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SINTERING MODEL FOR SiC_w/Si_3N_4 COMPOSITES

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ABSTRACT

This paper presents a model which suggests that it should be possible to pressureless sinter a SiC_w/Si_3N_4 composite to theoretical density. Prior failure to achieve complete densification by sintering is attributed to the use of compositions which result in a "glass deficit." There is one basic premise for this model. The ratio of glass amount to surface area of nonglass constituents must be the same for both composite and sinterable monolithic Si_3N_4. This model suggests that whisker and grain sizes and whisker loading influence the glass amount necessary for successful sintering of composites. According to the model, a large glass amount will be necessary for successful sintering of these composites. However, grain boundary thicknesses in the composite will be less than those in the analogous monolithic material. This suggests that good high temperature strength may still be attained. A recent report supports the predictions of the model.

NOMENCLATURE

A_{gC} area percent glass in WTC

A_{gM} area percent glass in monolithic Si_3N_4

A_{mC} area percent Si_3N_4 matrix in WTC

A_{mM} area percent Si_3N_4 grains in monolithic Si_3N_4

A_{wC} area percent whiskers in WTC

gb_t grain boundary thickness(es)

L_C total perimeter of nonglass constituents per unit area in WTC

L_M total perimeter of Si_3N_4 grains per unit area in monolithic Si_3N_4

m side of hexagonal grain cross section = ½ grain size

P_m perimeter/area ratio of Si_3N_4 grain

P_w perimeter/area ratio of SiC whisker

r radius of whisker cross section

SiC_w silicon carbide whisker(s)

Si_3N_4 silicon nitride

V_C ratio of area percent glass/total perimeter of nonglass constituents in WTC

V_M ratio of area percent glass/total perimeter of Si_3N_4 grains in monolithic Si_3N_4

WTC whisker toughened composite(s)

INTRODUCTION

Addition of silicon carbide whiskers (SiC_w) to a Si_3N_4 matrix should result in ceramic composites with good toughness, strength, and creep resistance. The properties of several ceramic matrix materials improved with SiC_w addition.[1-5] However, these materials were densified by either hot pressing or "canned" HIP'ing and thus have only limited application.

For complex shaped components densification by sintering or containerless HIP'ing is the more desirable approach. These processes should produce a more isotropic whisker toughened composite (WTC) material than hot pressing. Recently, Tiegs, and Becher[6] demonstrated improved flexural strength and toughness with pressureless sintered SiC_w/Al_2O_3. However, addition of SiC_w to Si_3N_4 makes it difficult to obtain high density by pressureless sintering.[7] Compositions that provide excellent sinterable monolithic Si_3N_4 do not completely sinter when used as matrix material in WTC. In WTC, such matrix compositions can only be densified by pressure approaches.[8,9]

The glassy grain boundary phase influences the rates of dissolution, diffusion, and redeposition of Si_3N_4 during liquid phase sintering.[10,11] During sintering of WTC, an adequate glass amount is necessary for the rearrangement of both SiC_w and Si_3N_4 grains. Lange[12] and Scherer[13] suggest that whiskers in WTC form a touching network at a relatively low whisker loading. The network would inhibit densification of the composite. Bordia and Raj[14] propose that unrelaxed shear stresses and poor wetting of the whiskers during sintering reduce the sintering rate.

This paper suggests that another possible reason previous attempts to sinter SiC_w/Si_3N_4 composites failed was because the selected composition led to a "glass deficit." This paper presents a simple two-dimensional model which predicts the glass amount required to sinter SiC_w/Si_3N_4 composites to theoretical density. It predicts a larger glass requirement for sintering a WTC than an analogous monolithic material. The extra glass would improve the wetting of the whiskers, aid in pore removal, and provide the whiskers with a medium for rearrangement during sintering. Additional glass may also allow a higher whisker loading without the nonsintering whiskers forming a constraining network. This paper suggests that this glass amount should not be detrimental to the high temperature properties of the material.

