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Thermal Expansion and Elastic Anisotropy in Single Crystal Al_2O_3 and SiC Reinforcements

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SUMMARY

In single crystal form, SiC and Al_2O_3 are attractive reinforcing components for high temperature composites. In this study, the axial coefficients of thermal expansion and single crystal elastic constants of SiC and Al_2O_3 were used to determine their coefficients of thermal expansion and Young's moduli as a function of crystallographic orientation and temperature. SiC and Al_2O_3 exhibit a strong variation of Young's modulus with orientation; however, their moduli and anisotropies are weak functions of temperature below 1000 °C. The coefficients of thermal expansion exhibit significant temperature dependence, and that of the non-cubic Al_2O_3 is also a function of crystallographic orientation.

INTRODUCTION

The use of ceramic fibers and whiskers as reinforcements in metal matrix (Ref. 1) and ceramic matrix (Ref. 2) composites has created a new class of advanced structural materials. Two reinforcing materials which are available as both single crystal whiskers and as continuous fibers (Refs. 3 and 4) are silicon carbide (SiC) (whiskers only) and aluminum oxide (Al_2O_3), the latter often being referred to as corundum or sapphire. In single crystal form, both are attractive reinforcing components for metal matrix and ceramic composites, as they exhibit high elastic moduli ($\cong 500$ GPa), and excellent high temperature thermal stability. Also, they display thermal and elastic anisotropy, and thus have potential to be manufactured in specific crystallographic orientations that will maximize the properties of composites using either as reinforcements. This paper describes the elastic moduli and thermal expansion values of these two single crystal materials, as well as their anisotropies and temperature dependencies.

CRYSTAL STRUCTURES AND WHISKER GROWTH

Silicon carbide (SiC) exists in a number of related crystal structures known as polytypes (Ref. 5), whereas aluminum oxide (Al_2O_3) only exists in a single trigonal structure (Ref. 6). The SiC structure of primary interest in this paper is the cubic (3C) beta structure. It resembles the structure of diamond, as it can be related to that of diamond by the substitution of Si atoms for half of the C atoms to yield the zinc blende structure. The cubic

(3C) beta structure of SiC is stable below about 2000 °C, but will transform to one of the hexagonal or rhombohedral polytypes with extended exposures above this temperature level (Ref. 7).

Single crystal whiskers of the cubic (3C) beta structure of SiC, F43m, can be readily produced in large quantities by the vapor-liquid-solid (VLS) process (Ref. 8). Another commercial scale growth technique utilizes rice hulls (Ref. 9). Typical whisker diameters are 0.1 to 10 μm with lengths of several millimeters to 1 cm. For the purpose of this paper, it is appropriate to note that most of the VLS whiskers have a <111> axial orientation, although axial <110> orientations have also been reported. The two are usually characterized by triangular <111> and elliptical <110> cross sections, respectively. Hexagonal structure SiC whiskers can also be grown by seeding with hexagonal grains for a form of epitaxial growth (Ref. 10). The anisotropy of hexagonal SiC is much more complicated than that of cubic SiC and it will not be addressed in this paper.

The crystal structure of Al₂O₃, R36, has been extensively addressed by Kronberg (Ref. 11). Although there is some ambiguity in the unit cell choice, the hexagonal structure will be applied in this paper. The numerous growth methods for whiskers and single crystals of Al₂O₃ have been reviewed by Belyaev (Ref. 12). Both the c-axis <0001> and the a-axis <2110> are common growth directions for Al₂O₃ whiskers, but any orientation may be grown by the Czochralski method of pulling from the melt. Recently, single crystal c-axis sapphire fibers, which are grown in continuous length by the edge defined film method (Ref. 13), are being evaluated for reinforcement of intermetallic and ceramic matrix composites (Ref. 14).

THERMAL EXPANSION

The formal description of thermal expansion is as that of any other symmetrical second rank tensor property (Ref. 15). The thermal expansion tensor relates the orientation and distance of a point from the origin in the crystal (a vector) after a temperature increase to the orientation and distance from the origin of the corresponding point before the temperature change. The thermal expansion coefficients will be designated as α_{ij} in this paper, their usual nomenclature.

As beta-SiC is cubic, it has only the single coefficient of thermal expansion, α_{11} , which specifies the expansion in all crystallographic directions. Cubic crystals are isotropic in thermal expansion. In Al₂O₃, however, two principal axial coefficients of thermal expansion, α_{11} and α_{33} , are necessary to describe the thermal expansion of the single crystal. For the hexagonal crystal structure, these are often written in the form:

$$\alpha_{\theta} = \alpha_{11}(1 - \cos^2\theta) + \alpha_{33}(\cos^2\theta) \quad (1a)$$

and

$$\alpha_{\theta} = \alpha_{11} + (\alpha_{33} - \alpha_{11}) \cos^2\theta \quad (1b)$$

where θ is the angle between the [0001] and the crystallographic direction of interest. In the hexagonal structure, the thermal expansion α_{11} is isotropic in the basal plane.

