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**CARBON IMPURITY EFFECT
ON THE THERMAL DEGRADATION
OF A $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$ CERAMIC**

HEINRICH KNOCH and GEORGE E. GAZZA
CERAMICS RESEARCH DIVISION

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ABSTRACT

The oxidation behavior of hot-pressed $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ compounds (principally $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$, N-melilite) was studied in air between 700 C and 1400 C. It is shown that carbon impurity strongly influences the thermal degradation of these compounds. Oxidation of carbon-containing materials in air at 1000 C leads to high weight gains, severe cracking, and complete destruction of the samples. Nominally carbon-free materials show good oxidation resistance without cracking.

INTRODUCTION

Although Y_2O_3 -doped Si_3N_4 exhibits improved high temperature (>1300 C) properties as compared with MgO -doped Si_3N_4 , it was found that severe degradation of the Y_2O_3 -doped Si_3N_4 occurred in an oxidizing environment at intermediate temperatures (~ 1000 C). This effect was shown¹ to be related to the existence in the material of certain quaternary compounds, such as $Si_3Y_2O_3N_4$, $YSiO_2N$, $Y_{10}Si_7O_{23}N_4$, and $Y_4Si_2O_7N_2$ (N-melilite, K-, H-, and J-phase, respectively). A method to prevent this detrimental problem for a Si_3N_4 material containing 13 wt% Y_2O_3 has recently been reported by Gazza et al.² The method involved post heat treatment of specimens in a nitriding environment. Further studies with $Si_3N_4 \cdot Y_2O_3$ compounds suggest that the instability of these compounds, principally $Si_3Y_2O_3N_4$, in air at ~ 1000 C is greatly influenced by the presence of carbon in the material. This finding is significant in that it is the state-of-the-art to hot press Si_3N_4 in graphite dies, i.e., in carbonaceous atmospheres.

EXPERIMENTAL

Two different commercial Si_3N_4 powders were used for producing the $Si_3N_4 \cdot Y_2O_3$ compounds. The Si_3N_4 powder obtained from Hermann C. Starck* was produced by nitridation of silicon while powder obtained from GTE† was produced by a vapor phase process. Both powders were individually mixed with Y_2O_3 ‡ in a 1:1 molar ratio, then ball milled in ethanol for 20 hours using polyethylene containers and tungsten carbide balls. Chemical analysis of the milled powder showed only 0.8 wt% tungsten in the samples. Therefore, the maximum percent carbon introduced by milling is less than 0.04%. Two additional Si_3N_4 (GTE)· Y_2O_3 mixtures were prepared by adding 2 wt% graphite powder to one and 2 wt% beta SiC powder to the other. Table 1 shows analyses of the starting materials together with the major impurities in the Si_3N_4 powders.

The milled and dried powders were hot pressed in graphite dies for 90 minutes at 1750 C in a nitrogen atmosphere with an applied pressure of 30 MN/m². The resulting disks were machined into test bars for oxidation measurements. Some bars were cut leaving as-pressed surfaces on opposite faces, while all faces were ground on the remainder of the specimens.

The oxidation behavior was studied at temperatures between 700 C and 1400 C in air. The samples rested on hot-pressed SiC** pedestals with sharp edges. During the oxidation treatment the samples were periodically removed from the furnace for weight gain measurements and examination.

*H. C. Starck, Goslar, West Germany

†GTE, Towanda, Pennsylvania

‡Molycorp, White Plains, New York

**Norton Company, Worcester, Massachusetts

1. LANGE, F. F., SINGHAL, S. C., and KUZNICKI, R. C. *Phase Relations and Stability Studies in the Si_3N_4 - SiO_2 - Y_2O_3 Pseudoternary System*. J. Amer. Ceram. Soc., v. 60, no. 5-6, 1977, p. 249-252.
2. GAZZA, G. E., KNOCH, H., and QUINN, G. D. *Hot-Pressed Si_3N_4 with Improved Thermal Stability*. Amer. Ceram. Soc. Bull., v. 57, no. 11, 1978, p. 1059-1060.

RESULTS AND DISCUSSION

X-ray analysis showed each of the four hot-pressed materials to be principally $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$ (N-melilite) with some weak traces of YSiO_2N (K-phase) present. Table 2 shows the X-ray diffraction data obtained together with those reported by Rae et al.³ and Lange et al.⁴ for this particular phase. The patterns are in good agreement.