In addition, this model provides guidance in producing a WTC with the same grain size, grain boundary thickness (gb_t), and glass composition as a good monolithic Si_3N_4. Comparison of these two materials will directly clarify the effect of whisker addition on the properties of Si_3N_4.

SINTERABLE COMPOSITE MODEL

Basis

This section defines the "glass deficit" condition. It shows the effect of a "glass deficit" on sintering of monolithic Si_3N_4 and presents the reason for developing the model.

One method for understanding the important effects caused by whisker additions is to compare idealized microstructures for the monolithic and WTC ceramics. Figure 1 is an idealized two-dimensional representation of a monolithic Si_3N_4 microstructure. Si_3N_4 grains are hexagonal and monosized. They are in an hexagonal array and their gb_t is uniform. For convenience, the inscribed hexagonal area is the unit cell. The small, crosshatched region is a "grain boundary glass part." There are 3 grains and 9 glass parts per unit cell.

Now consider the current practice of simply replacing a portion of the monolithic starting composition with SiC_w. As an example, the following describes the formation of a 33 vol % WTC. Remove one Si_3N_4 grain and its associated glass from the idealized microstructure shown in Fig. 1. Substitute one hexagonal SiC_w to make a 33 vol % WTC. Assume the hexagonal whisker for ease of visualization. A "glass deficit" occurs as shown in Fig. 2. There are now 1 whisker, 2 grains, and 6 glass parts per unit cell (same definition as in Fig. 1). Thus, the volume of glass decreased by 1/3. Clearly, this reduction in glass volume will result in a decrease in average gb_t and in the ease of sintering.

In this paper, the baseline sintered monolithic Si_3N_4 is the NASA 6Y[15] which contains 6 wt % Y_2O_3. The glassy grain boundary phase occupies 13.11 vol % of the material. Addition of 33 vol % SiCw to a NASA 6Y matrix reduces the glassy grain boundary amount in the composite to 8.7 vol %. Figure 3 shows the density of a sintered monolithic Si_3N_4 material as a function of glass amount. The density reduces considerably as the glass amount decreases. The decreased sinterability of the "glass deficit" monolithic material is in part due to diminished dissolution and grain rearrangement during sintering.[16] Similarly, "glass deficit" conditions should prevent attainment of theoretical density during sintering of a WTC. Therefore, sintering a WTC requires modification of the starting composition.

To sinter a WTC the glass amount must increase and the Si_3N_4 amount must decrease in order to overcome the "glass deficit." Experimental determination of the proper composition of Si_3N_4 powder and glass formers for sintering a WTC to full density is possible. However, it is desirable to be able to calculate the sinterable composition, especially when powder, whisker, and additive characteristics change. The remainder of the paper will develop a simple two-dimensional model for selecting a sinterable SiC_w/Si_3N_4 composite composition.

Definitions

There is one basic premise for the sinterable SiC_w/Si_3N_4 composites model. The WTC and sinterable monolithic Si_3N_4 materials must both contain the same ratio of glass amount to surface area of nonglass constituents. Using the same glass composition in the WTC should then provide the same grain boundary characteristics as in the monolithic.

The model includes four factors which influence the gb_t. They are Si_3N_4 grain size, SiC_w diameter, volume percent SiC_w loading, and volume percent glass in the monolithic. For example, Fig. 4 shows the effect of grain size on the calculated gb_t for a monolithic Si_3N_4. The calculations use the monolithic microstructure depicted in Fig. 1 and assume the NASA 6Y composition. These calculated values and those used in the remainder of the paper are about an order of magnitude greater than measured gb_t. This is because of the idealized two-dimensional representation of monolithic Si_3N_4 used for these calculations. Use of a three-dimensional representation with a grain geometry that allows for glassy triple points would decrease the calculated gb_t.