The principal axial thermal expansion coefficients of both beta-SiC (Ref. 16) and Al₂O₃ (Ref. 17) have been determined by X-ray diffraction methods from room temperature to 1000 °C. For SiC, α_{11} can be described by:

$$\alpha_{11} = 3.19 \times 10^{-6} + 3.60 \times 10^{-9}T - 1.68 \times 10^{-12}T^2 \quad (2)$$

where T is in °C and the units of α_{11} are (1/°C). For Al₂O₃, both α_{11} and α_{33} must be specified as a function of T. The published α_{11} and α_{33} values are:

$$\alpha_{11} = 6.34 \times 10^{-6} + 3.17 \times 10^{-9} T - 3.88 \times 10^{-14} T^2 \quad (3a)$$

and

$$\alpha_{33} = 7.12 \times 10^{-6} + 4.03 \times 10^{-9} T - 5.69 \times 10^{-14} T^2. \quad (3b)$$

These thermal expansion coefficients are illustrated from room temperature through 1000 °C in Fig. 1. In addition to the point that the expansion of Al₂O₃ exceeds that of SiC for all temperatures to 1000 °C, it is also apparent from Fig. 1 that the thermal expansion anisotropy of single crystal Al₂O₃ increases with increasing temperature. The effect of this thermal expansion anisotropy is to cause a shape change in the whisker, even when constrained by a surrounding matrix.

Figure 2 depicts the thermal expansion anisotropy of Al₂O₃ by illustrating α within the (10 $\bar{1}$ 0) plane that contains the [0001] and the [$\bar{1}$ 2 $\bar{1}$ 0], where a [10 $\bar{1}$ 0] is equal to a [$\bar{1}$ 2 $\bar{1}$ 0] within the basal plane. Both the anisotropy of the thermal expansion coefficient and its change with temperature are evident in this form of representation.

ELASTIC MODULI

The elastic behavior of crystalline solids is described by a fourth rank tensor, and as such is directionally dependent, even for cubic crystals (Ref. 18). This presents a more difficult descriptive process than previously described for the coefficients of thermal expansion. Kriz and Ledbetter (Ref. 19) applied a technique of elastic representation surfaces, but that procedure will not be followed in this presentation, as a more crystallographic perspective is desired to later interface with the earlier coefficient of thermal expansion presentation.

As both the single crystal elastic constants of beta SiC and Al₂O₃ are of interest, as well as their temperature dependencies, the two characteristics will be addressed in that sequence. For single crystal SiC in the cubic beta form, only three elastic constants are necessary. Either C₁₁, C₁₂, and C₄₄ or S₁₁, S₁₂, and S₄₄ will suffice. The C_{ij} of Tolpygo (Ref. 20) will be utilized in this presentation. The C_{ij} and S_{ij} are listed in Table I along with the room temperature elastic constants of single crystal Al₂O₃ as reported by Tefft (Ref. 21).

Tefft has also reported measurements of the temperature dependencies of the elastic constants and these are illustrated in Fig. 3. The elevated temperature C_{ij} and/or S_{ij} of beta SiC have never been measured; however, Li and Bradt (Ref. 22) have applied a self-consistent method utilizing polycrystalline SiC elevated temperature data to estimate the high temperature C_{ij} of beta SiC. The SiC stiffness values are illustrated in Fig. 4. Between room temperature and 1000 °C, the single crystal elastic stiffnesses of these two compounds do not reveal a large change of the elastic properties.

The simplest method of representing elastic anisotropy of crystalline solids is to consider the crystallographic or directional dependence of Young's modulus, E. It can be expressed as:

$$\frac{1}{E} = S'_{1111} \quad (4)$$

where:

$$S'_{1111} = l_{1m} l_{1\sigma} l_{1\theta} l_{1\phi} S_{m\sigma\theta\phi} \quad (5)$$

and the l_{ij} are the direction cosines. It is possible to plot either S'_{1111} as a function of orientation or to plot Young's modulus, E . Since a better physical perspective is usually gained from the Young's modulus rather than its reciprocal, E will be examined in this study.

Even though SiC is a cubic crystal, it is quite elastically anisotropic. In fact it is much more elastically anisotropic than Al_2O_3 . Since all cubic crystals have their maxima and minima values of Young's modulus in either the $\langle 100 \rangle$ or the $\langle 111 \rangle$, it is advantageous to plot $E = f\langle hkl \rangle$ for a crystallographic plane which contains both of those directions. The (110) is such a plane, as it contains both the [001] and the $[\bar{1}11]$. Figure 5 depicts $E = f\langle hkl \rangle$ on the (110) of SiC. The elastic anisotropy is obvious in this representation, as $E[001]$ is only about 275 GPa, but $E[\bar{1}11]$ is about 520 GPa. The $[\bar{1}10]$ is about 420 GPa. As previously noted the effect of temperature on these E values does not greatly alter the elastic anisotropy, but it does cause a decrease in each of the E values as illustrated in Fig. 6. It is apparent that if the Young's modulus along the length of the whisker is an important composite design factor, then $\langle 111 \rangle$ oriented whiskers should be employed for a maximum elastic modulus and $\langle 100 \rangle$ fibers used to achieve the minimum elastic modulus.