Initial oxidation experiments were conducted with hot-pressed Si_3N_4 (GTE)· Y_2O_3 and Si_3N_4 (Starck)· Y_2O_3 compounds (material B1 and A, from Table 1). Specimens tested had two as-cut surfaces and two surfaces remaining as-pressed. Visual appearance of as-cut test specimens after oxidation at 1000 C in air was similar to that shown in the literature.^{1,5} However, there was a peculiarity of the oxidation effect on the B1 material. Figure 1a shows a piece of sample B1 after 117 hours in air. Only the surfaces which were in contact with the graphite die during hot pressing seemed to be affected and showed signs of volume expansion and cracking. This result suggests that some reaction layer produced during hot pressing at the surface of this material strongly influences its oxidation behavior. The tests were repeated with A and B1 materials, but the as-pressed surfaces were ground off prior to testing. Specimens of material A still exhibited severe cracking while material B1 remained uncracked. Figure 1b shows a sample of this material after 260 hours at 1000 C in air.

Since only unground, as-pressed surfaces (which are most susceptible to graphite die reactions) appear to severely degrade the oxidation resistance of B1 material, it is highly probable that carbon impurity significantly influences

Table 1. IMPURITY CONCENTRATIONS OF THE STARTING Si_3N_4 POWDERS BEFORE MILLING

Impurity Elements	Si_3N_4 (Starck)	Si_3N_4 (GTE)*
	A	B1
ppm Al	1800	<30
Fe	700	<10
Ca	1200	<30
Mg	300	<40
Oxygen, %	1.1	2.6
Carbon, %	0.6	-
Free	<2	-
Silicon, %		

*B2 material had 2 wt% graphite added to base composition
B3 material had 2 wt% SiC added to base composition

Table 2. OBSERVED d - SPACINGS TOGETHER WITH THOSE REPORTED IN THE LITERATURE FOR $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$, N-MELILITE

d (Å)		
Rae et al. ³	Lange et al. ⁴	This Study
5.37	5.37	5.37
4.91	4.91	4.92
4.12	-	4.13
3.80	-	3.80
3.62	3.63	3.62
3.40	3.39	3.40
3.00	3.00	3.01
2.79	2.80	2.80
2.69	2.68	2.69
2.45	2.46	2.46
2.40	2.40	2.40

- RAE, A. W. J. M., THOMPSON, D. P., PIPKIN, N. J., and JACK, K. H. *The Structure of Yttrium Silicon Oxynitride and its Role in the Hot-Pressing of Silicon Nitride with Yttria Additions* in Special Ceramics 6, P. Popper, ed., British Ceramic Research Association, Stoke-on-Trent, England, 1975, p. 347-360.
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- RAE, A. W. J. M., THOMPSON, D. P., and JACK, K. H. *The Role of Additives in the Densification of Nitrogen Ceramics in Ceramics for High Performance Applications - II*, J. J. Burke, E. M. Lenoe, and R. N. Katz, ed., Army Materials Technology Conference, Newport, Rhode Island, 1977, p. 1039-1067.