For the sintering model, Si_3N_4 grains are parallel, monosized hexagonal prisms. The SiC_w are parallel, monosized right cylinders. In two-dimensions, the grain cross sections are hexagons and the whisker cross sections are circles. The hexagons have 6 equal sides of length m and the circles have radii of length r. The ratio of volume percent glass to surface area of nonglass constituents becomes the ratio of area percent glass to perimeter of nonglass constituents. A_g, A_m, and A_w are the area percent glass, area percent Si_3N_4 grains, and area percent SiC_w, respectively.

For circular whiskers, the ratio of perimeter to area, P_w, is equal to $2\pi r/\pi r^2$ or $2/r$. For the hexagonal Si_3N_4 grains, the similar ratio, P_m, is equal to $6m/[3(3)^{1/2}m^2/2]$ or $4/(3^{1/2}m)$. Thus, the total perimeter of the grains per unit area in the monolithic material, L_M, is equal to $P_m \times A_{mM}$. For the monolithic, the ratio of area percent glass to total perimeter of nonglass constituents, V_M, is equal to A_{gM}/L_M. In the WTC, L_C, the total perimeter per unit area, is equal to $P_m \times A_{mC} + P_w \times A_{wC}$. These products are the total perimeters of the grains and whiskers, respectively. For the WTC, V_C is equal to A_{gC}/L_C.

Model

The ratio of area percent glass to total perimeter of nonglass constituents must be the same in the WTC as the monolithic Si_3N_4. This is the basic premise of the model. Thus, V_M is equal to V_C or $A_{gC}/L_C = A_{gM}/L_M$, and

$$A_{gC}/(P_w \times A_{wC} + P_m \times A_{mC}) = A_{gM}/(P_m \times A_{mM}) \quad (1)$$

Since

$$A_{gC} + A_{mC} + A_{wC} = 100 \text{ percent},$$

then

$$A_{mC} = 100 - A_{gC} - A_{wC} \quad (2)$$

By substituting Eq. (2) for A_{mC} in Eq. (1) and rearranging to solve for A_{gC},

$$A_{gC} = (A_{gM}/100) \times [(P_w/P_m) - 1] \times A_{wC} + A_{gM}$$

Since

$$P_w = 2/r \quad \text{and} \quad P_m = 4/(3^{1/2} \times m),$$

then

$$A_{gC} = (A_{gM}/100) \times \{[(3^{1/2} \times m)/(2 \times r)] - 1\} \times A_{wC} + A_{gM} \quad (3)$$

Thus, knowledge of only four variables is necessary to solve Eq. (3). The four variables are sintered Si_3N_4 grain size, SiC_w diameter, volume percent SiC_w loading, and volume percent glass in the monolithic.

For example, the NASA 6Y monolithic Si_3N_4 has 13.11 vol % glass, and a 2 μm average grain size.[15] So, $A_{gM} = 13.11$ and $m = 1$. To make a WTC, assume that the SiC_w loading is 30 vol % and the whiskers have a 1 μm diameter. Then, $A_{wC} = 30$ and $r = 0.5$. Applying Eq. (3),

$$A_{gC} = (13.11/100) \times \{[(3^{1/2} \times 1)/(2 \times 0.5)] - 1\} \times 30 + 13.11 = 15.99$$

Thus, the composite composition is 15.99 vol % glass, 54.01 vol % Si_3N_4, and 30 vol % SiC_w. Therefore, more glass is necessary to form this composite than the monolithic with the same ratio of area percent glass to perimeter of nonglass constituents (13.11 vol % in the monolithic).

MODEL PREDICTIONS AND DISCUSSION

Table I provides the calculated A_{mC} and A_{gC} values for WTC with 30 vol % SiC_w. The table lists values for a series of whisker diameters which encompass the range of commercially available whiskers. There are separate values for 1, 2, and 3 μm Si_3N_4 grain sizes. There is also a list of calculated gb_t. The calculations assume that the whiskers and grains have the same ratio of area percent glass to total perimeter. These values compare with calculated monolithic gb_t of 63, 126, and 189 nm for 1, 2, and 3 μm grain sizes, respectively.