In Al_2O_3 , the room temperature Young's modulus is 465 GPa along the hexagonal c -[0001] axis and 430 GPa in all directions in the basal plane, either the $[\bar{1}2\bar{1}0]$ or $[10\bar{1}0]$. Figure 7 illustrates the Young's modulus of single crystal Al_2O_3 as it varies from the c -[0001] axis, to the basal plane, either the $[10\bar{1}0]$ or $[\bar{1}2\bar{1}0]$. It has a minimum of about 380 GPa at an orientation that is intermediate to the two orientations. It is apparent that Al_2O_3 is not very anisotropic in an elastic sense, certainly it is far less elastically anisotropic than SiC. This factor could be important in dictating a choice of the use of SiC or Al_2O_3 whiskers or fibers as the reinforcing constituent of a composite, especially where the transverse elastic properties are a consideration.

Figure 8 illustrates the temperature dependencies of Young's modulus along the two principal axes of Al_2O_3 . The temperature dependence is similar to that previously reported for SiC, as the Young's moduli decrease nearly linearly from room temperature through 1000 °C.

THERMOELASTIC STRESS INDEX

The calculation of the thermal and residual stresses about single crystal whiskers within either metallic or ceramic matrices is a problem of considerable interest (Ref. 23). Such stresses are central to the generation of dislocations in the matrix of metal matrix composites and can cause degradation of whisker or fiber strength. Whenever the problem is considered, the coefficient of thermal expansion, α , and the Young's modulus, E , appears in the equations. A special case is the uniaxial case of thermal loading with complete restraint:

$$\sigma = \alpha E \Delta T \quad (6)$$

where σ is the resulting stress from a temperature change of ΔT . It is evident from Eq. (6) that a greater αE product will yield a higher stress for equivalent magnitudes of ΔT . Since both α and E will be dependent upon the crystallographic orientations of the whiskers for both SiC and Al_2O_3 whisker reinforced composites, it is appropriate to examine the product of the two, αE , as it varies with crystallographic orientation and temperature. That product, namely αE , will be referred to as the thermoelastic stress index.

Since SiC is cubic and therefore isotropic with regard to thermal expansion, the thermoelastic stress index is similar in overall anisotropy to the elastic properties. Figure 9 depicts the thermoelastic stress index, αE , as it varies on the (110) plane. Figure 9 should be compared with Fig. 5 to observe the similarity of the anisotropies of the Young's modulus and the thermoelastic stress index. While the profiles of the two anisotropies on the (110) plane are quite similar, it is also obvious that the temperature dependence of the thermoelastic stress index anisotropy is much greater than that for Young's modulus. This rapid increase of the thermoelastic stress index

is a direct result of the increase of the coefficient of thermal expansion of SiC with increasing temperature. It clearly illustrates that much greater stresses will be generated for equivalent temperature differentials at elevated temperatures. Thermal cycling of SiC reinforced composites at elevated temperatures could be a more serious problem than cycling at lower temperatures.

The thermoelastic stress index for single crystal Al_2O_3 is presented in Fig. 10. It should be compared with Figs. 2 and 7 which individually illustrate the α and E anisotropies on the same crystallographic plane. The thermoelastic stress index has a profile which closely resembles that of the Young's modulus in Fig. 7 and also exhibits the intermediate minimum indicative of the Young's modulus. However, the temperature dependence of the thermoelastic stress index is much greater than that for the Young's modulus, as it is amplified by the increase of the coefficients of thermal expansion with increasing temperature.

It is of further interest to compare the thermoelastic stress indices for SiC and Al_2O_3 as illustrated in Figs. 9 and 10. On an absolute scale, that of Al_2O_3 is always much greater than that for SiC. However, simply comparing the absolute magnitudes is not the only means of contrasting these two materials. Another comparison is the degree of anisotropy of the thermoelastic stress index and its temperature dependence in the direction of its maximum. In SiC the thermoelastic stress index is a maximum in the $\langle 111 \rangle$, ranging from about 1.6 MPa/°C at room temperature to 3.0 MPa/°C at 1000 °C, an increase of more than 50 percent. In Al_2O_3 the maximum is parallel to the c -[0001] axis, and it only changes from 3.4 MPa/°C to 4.8 MPa/°C, about a 30 percent increase. Even though Al_2O_3 has a much greater absolute thermoelastic stress index, that of SiC is much more anisotropic and exhibits a much greater variation with increasing temperature. Thus, even though Al_2O_3 is anisotropic both elastically and in its thermal expansion, cubic SiC has a much more anisotropic thermoelastic stress index.