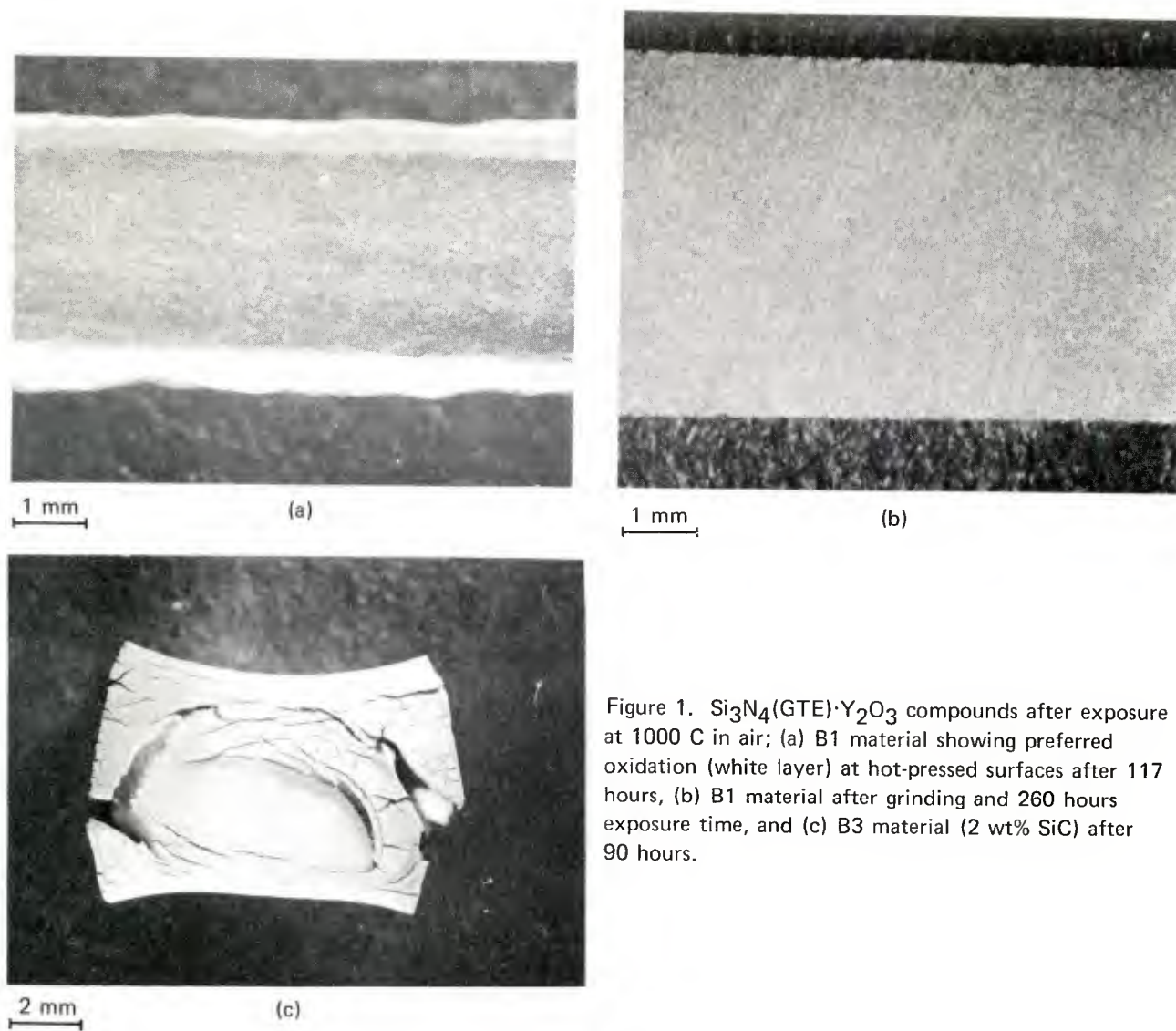


Figure 1. $\text{Si}_3\text{N}_4(\text{GTE}) \cdot \text{Y}_2\text{O}_3$ compounds after exposure at 1000 C in air; (a) B1 material showing preferred oxidation (white layer) at hot-pressed surfaces after 117 hours, (b) B1 material after grinding and 260 hours exposure time, and (c) B3 material (2 wt% SiC) after 90 hours.

the oxidation resistance of these materials. This assumption is supported by the fact that Starck Si_3N_4 contains 0.6 wt% carbon, Table 1, and material A always exhibits cracking at 1000 C. In contrast, GTE Si_3N_4 powder is essentially carbon-free.

In order to verify the apparent influence of carbon on the intermediate temperature degradation of the $\text{Y}_2\text{O}_3 \cdot \text{Si}_3\text{N}_4$ compacts, additional samples (using GTE powder) were individually doped with 2 wt% carbon and 2 wt% SiC, samples labelled B2 and B3. Specimens were machined from each hot-pressed compact. All surfaces were ground. After annealing at 1000 C in air, all carbon-containing materials exhibited severe cracking. Figure 1c shows a piece of B3 material after 90 hours at 1000 C.