The model shows that the whisker diameter strongly influences the composition needed to sinter to full density. As shown in Fig. 5, the glass requirement for successful sintering increases as the whisker diameter decreases. Similarly, for a constant whisker diameter less than the grain size, the glass requirement increases as the grain size increases. These observations are important because of the tendency to substitute different whiskers from different manufacturers into the same matrix composition. To compare different whiskers directly, modification of the matrix composition is necessary for each WTC.

Figure 6 shows that for a fixed grain size, as the volume percent SiC_w increases, the volume percent glass required for sintering increases. This is true as long as the grain size exceeds the whisker diameter. In such cases, more than 1 whisker is necessary to replace one grain. The smaller whiskers require more glass than the grains they replace to maintain the same ratio of area percent glass to total perimeter. However, the glass requirement diminishes when the grain size is less than the whisker diameter. Therefore, the model shows that the volume percent SiCw influences the composition of the matrix phase.

Table II also shows the increase in volume percent glass required as the whisker load increases. There is a nominal increase in glass amount from 13.11 to 15.99 vol % as the volume percent SiC_w increases from 0 to 30. However that increase is from 13.11 to 22.84 vol % glass in the matrix. In a monolithic Si_3N_4 that increase in glass would result in reduced strength at high temperature as the glass phase softens.[17] However, even though the glass content in the WTC matrix increases by 74.2 percent, the calculated average gb_t decreases slightly. Therefore, this suggests that high temperature properties should not decrease more than the monolithic material despite the large increase in glass amount.

Figure 7 is a simplified ternary phase diagram for the Si_3N_4-SiO_2-Y_2O_3 system. In the lower left hand corner is the NASA 6Y monolithic composition. The compositions lying on the dashed line through that point have a constant

SiO_2/Y_2O_3 ratio. After calculating the glass amount for the composite, select the additive amount so the composition of the matrix lies on that line. By this method the composition of the glassy grain boundary will be the same in the composite as in the comparable monolithic.

Recent literature supports the model. Figure 8 presents the data of Tamari, et al.[18] Converting from the reported mole percent sintering aids to volume percent glass made the data compatible with the model developed in this paper. The conversion assumed glass densities based on the rule of mixtures. The figure clearly shows that as the volume percent glass in a SiC_w/Si_3N_4 composite increases, the sintered density also increases. In that study, both 10 and 20 wt % (11.44 and 23.81 vol %) SiC_w/Si_3N_4 composites sintered to 100 percent density. A 30 wt % (35.61 vol %) composite did not sinter to full density even with 24 vol % glass. Those composite systems contained 0.5 μm diameter whiskers. By assuming a 2.0 μm grain size and a 15.43 vol % monolithic glass amount, the model offers a possible explanation. In Fig. 9, the 10 and 20 wt % composites which sintered to full density fall on the line predicted by the model. The 30 wt % composite falls below the model line suggesting that not enough glass was available for sintering in that composite.

CONCLUSIONS

Presented in this paper is a simple two-dimensional model which suggests that pressureless sintering of SiC_w/Si_3N_4 composites is possible provided there is an adequate glass amount. The model assumes parallel alignment of both Si_3N_4 grains and SiC_w. Only pressure consolidation or extrusion could cause this type of microstructure. However, this is a reasonable simplifying assumption for a first effort in determining sinterable compositions. The model also postulates that grain size, gb_t, and glassy grain boundary composition are the same in the WTC and the analogous monolithic Si_3N_4. This is a logical assumption since both the glass viscosity and amount determine the grain morphology during liquid-phase sintering. The effect of the first assumption and the validity of the second assumption are now under investigation.

If the simple two-dimensional model does not accurately predict the glass amount needed for sintering to full density, there are two considerations. First, the whisker packing is less efficient in an isotropic composite than in the assumed anisotropic alignment. Under isotropic conditions, the whiskers may indeed form a constraining network and inhibit complete densification. However, the successful pressureless sintering of SiC_w/Si_3N_4 composites by Tamari, et al.[18] suggests that this is not true. Further, the Tamari data shows that the two-dimensional model can predict compositions with enough glass to allow full densification. Second, the simple two-dimensional model may not adequately describe the complexities of WTC sintering. It may require a more rigorous two-dimensional model or a three-dimensional model. A three-dimensional model would consider whisker aspect ratio and three-dimensional random packing of both the whiskers and the grains. Other considerations for both the two-dimensional and three-dimensional models include a distribution of both grain sizes and whisker diameters.