One final point of interest is to examine the axial stresses generated along the whiskers and fibers during cooling. This would require the inclusion of suitable matrix properties in a real composite. However, for purposes of contrast in this study, the hypothetical case of total restraint will be assumed and a form of Eq. (6) integrated. Figures 11 and 12 illustrate the results for SiC and Al_2O_3 , respectively. It is evident that different reinforcement orientations will result in significantly different levels of stresses and that those in Al_2O_3 will be about twice the level of those in SiC. Since the Young's moduli of the two are comparable, it is obvious that the larger stresses in Al_2O_3 are a consequence of the much larger coefficients of thermal expansion. It is also apparent that these thermoelastic generated stresses can be quite substantial.

Since there exists a strong tendency to utilize reinforcements in an orientation with the greatest Young's modulus (Ref. 24), it seems imperative to further consider the variation of their thermoelastic stress index. In the case of cubic crystals, including SiC, these crystallographic orientations are the very ones which will generate the highest thermoelastic stresses. That is also the case for Al_2O_3 ; however, other noncubic crystals may exhibit different tendencies depending on the specific relationships between their elastic and thermal expansion anisotropies.

CONCLUSIONS

SiC exhibits a strong variation of Young's modulus with orientation. The Young's modulus and its anisotropy are insensitive to temperature below 1000 °C. Al_2O_3 exhibits a smaller elastic anisotropy and a similar insensitivity to temperature. However, the coefficient of thermal expansion of Al_2O_3 depends on orientation and is a strong function of temperature. The product of Young's modulus and coefficient of thermal expansion, the thermoelastic stress index, was found to exhibit a directional profile similar to that of the Young's modulus, but much more variation with temperature because of the amplifying effect of the temperature dependence of the coefficients of thermal expansion. That feature emphasizes the importance of the level of temperature, as well as simply the value of ΔT during thermal cycling as it directly determines the resulting thermal stress levels which

are generated. The role of reinforcement orientation is demonstrated to be significant in determining the stresses which result in high temperature composites.

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TABLE I.—ELASTIC STIFFNESSES AND COMPLIANCES OF CUBIC SiC

	11	12	44
$S_{ij}(\times 10^{-3}/\text{GPa})$	3.673	-1.047	4.294
C_{ij}, GPa	352.3	140.4	232.9

TABLE II.—ELASTIC STIFFNESSES AND COMPLIANCES OF SINGLE CRYSTAL Al_2O_3

	11	33	12	13	14	44
$S_{ij}(\times 10^{-3}/\text{GPa})$	2.326	2.151	-0.693	-0.362	0.465	6.765
C_{ij}, GPa	500.1	502.4	161.7	111.4	-23.26	151.0

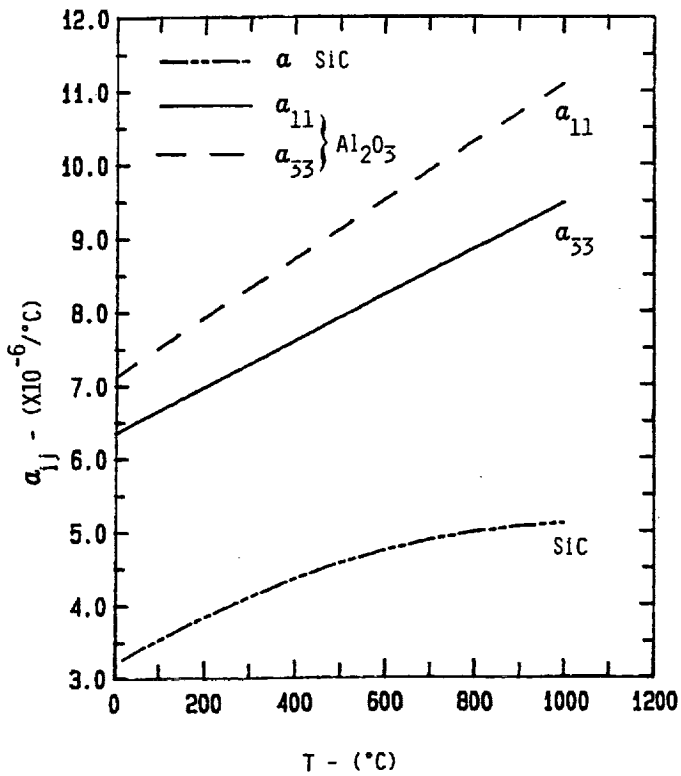


Figure 1.—The coefficients of thermal expansion, α_{ij} , as a function of temperature for cubic SiC and Al_2O_3 .

