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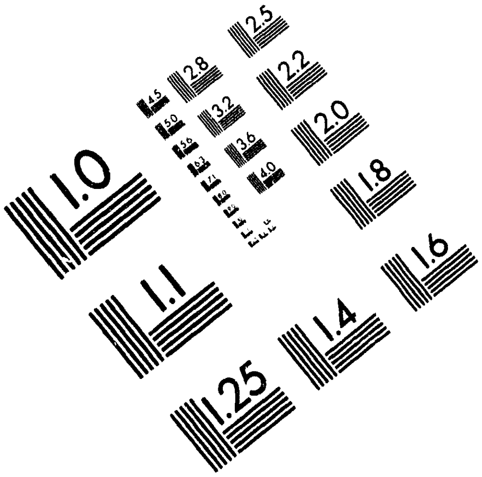

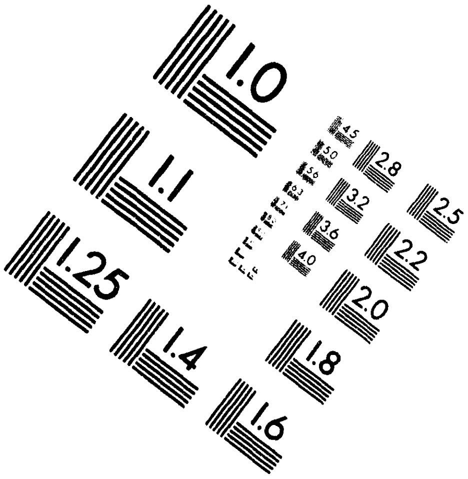

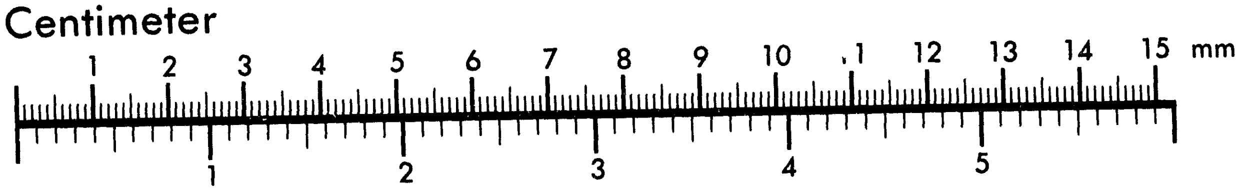

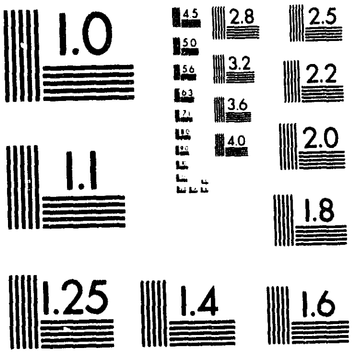

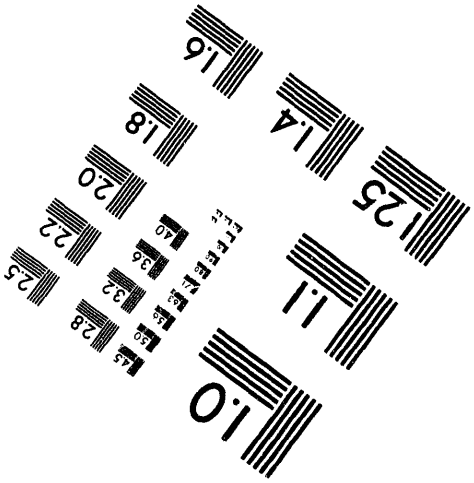

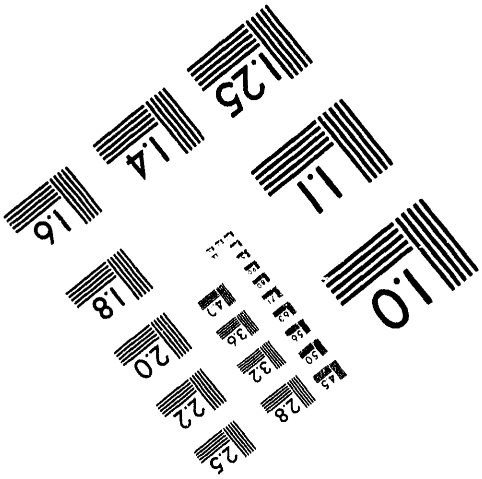

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ENHANCEMENT OF CREEP RESISTANCE OF A SINTERED Si_3N_4 CERAMIC BY MICROWAVE ANNEALING

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ABSTRACT

Creep behavior was investigated for a sintered Si_3N_4 ceramic following microwave annealing at 1400 and 1500°C for 20 h. Results of creep tests at 1200°C showed that microwave annealing can enhance the creep resistance in terms of lowering creep rate and, hence, extending creep rupture life, but the 1400°C annealing was most effective. X-ray diffraction analyses showed that the crystalline second phase was transformed as a result of microwave annealing from single phase $Y_{10}Al_2Si_3O_{18}N_4$ to several different phases. The situation was further complicated by evolution of additional phases during high temperature creep experiments.