Use of a three-dimensional model will likely increase the glass requirement because of the added surface area at the whisker ends. Thus, the glass amount predicted by the two-dimensional model is the minimum amount required, since more rigorous models are likely to predict a larger amount.

SUMMARY

This paper presents a simple two-dimensional model to predict the compositions of SiC_w/Si_3N_4 composites that should be sinterable to full density. Use of this model should make it possible to produce a WTC with the same grain size and grain boundary characteristics as a good monolithic Si_3N_4. This would allow a direct determination of the effect of whisker additions on the matrix material. The model shows that the glass amount needed to successfully sinter the WTC is more than that needed to sinter the monolithic. However, the excess glass does not increase the calculated gb_t. The relative sizes of the whiskers and grains strongly influence the necessary glass amount. The sinterable composition also depends on the size and loading of SiC_w. Recent data in the literature support the model.

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TABLE I. - MODEL MICROSTRUCTURAL PARAMETERS FOR 30 VOL % SiC_w/Si_3N_4

Whisker diameter, μm	$[A_{mC}]$ Si_3N_4, vol %	$[A_{gC}]$ glass, vol %	Calculated gb_t, nm
Grain size = 1 μm			
0.500	54.01	15.99	62.1
0.750	56.28	13.72	62.9
1.000	57.42	12.58	63.1
1.250	58.10	11.90	63.2
1.500	58.55	11.45	63.2
2.000	59.12	10.88	63.2
Grain size = 2 μm			
0.500	47.20	22.80	117.9
0.750	51.74	18.26	122.1
1.000	54.01	15.99	124.1
1.250	55.37	14.63	125.2
1.500	56.28	13.72	125.8
2.000	57.42	12.58	126.3
Grain size = 3 μm			
0.500	40.39	29.61	168.8
0.750	47.20	22.80	176.9
1.000	50.60	19.40	181.5
1.250	52.65	17.35	184.4
1.500	54.01	15.99	186.2
2.000	55.71	14.29	188.2

TABLE II. - MODEL SiC_w/Si_3N_4 FORMULATIONS

Volume percent whisker (A_{wC})	Calculated volume percent glass (A_{gC})	Calculated volume percent Si_3N_4 (A_{mC})	Volume percent glass in matrix only	Calculated average gb_t, nm
0	13.11	86.89	13.11	126.0
[a]10	14.07	75.93	15.63	125.2
[a]20	15.03	64.97	18.79	124.6
[a]30	15.99	54.01	22.84	124.1

[a]Assume: 2 μm grain size; 1 μm whisker diameter.

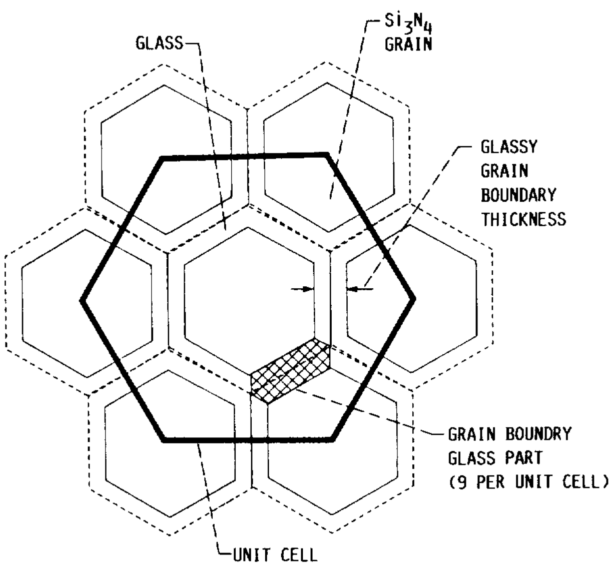

FIGURE 1. - IDEALIZED 2-D REPRESENTATION OF A MONOLITHIC Si_3N_4 MICROSTRUCTURE. ASSUMES: UNIFORM, HEXAGONAL Si_3N_4 GRAINS; HEXAGONAL PACKING; AND A UNIFORM COATING OF GLASS AROUND THE GRAINS.

