

14. SUBJECT TERMS
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Integrated Synthesis and Post-Processing of Silicon Carbide and Aluminum Nitride

Interim Technical Report for Year 1
Covering the Period September 15, 1989-September 14, 1990

Dr. A. I. Kingon, Dr. R. F. Davis and Dr. A. K. Singh

Submitted to U. S. Army Research Office
Research Triangle Park, North Carolina

on December 14, 1990

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Brief Outline of Research Findings

The objectives of this program are to:

(1) Determine whether crystalline nonoxide powders can be synthesized in a nonthermal microwave plasma.
(2) Develop the necessary equipment to undertake the synthesis investigations, and later develop it for completely anaerobic synthesis and post-processing.
(3) Characterize the properties of ultrafine nonoxide particles, particularly diffusion and creep in assemblies of these particles. The interest is in "superplastic" effects.

In the first year of research, significant progress has been made. It has been shown that nonthermal synthesis is indeed feasible. The demonstrations have been performed on SiC, Si_3N_4 and Si to date. Preparations are being made to handle the precursors for AlN synthesis. Two presentations have been made of this work, at the Fall MRS meeting in Boston. Three manuscripts have been prepared, one of which is in the review process, and two of which are in draft form (attached.)

In addition, the first investigation has been made of the high temperature creep of polycrystalline AlN. Under constant compressive stress the conditions investigated were 100-350 MPa and 1150-1400°C. The activation energy curve shows a knee at ~1300°C. In the low temperature range (activation energy ~400 kcal/mol) the mechanism is grain boundary diffusion controlled creep; at high temperatures (activation energy ~700 kcal/mol) it is lattice diffusion controlled.

Some further details of our results are presented below:

Nonthermal Synthesis of Silicon Carbide

Synthesis was performed at 1torr using SiH_4, C_2H_2 and Ar carrier. Chemical composition was confirmed by XPS and Auger. Phase analysis was by XRD and TEM. Particles are nanosized, 2–5 nm. Many structural modifications were observed, including 3C, 2H, 4H, 15R, 21R. We showed that the results have implications for existing models of polytype formation.

Non-Thermal Synthesis of Silicon Nitride

Similar procedures were utilized for Si_3N_4, although growth rates were lower. The phase is a (hexagonal symmetry). Particle sizes 2–5 nm. Reaction efficiencies have been measured.

Crystalline Nanoparticles of Si

It was observed that the formation of SiC is via Si intermediates or nuclei. Thus synthesis of Si was investigated. Conditions were optimized, and particles characterized as above. The crystallinity is unusually high, considering the size of the particles.

Plasma Characterization

After design and construction of the apparatus, the microwave plasma was characterized by Langmuir probe.

Manuscripts Submitted

1. Synthesis of SiC Clusters in a Nonthermal Microwave Plasma
   A. K. Singh, A. I. Kingon, G14.2, MRS Fall Meeting 1990, 11/26-12/1/90 (in review)
2. Nonthermal Synthesis of SiC and Si_3N_4 Ultrafine Particles
   A. K. Singh, A. I. Kingon, to be submitted to J. Mat. Res. (draft attached)
3. Crystalline Nano-particles of Si by Nonthermal Microwave Plasma
   A. K. Singh, A. I. Kingon, to be submitted to J. Mat. Res. (draft attached)

Presentations

1. Synthesis of SiC Clusters in a Nonthermal Microwave Plasma
   G14.2, MRS Fall Meeting 1990
2. Nonthermal Synthesis of Ultrafine SiC Powders
   N2.7, MRS meeting Fall 1990

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