1. Introduction

Although hot-isostatically pressed (HIPed) Si_3N_4 advanced ceramic materials offer outstanding mechanical properties for high temperature structural applications, prevalent uses of the materials in practical engineering have been hindered by the high cost. On the other hand, sintered Si_3N_4 ceramics can be produced at a much more affordable cost compared to HIPed types because of the ease of manufacturing processes. However, the mechanical properties and reliability of sintered ceramics are, in general, somewhat inferior compared to those of HIPed ceramics due to differences in composition and inherent process defects. An attempt is made here to show that the creep resistance of the low-cost ceramic materials can be enhanced by post-sintered microwave annealing so that improved ceramic materials can be produced cost-effectively and eventually should become economically viable.

2. Experimental Details

A commercial grade of sintered Si_3N_4 was used, known as SNW-1000 furnished by WESGO, Inc. of Belmont, California. The material contains 12-13 wt% Y_2O_3 and 2-3 wt% Al_2O_3 as densification aids. Specimen blanks, 20-mm dia by 180-mm length, were annealed in a microwave oven. Six blanks were annealed at 1400°C for 20 h in two batches (specimens MA-1 to -3 / MA-4 to -6) and four at 1500°C for 20 h in a single batch (MA-7 to -10), all in 0.1 MPa nitrogen. No more than four samples were annealed in a batch to avoid heating bias. A molybdenum-sheathed thermocouple was used to monitor and control the annealing temperature. Buttonhead tensile specimens having a uniform gage section of 6.3-mm diam by 25-mm length were tested in air. Experimental details are referred to elsewhere[1].

3. Test Results and Discussion

All the annealed specimens were tested at 1200°C mostly under constant loads until rupture occurred, except specimen MA-2 which was tested with a step-up load history (not shown) because of the outstanding creep resistance exhibited by this particular specimen. The test load for specimen MA-2 was increased successively in steps after completing about 1000 h of testing at each load step until specimen rupture occurred. Creep curves (broken lines) of specimens annealed at 1400 and 1500°C are plotted, respectively, in Figs. 1 and 2 for comparison with those (solid lines) obtained by North Carolina A & T State University[2] for the as-sintered material.

Examination of Figs. 1 and 2 indicates that microwave annealing was, generally speaking, effective for enhancement of creep resistance in terms of lowering creep rate and, hence, extending creep rupture life. The creep resistance was enhanced more effectively by annealing at 1400°C compared to that at 1500°C. However, inconsistencies in creep behavior within each of the annealing temperature groups are clearly discernible. In Fig. 1, contrasting creep behavior is illustrated by specimens MA-5 showing high creep rate and short creep life whereas MA-2 showing low creep rate and long creep life. Although both specimens were supposedly annealed at the same temperature, the actual annealing environments could have been different since they were annealed independently in two different batches. Therefore, the contradictory creep behavior may be due to the variation in actual annealing temperatures. Another inconsistency is that specimen MA-2 tested at 160 MPa produced a lower creep rate compared to those for specimens MA-1 and MA-6 tested at a lower stress of 140 MPa. Figure 2 shows that annealing at 1500°C somewhat enhanced the creep resistance but was much less effective compared to annealing at 1400°C. However, creep behavior was more orderly and predictable for the specimens annealed at 1500°C. Some annealed specimens such as MA-5, -7, and -8 showed inferior creep rupture lives compared to those for the unannealed specimens tested under the same applied stresses.

Some plausible reasons for the inconsistencies discussed above can be cited. Since these specimen blanks were annealed at an earlier date while the developing

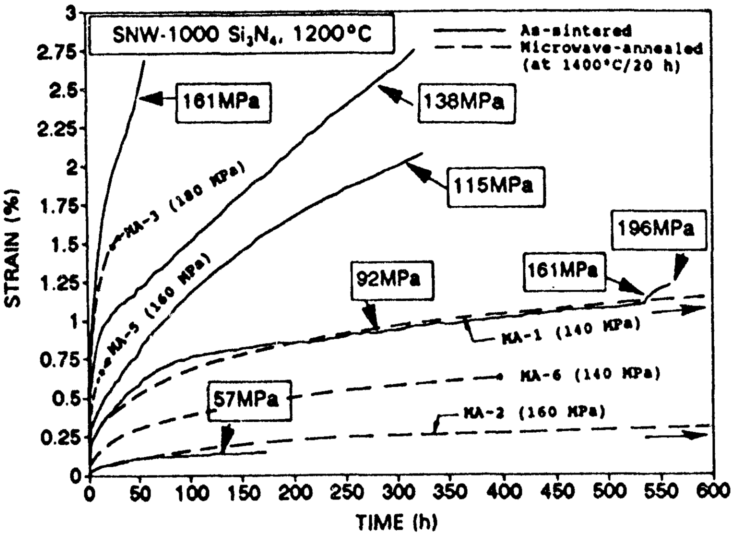

Figure 1. Comparisons of creep curves for unannealed and annealed specimens (at 1400°C for 20 h) tested at 1200°C under applied stresses as indicated. Annealing at 1400°C appeared to have enhanced the creep resistance of this material. However, the inconsistency in creep behavior exhibited by specimen MA-2 and MA-5, both under the same stress, remained to be resolved.