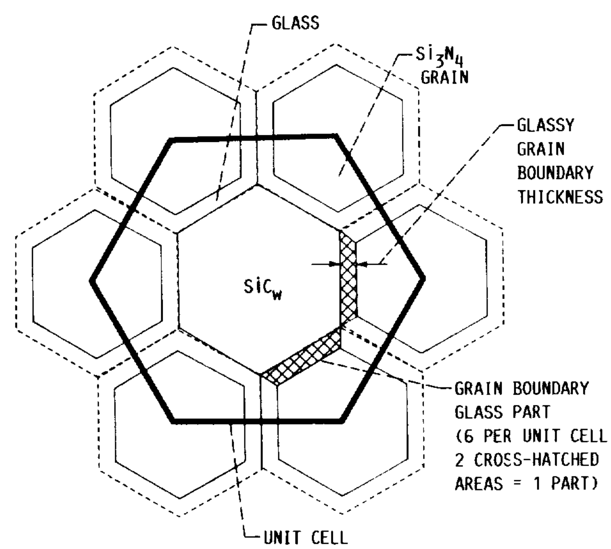

FIGURE 2. - IDEALIZED 2-D REPRESENTATION OF A 33 VOL % SiC_w/Si_3N_4 COMPOSITE MICROSTRUCTURE. ASSUMES: MATRIX COMPOSITION SAME AS MONOLITHIC (FIG. 1); HEXAGONAL SiC_w WITH SIZE EQUIVALENT TO SIZE OF Si_3N_4 GRAIN AND ITS ASSOCIATED GLASS.

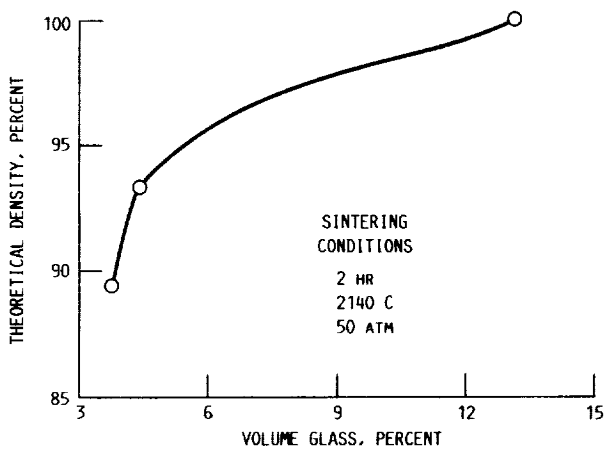

FIGURE 3. - DENSITY OF SINTERED MONOLITHIC Si_3N_4 MATERIAL CONTAINING Y_2O_3 AS A FUNCTION OF GLASS AMOUNT.

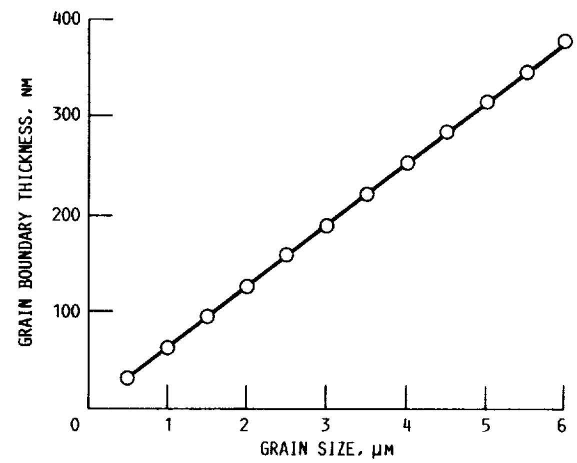

FIGURE 4. - CALCULATED AVERAGE GRAIN BOUNDARY THICKNESS AS A FUNCTION OF GRAIN SIZE FOR NASA 6Y MONOLITHIC Si_3N_4.