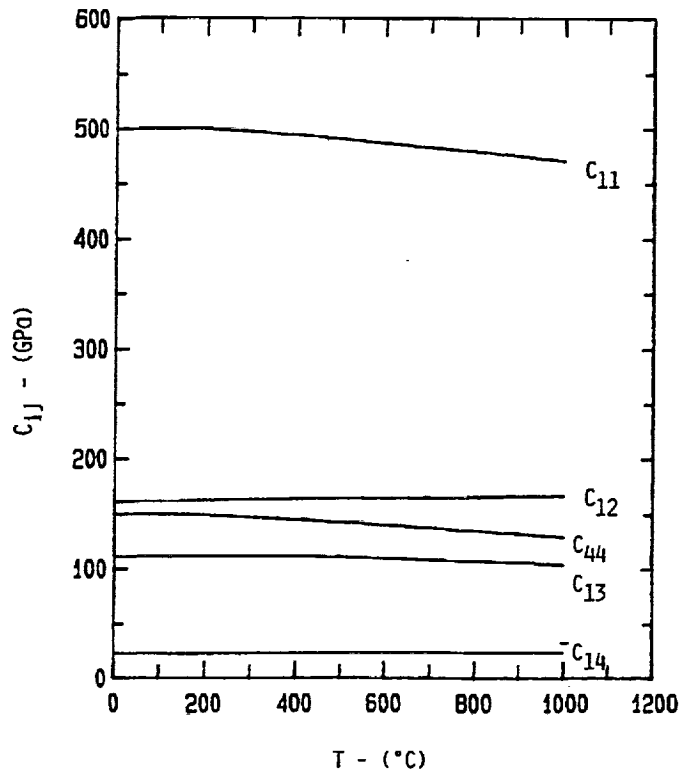


Figure 3.—The single crystal elastic stiffnesses, C_{ij} , of Al_2O_3 as a function of temperature.

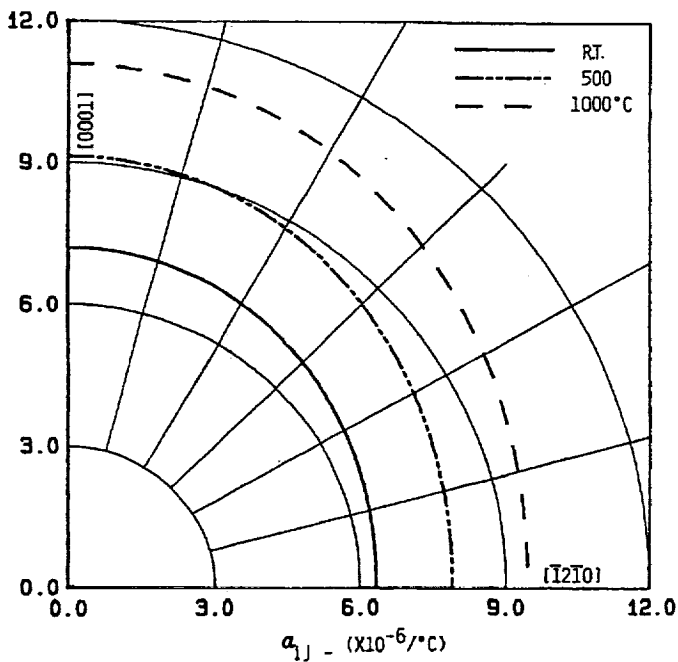


Figure 2.—The coefficients of thermal expansion, α_{ij} , of Al_2O_3 as a function of orientation on the (1010).

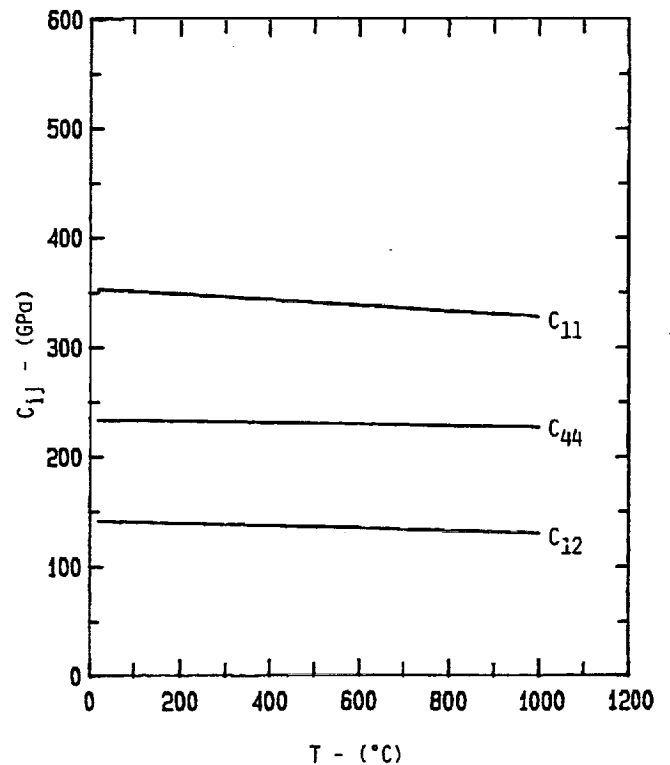


Figure 4.—The single crystal elastic stiffnesses, C_{ij} , of cubic SiC as a function of temperature.