The weight gain of the four investigated materials as a function of oxidation time at 1000 C is shown in Figure 2. The undoped material (B1) shows good oxidation resistance with parabolic kinetics. No cracking was observed. In contrast,

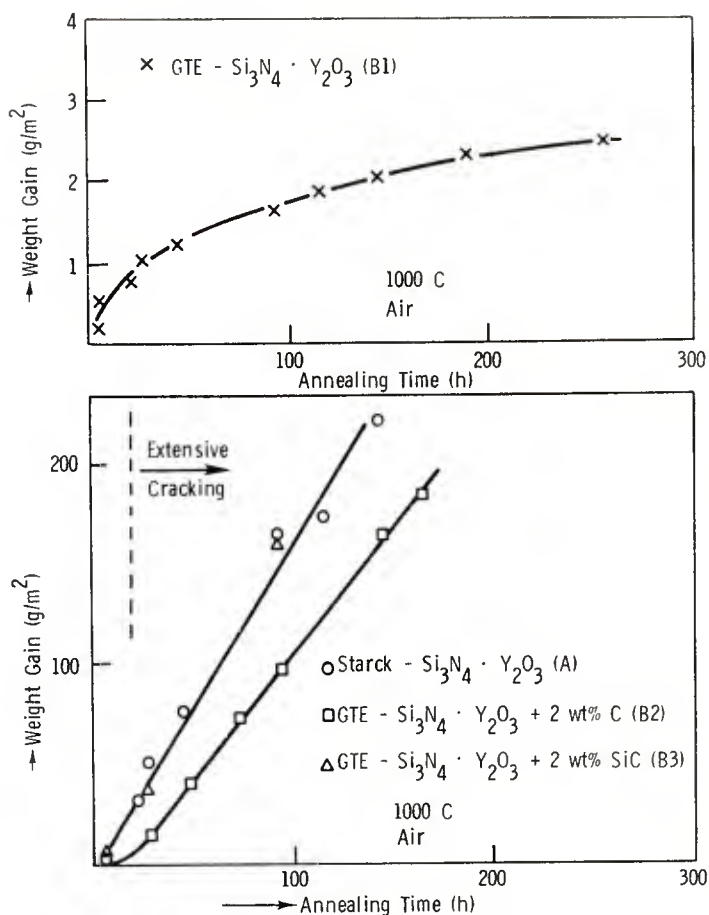


Figure 2. Weight gain of different Si₃N₄·Y₂O₃ compounds at 1000 C in air as a function of exposure time.

all carbon-containing materials exhibited highly unstable behavior. The weight gains were approximately two orders of magnitude higher than with the undoped specimens, and severe cracking occurred after only short annealing times. The weight gain of these materials is based on the surface area of samples prior to testing. Since during oxidation new surface area is produced by cracking, the data in the figure should be considered only as apparent oxidation kinetics data. The carbon-containing material (B2) initially showed a slight weight loss, probably due to oxidation of unreacted graphite particles in the sample.

Figure 3 shows the integrated weight gain of A, B1, and B2 materials as a function of temperature. Holding time at each temperature was 24 hours. Measurable weight gain is noted as low as 800 C for the carbon-doped materials, and the reaction is most severe between 900 and 1100 C. Again, the carbon-free material (B1) exhibits much superior oxidation resistance. These results clearly indicate the highly deleterious effect of carbon impurity on the thermal degradation of hot-pressed Si₃N₄·Y₂O₃ compounds, which are principally N-melilite.