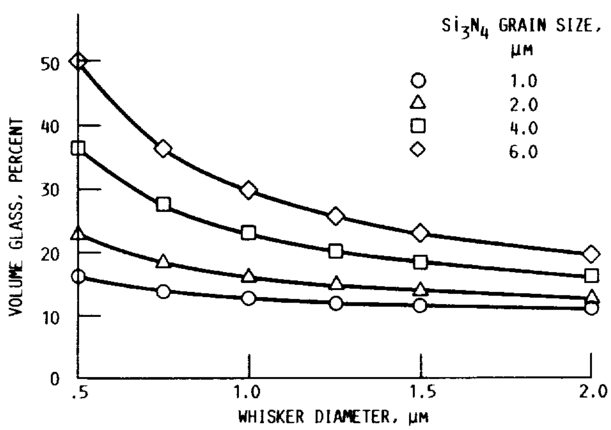

FIGURE 5. - CALCULATED VOLUME PERCENT GLASS NEEDED TO SINTER 30 VOL % SiC_w/Si_3N_4 COMPOSITES AS A FUNCTION OF WHISKER DIAMETER FOR VARIOUS AVERAGE GRAIN SIZES. ASSUMES NASA 6Y GLASS COMPOSITION.

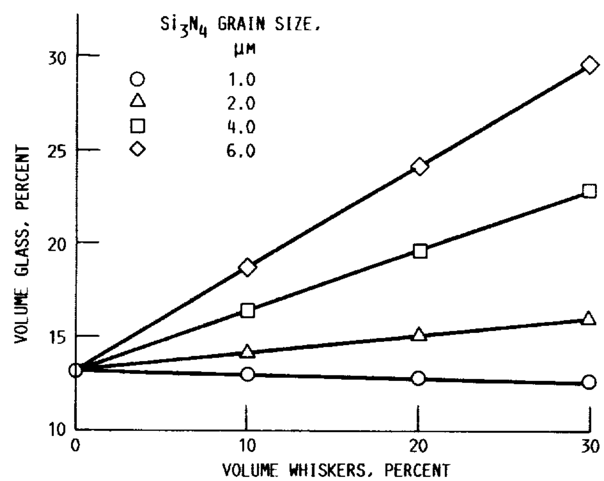

FIGURE 6. - CALCULATED VOLUME PERCENT GLASS NEEDED TO SINTER SiC_w/Si_3N_4 COMPOSITES AS A FUNCTION OF VOLUME PERCENT WHISKERS FOR VARIOUS AVERAGE GRAIN SIZES. ASSUMES AN AVERAGE WHISKER DIAMETER OF 1 μM AND NASA 6Y GLASS COMPOSITION.