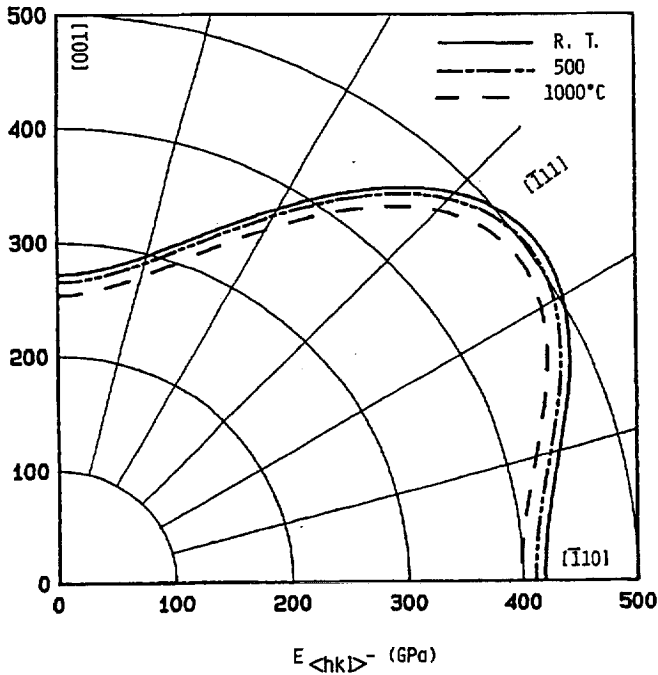


Figure 5.—The elastic modulus, $E_{\langle hkl \rangle}$, of cubic SiC as a function of orientation on the (110).

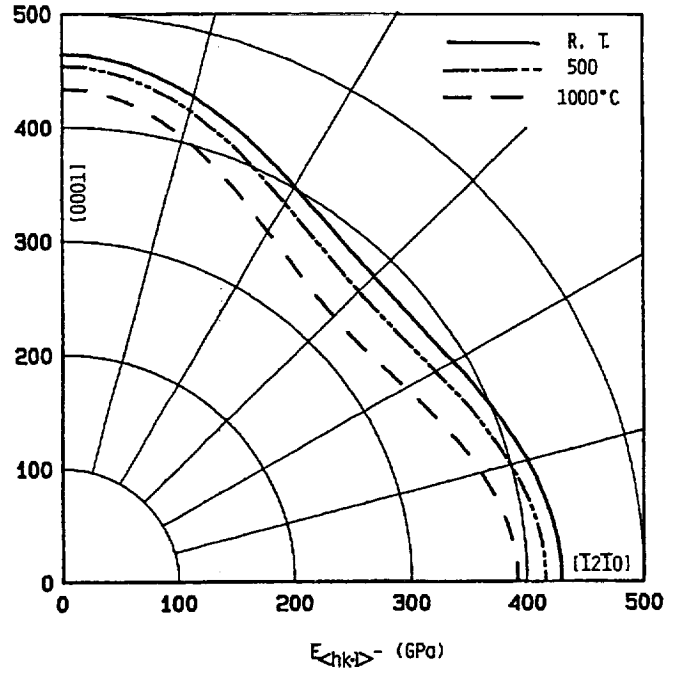


Figure 7.—The elastic modulus of, Al_2O_3 , $E_{\langle hkl \rangle}$ as a function of orientation on the $(10\bar{1}0)$.

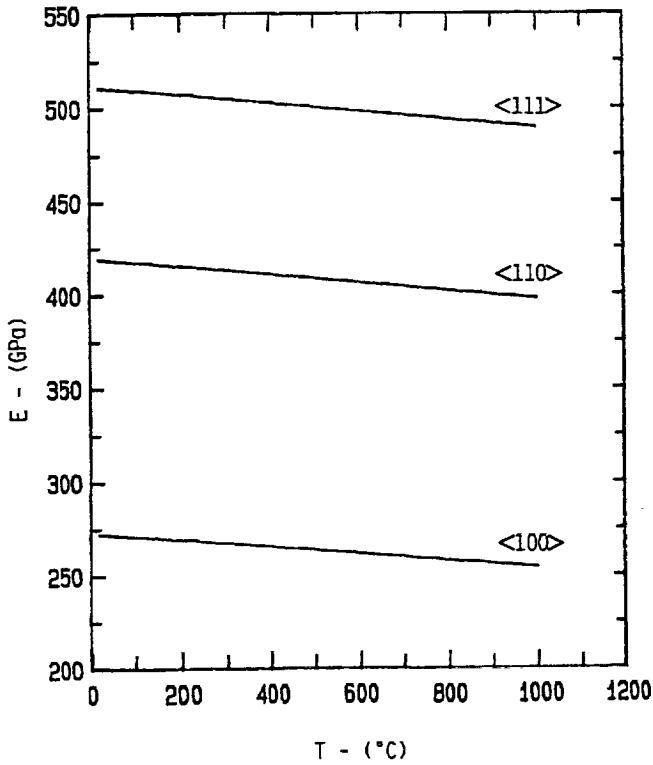


Figure 6.—The elastic modulus, $E_{\langle hkl \rangle}$, of cubic SiC as a function of temperature.