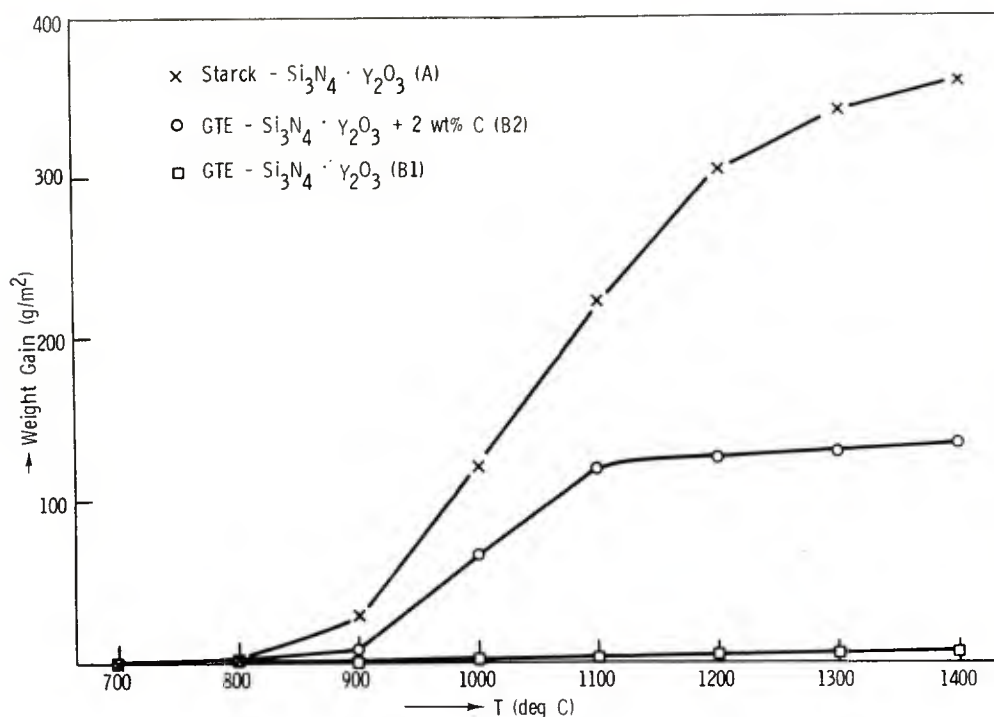


Figure 3. Integrated weight gain of three $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ compounds as a function of temperature. Holding time at each data point 24 hours.

The significance of these results may be considered from various aspects of Si_3N_4 processing technology. First, it is the state-of-the-art to hot press Si_3N_4 in graphite dies. Reactions between the hot-pressed compound and graphite occur which may alter material properties, particularly for the $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$ system. Also, prior to hot pressing, milling with media such as WC can introduce considerable carbide impurity into the starting powder when large numbers of WC balls are used for long milling times. Although significant quantities of WC were not intentionally added into the starting powder in this study, WC will form WSi_2 at high hot-pressing temperatures thereby introducing carbon into the system.

As recently shown by Lou et al.,⁶ commercial hot-pressed Si_3N_4 contains a rather high amount of carbide phases. Starting powders of Si_3N_4 produced by the nitridation of silicon may contain carbon as a process impurity, whereas powders produced by vapor phase reactions, e.g., $\text{SiCl}_4 + \text{NH}_3$, will be essentially carbon-free.

It is apparent that there is a significant effect of carbon on the crystal chemistry and phase equilibria in the Si-Y-N-O system. The role of carbon in

6. LOU, L. K. V., MITCHELL, T. E., and HEUER, A. H. *Impurity Phases in Hot-Pressed Si_3N_4* . J. Amer. Ceram. Soc., v. 61, no. 9-10, 1978, p. 392-396.

this system is essentially unknown. Using the simple method of weight gain measurements in air it has been demonstrated that the addition of small amounts of carbon to $Y_2O_3 \cdot Si_3N_4$ compounds changes their behavior in air from "stable" to "unstable."

The necessity to further define the role of carbon in the Si-Y-N-O system has been demonstrated. Better understanding of its behavior may have an important influence on the development of high-strength, high-temperature nitrogen ceramics.

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2	ATTN: DRXMR-PL
1	DRXMR-WD
2	Authors

Army Materials and Mechanics Research Center,
Watertown, Massachusetts 02172
CARBON IMPURITY EFFECT ON THE THERMAL
DEGRADATION OF A $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$ CERAMIC -
Heinrich Knoch and George E. Gazza

Technical Report AMMRC TR 79-27, May 1979, 8 pp -
illus-tables, D/A Project 1L162105AH84,
AMCMS Code 612105.H840011

The oxidation behavior of hot-pressed $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$ compounds (principally $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$, N-mellilite) was studied in air between 700 C and 1400 C. It is shown that carbon impurity strongly influences the thermal degradation of these compounds. Oxidation of carbon-containing materials in air at 1000 C leads to high weight gains, severe cracking, and complete destruction of the samples. Nominally carbon-free materials show good oxidation resistance without cracking.

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Key Words

Ceramics
Silicon nitride
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