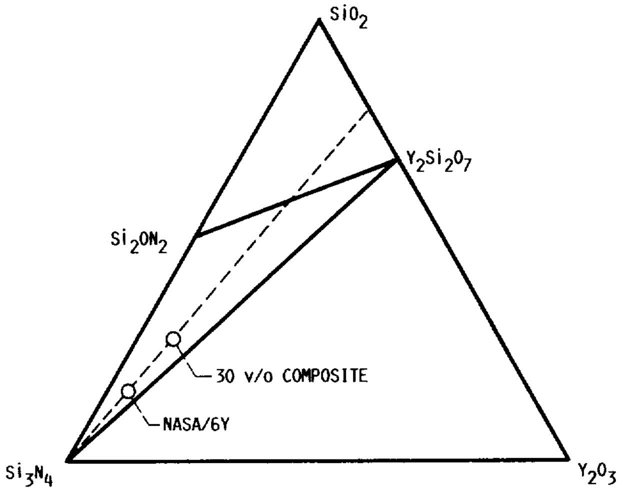

FIGURE 7. - SIMPLIFIED TERNARY PHASE DIAGRAM FOR THE Si_3N_4-SiO_2-Y_2O_3 SYSTEM. COMPOSITIONS WITH THE SAME SiO_2/Y_2O_3 RATIO LIE ON THE DASHED LINE. SHOWN ARE THE NASA 6Y MONOLITHIC COMPOSITION AND THE MATRIX COMPOSITION FOR A 30 VOL PERCENT SiC_w/Si_3N_4 COMPOSITION BASED ON THAT MONOLITHIC MATERIAL.

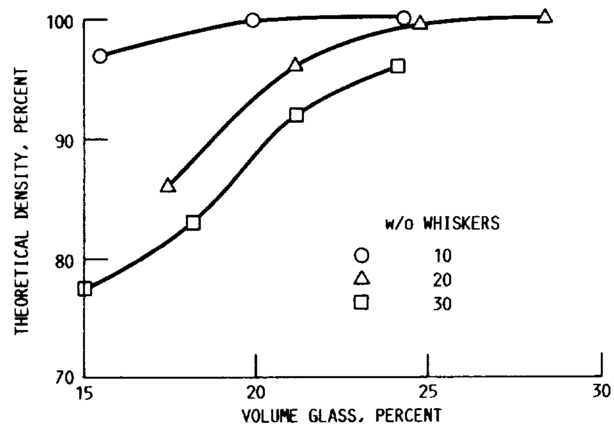

FIGURE 8. - PERCENT THEORETICAL DENSITY AS A FUNCTION OF VOLUME PERCENT GLASS FROM DATA OF TAMARI, ET AL.

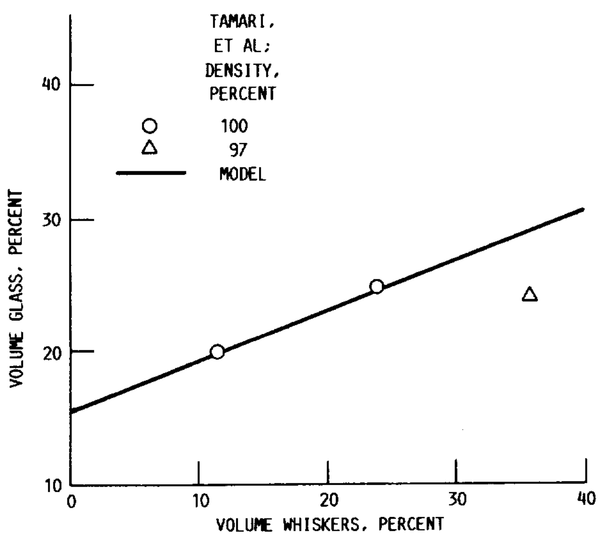

FIGURE 9. - COMPARISON OF TAMARI, ET AL [18] SINTERING DATA WITH THE CALCULATED GLASS AMOUNT NEEDED TO FULLY SINTER A SiC_w/Si_3N_4 COMPOSITE AS A FUNCTION OF WHISKER LOADING. TAMARI, ET AL. USED A 0.5 μM DIAMETER WHISKER. ASSUMES AN AVERAGE GRAIN SIZE OF 2.0 μM.

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16. Abstract

This paper presents a model which suggests that it should be possible to pressureless sinter a SiC_w/Si_3N_4 composite to theoretical density. Prior failure to achieve complete densification by sintering is attributed to the use of compositions which result in a ''glass deficit.'' There is one basic premise for this model. The ratio of glass amount to surface area of nonglass constituents must be the same for both composite and sinterable monolithic Si_3N_4. This model suggests that whisker and grain sizes and whisker loading influence the glass amount necessary for successful sintering of composites. According to the model, a large glass amount will be necessary for successful sintering of these composites. However, grain boundary thicknesses in the composite will be less than those in the analogous monlithic material. This suggests that good high temperature strength may still be attained. A recent report supports the predictions of the model.

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