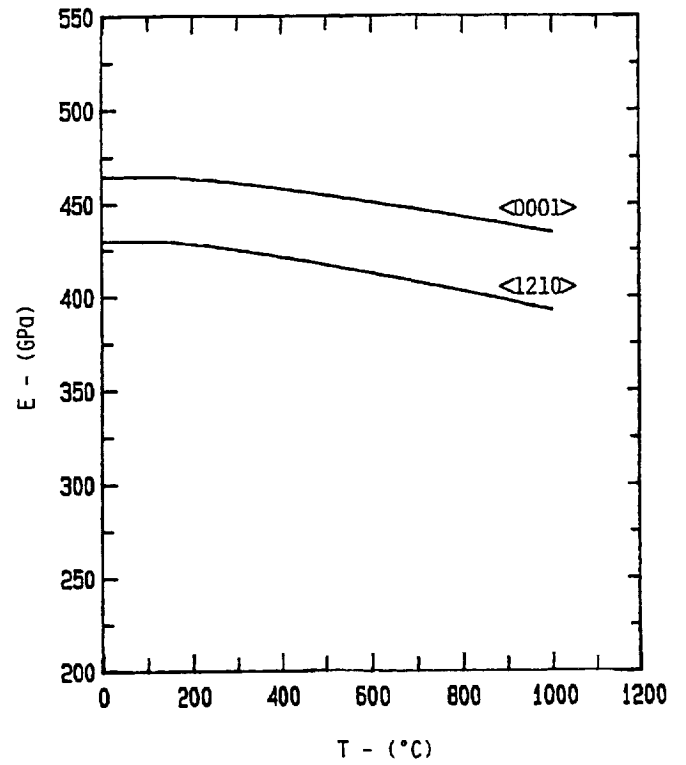


Figure 8.—The elastic modulus, $E_{\langle hkl \rangle}$, of Al_2O_3 as a function of temperature.

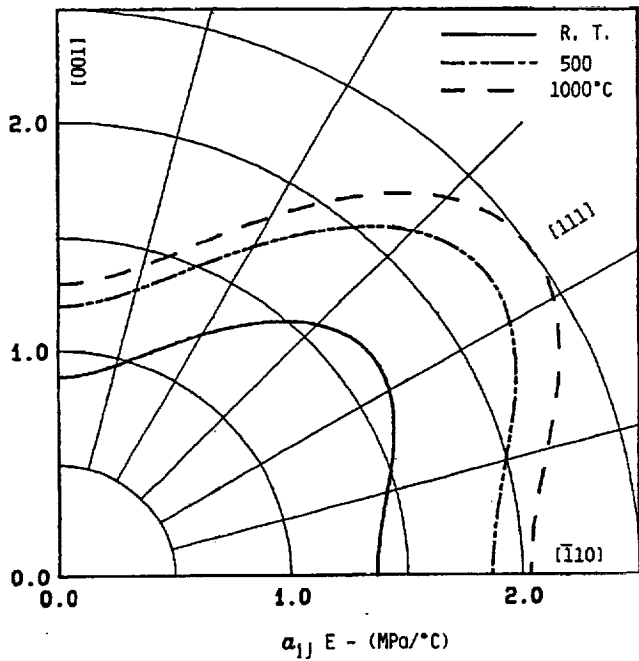


Figure 9.—The thermoelastic stress index for cubic SiC as a function of orientation on the (110).