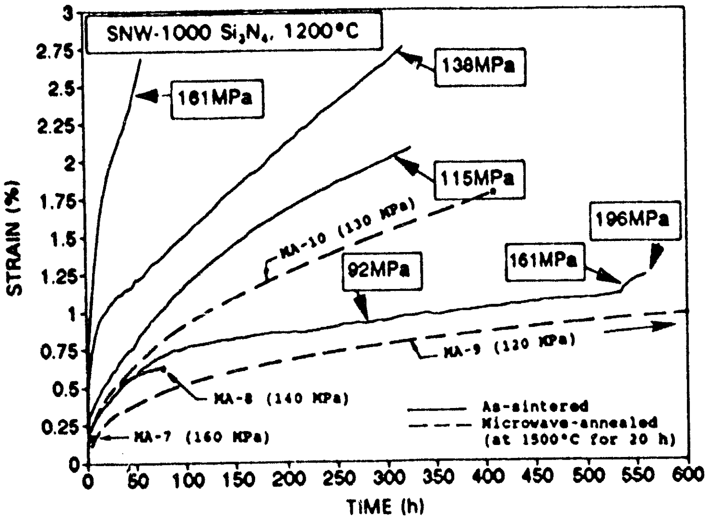

Figure 2. Comparisons of creep curves for unannealed and annealed specimens (at 1500°C for 20 h) tested at 1200°C under applied stresses as indicated. Annealing at 1500°C enhanced the creep resistance also, but was less effective compared to that at 1400°C. However, inferior creep rupture lives exhibited by specimens MA-7 and -8 showed otherwise.

technology was still in its infancy, some degree of nonuniformity in annealing was not entirely unexpected. Furthermore, the annealing was done in three separate batches; therefore, batch-to-batch variations in annealing can not be ruled out. Another reason is that heating may bias toward one or two specimens when multiple numbers of specimen blanks are heated together, due to the nature of microwave heating. The heating bias can be avoided if specimen blanks are annealed individually in a highly thermal conductive container to dissipate the localized heat.

4. X-Ray Diffraction Analysis

X-ray diffraction analyses were performed on specimens MA-1 to -3 because they were annealed in the same batch yet showed diversely different characteristics in creep resistance. As a result of microwave annealing alone, the crystalline second phase was found to have transformed from $Y_{10}Al_2Si_3O_{18}N_4$ (JCPDS #32-1426), as the only phase in the as-sintered material, to several different phases as shown in Table 1. Moreover, the new phases in each of the annealed specimens were not the same from one specimen to the next, indicating that the microwave process was not reproducible or consistent within the batch. Following high temperature creep testing, additional phases were found, as shown in Table 1. Note that the same phases were found in specimens MA-1 and -2 but MA-3 showed different phases in the fractured samples.

Direct correlations between creep behavior or creep rate and each of the crystalline minor phases found in the annealed and tested specimens remain unclear at this time due to the complexity of the phase transformations occurring in the minor phases. However, the differences in creep rate or creep life among the three specimens can be inferred from the composition of the minor phases as a group presented in the annealed and tested specimens. The similarity in phase composition for both MA-1 and -2 may account for the low creep rate, while the difference in phase compositions may explain why specimen MA-2 exhibited superior creep resistance compared to MA-1. While the lack of O-Apatite phase differentiates MA-3 from MA-1, the high creep resistance exhibited by MA-2 may be due to the presence of YAG.

Table 1. Summary of X-ray diffraction results

	Minor phases	
Specimen	After microwave annealing	After creep testing
Standard	$Y_{10}Al_2Si_3O_{18}N_4$ 32-1426	
MA-1	Y_2SiO_5 (36-1476) $YSiO_2N$ (36-1462) Wollastonite $Y_{4.67}(SiO_4)_3O$ (30-1457) O-Apatite	Y_2SiO_5 $YSiO_2N$ (36-1462) $Y_{4.67}(SiO_4)_3O$ (30-1457) $Y_2Si_3N_4O_3$ (28-1457) Mellilite
MA-2	Y_2SiO_5 (36-1476) $YSiO_2N$ (36-1462) Wollastonite $Al_5Y_3O_{12}$ (33-40) YAG	Same 4 phases as above
MA-3	Y_2SiO_5 (36-1476) $YSiO_2N$ (36-1462) Wollastonite	No new phases

Notes: (1) β-Si_3N_4 was the major phase; no α-Si_3N_4 was detected.
(2) Some O-Apatite may be present in MA-2 and -3 but in much smaller quantities than in MA-1.

5. Summary

Creep resistance of a sintered Si_3N_4 ceramic can be enhanced by microwave annealing but more effectively at 1400°C than at 1500°C. However, the optimum annealing temperature and duration remain to be investigated. Inconsistencies in creep behavior observed may be attributed to the deficiency in the current microwave annealing process to produce a consistent optimum composition of the crystalline second phases. Extended studies are currently ongoing.

6 Acknowledgement

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