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CERAMIC MICROSTRUCTURE DEVELOPMENT.(U)
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RF Project 762134/712323

Report No.

PROGRESS

REPORT

By

THE OHIO STATE UNIVERSITY
RESEARCH FOUNDATION

1314 KINNEAR RD.
COLUMBUS, OHIO 43212

To DEPARTMENT OF THE NAVY
Office of Naval Research
800 N. Quincy Street
Arlington, Virginia 22217
Contract No. N00014-80-C-0523

On CERAMIC MICROSTRUCTURE DEVELOPMENT

Progress rept. 1 Apr-15 Aug 80.

For the period April 1, 1980 - August 15, 1980

Submitted by Dennis W. Readey
Department of Ceramic Engineering

Date October, 1980

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Progress Report

on

"Ceramic Microstructure Development"

Contract No. N 0014-80-C-0523

April 1 to August 15, 1980

D. W. Readey

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Personnel

In the original proposal, during the period between the initiation of this contract, April 1, 1980, and October 1, 1980, only one graduate student was anticipated to be available for work on this program. However, due to a number of circumstances, four students, two M.S. and two Ph.D. candidates became available during June, and all four have been appointed graduate research associates on this contract. In addition, two undergraduate students have been supported by the funds provided under this contract. One of these students will receive his B.S. at the end of Autumn Quarter, and it is expected he will enter graduate school at OSU in the Winter Quarter and continue the research initiated this summer which will lead to a masters thesis. In addition, one other undergraduate student, supported by departmental funds, has been working on the research reported here.

Halide Oxidation

Ternary eutectic melts of LiCl-KCl-NaCl have been used as solvents for $NiCl_2$ in the initial experiments on

the preparation of oxide powders by oxidation of halide melts utilizing the reaction:

$$NiC\ell_{2(\ell)} + H_2O_{(g)} = NiO_{(s)} + 2HC\ell_{(g)}.$$

Nickel oxide was chosen for the initial studies because of its ease of identification by color. The ternary eutectic melt was chosen so that a wide range of temperatures could be used to examine the effect of temperature on the oxidation process. Initial experiments have shown that nickel chloride is indeed preferentially oxidized and can be easily separated from the remaining halides by dissolving the latter in water. Scanning electron microscopy indicates a great deal of agglomeration of the powder probably due to uncontrolled nucleation at a number of sites in the apparatus and too high supersaturations. Equipment modifications are being made to permit more controlled oxide particle nucleation and growth from the melt surface only.

Oxide Precursor Melt Decomposition

Most ceramic oxide powders are made by decomposing solid precursor materials such as nitrates, sulfates, etc., by reactions such as:

$$Ni(NO_3)_{2(s)} = NiO_{(s)} + 2NO_{2(g)} + \tfrac{1}{2}O_{2(g)}$$

which results in highly agglomerated powders. By performing such decompositions in the molten state, so that the activity of the precursor and the nucleation and growth of the resulting oxide can be controlled, it is anticipated that nonagglomerated, monodisperse powder can be prepared. After some initial experiments on carbonates and sulfates, nitrates were chosen because of their lower melting points, lower

[illegible] temperature, and greater solubility in H_2O.

Nickel oxide powders have been successfully produced by the above reaction by decomposing nickel nitrate at temperatures between 300°C and 400°C in melts of $LiNO_3$, $NaNO_3$ or KNO_3. Experiments are underway to determine the effect of reactant concentration on the decomposition temperature, particle size and degree of agglomeration of the resultant oxide powders.

Ostwald Ripening in Halide Melts

A few experiments have been performed to determine the possibility of controlling particle size and degree of agglomeration of oxide powders by controlled particle growth or Ostwald ripening in halide melts. Unfortunately, the solubility of oxides in halide melts is not well documented except for the solubility of a few oxides in cryolite melts. Initial experiments in molten KF to 810°C indicate little or no solubility of NiO but considerable silicate solubility as evidenced by considerable crucible attack. Other melt compositions are being examined.

Aqueous Precipitation

Nickel and zinc hydroxides have been prepared by homogeneous precipitation from aqueous urea solutions at elevated temperatures by reactions such as:

$$(NH_2)_2CO + 3H_2O + Ni^{2+} = 2NH_4^+ + NiO\downarrow + CO_2\uparrow.$$

The zinc hydroxide morphology was not very encouraging. On the other hand, spherical particles of $Ni(OH)_2$ have been

obtained as shown in Figure 1. These particles are on the order of one to two micrometers in diameter and are of an ideal size. Further studies on precipitation are in progress to define the conditions under which spherical precipitates occur. Calcining the hydroxide to the oxide is also being studied to determine the morphology of the calcined oxide and whether dense monodisperse spherical oxide powders result.

Particle Packing

After surveying commercial equipment availability and capabilities, a high speed, large volume centrifuge has been ordered for particle packing in aqueous suspension studies.

Sintering and Vapor Transport

Two sintering furnaces are being constructed. One will be to study sintering kinetics as a function of oxygen partial pressure for materials such as ZnO and MgO which undergo significant vapor transport in low oxygen partial pressures by reactions such as:

$$MgO_{(s)} + H_{2(g)} = Mg_{(g)} + H_2O_{(g)}$$

The other is to study the effect of tansport as halides on the densification of oxides. Most oxides can be transported as volatile halides by reactions such as:

$$MgO_{(s)} + 2HC\ell_{(g)} = MgC\ell_{(g)} + H_2O_{(g)}$$

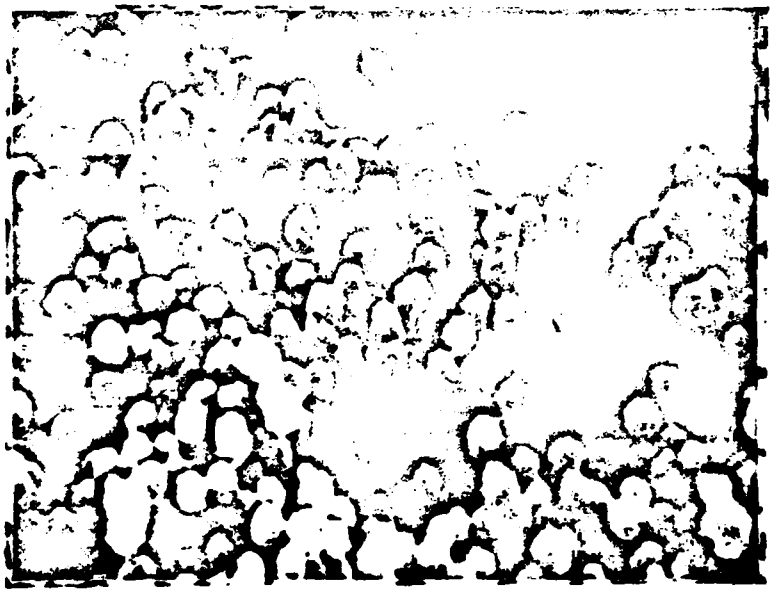

Figure 1. Nickel hydroxide particles prepared by homogeneous precipitation from aqueous solution by decomposition of urea. Top, 10,000X; bottom, 2000X. (Fibers in top photo are from the filter paper.)

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