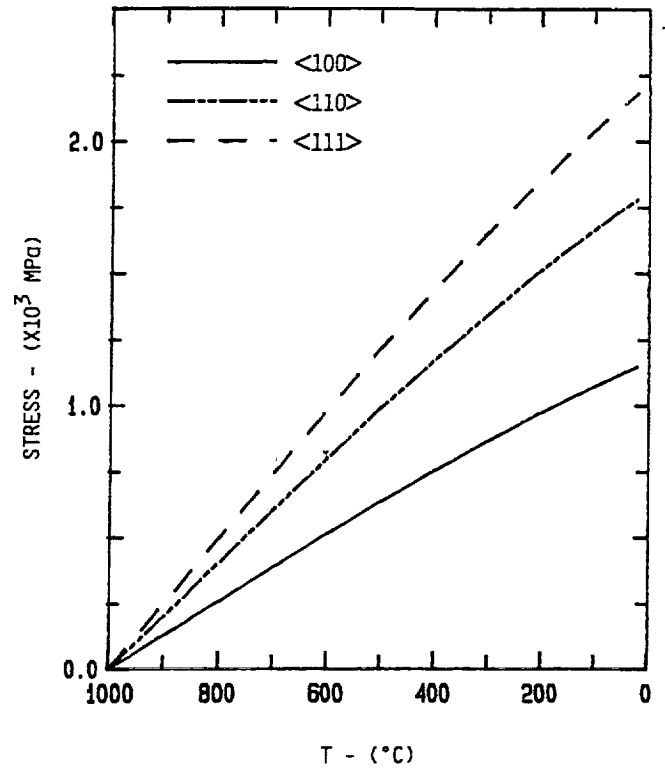


Figure 11.—The thermal stress in a completely restrained cubic SiC fiber as a function of temperature in the <100>, <110> and <111> directions.

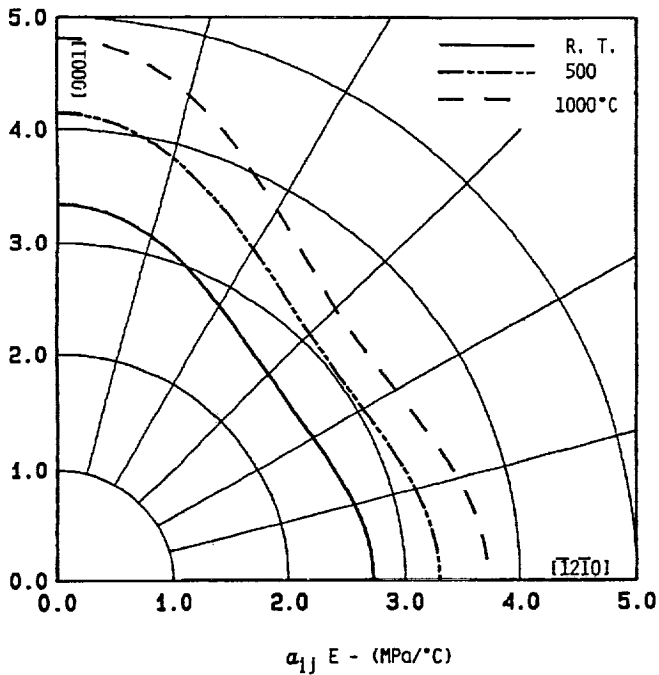


Figure 10.—The thermoelastic stress index for Al₂O₃ as a function of orientation on the (1010).

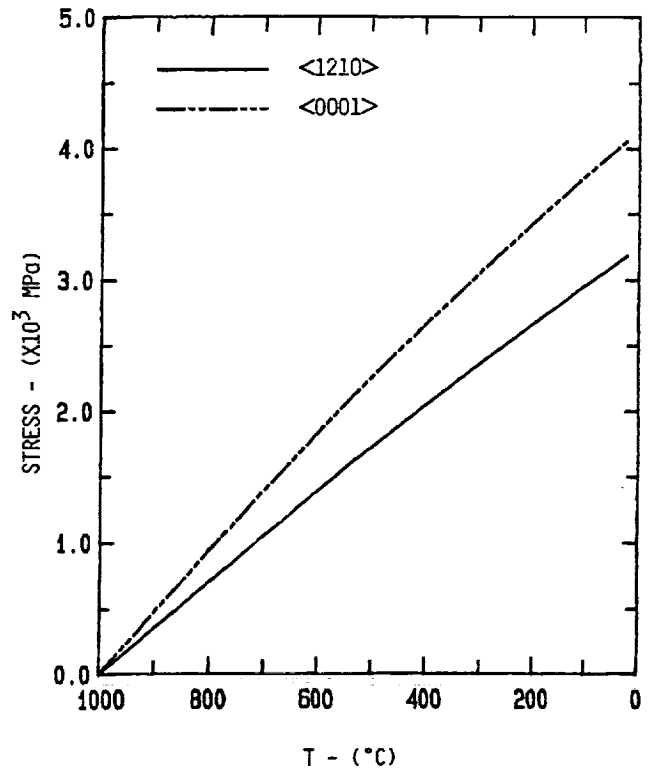


Figure 12.—The thermal stress in a completely restrained Al₂O₃ fiber as a function of temperature in the <1210> and <0001> directions.



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13. ABSTRACT (<i>Maximum 200 words</i>) In single crystal form, SiC and Al ₂ O ₃ are attractive reinforcing components for high temperature composites. In this study, the axial coefficients of thermal expansion and single crystal elastic constants of SiC and Al ₂ O ₃ were used to determine their coefficients of thermal expansion and Young's moduli as a function of crystallographic orientation and temperature. SiC and Al ₂ O ₃ exhibit a strong variation of Young's modulus with orientation; however, their moduli and anisotropies are weak functions of temperature below 1000 °C. The coefficients of thermal expansion exhibit significant temperature dependence, and that of the non-cubic Al ₂ O ₃ is also a function of crystallographic orientation.			
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