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# Thermal and Elastic Properties of Alumina–Silicon Carbide Whisker Composites

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***Abstract***

*The study described in this paper is specifically related to the thermal and elastic properties of $Al_2O_3$ matrix with SiC whisker reinforcements. Measurements of Young's modulus and thermal diffusivity/conductivity were performed on various $Al_2O_3$–$SiC_w$ compositions obtained by varying both the raw materials and the whisker volume fractions. While the Young's modulus was quite independent of the choice of raw materials, a remarkable difference was found for the thermal conductivity of composites processed with different whisker sources.*

*In dieser Veröffentlichung werden Ergebnisse zu den thermischen und elastischen Eigenschaften einer $Al_2O_3$-Matrix mit SiC-Einkristallverstärkungen vorgestellt. Messungen des Elasizitätsmodulus sowie der thermischen Diffusivität/Leitfähigkeit wurden an verschiedenen $Al_2O_3$–$SiC_w$-Zusammensetzungen durchgeführt. Dabei wurden die Volumenanteile sowohl des Trägermaterials als auch der Einkristalleinlagerungen verändert. Es ergab sich, daß der Elasizitätsmodulus unabhängig von der Wahl des Trägermaterials ist, während sich die thermische Leitfähigkeit der Verbindungen mit unterschiedlichen Einkristalleinlagerungen signifikant unterscheidet.*

*L'étude décrite dans cet article porte sur les propriétés thermiques et élastiques d'une matrice $Al_2O_3$ renforcée par des whiskers de $SiC_w$. Des mesures du module d'Young, de la diffusivité/conductivité thermique ont été effectuées pour plusieurs compositions de $Al_2O_3$–$SiC_w$ obtenues en faisant varier à la fois les matériaux de base et les fractions volumiques de whiskers. Alors que le module de Young semble relativement indépendant du choix des matériaux de base, on note un écart remarquable pour la conductivité thermique des composites fabriqués à partir de différentes origines de whiskers.*

## 1 Introduction

In the last few years, a strong effort has been devoted to the development of ceramic matrix composites with fibre, particulate or whisker reinforcements.[1,2] The driving force has been the improvement in mechanical properties of monolithic ceramics, in order to obtain an enhanced fracture toughness and greater reliability. Several mechanisms have been proposed to explain the observed toughening: crack deflection, pull-out, micro-crack formation, etc. In contrast, less attention has been paid to the thermal and elastic properties, which are critical in the material design for high temperature applications. Nevertheless, the addition of a second phase in a monolithic ceramic can change significantly not only the fracture behaviour but also a different set of properties named 'field properties'[3] such as the thermal conductivity, thermal expansion and elastic moduli.[4–9]

Alumina–SiC whisker composites ($Al_2O_3$–$SiC_w$) have received particular attention due to their successful application as cutting tools.[10,11] In all these applications the thermal conductivity must be as high as possible in order to reduce thermal shock failures. As is well known, the heat transfer properties strongly depend upon the purity level of the crystal lattice of the grains. Thus, a greater control of impurities, originating both from the raw materials and the processing conditions, has to be obtained.

In this paper, $Al_2O_3$–$SiC_w$ composites prepared by varying the raw material sources and processing times were studied. Different compositions for each specimen were obtained by varying the

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relative amount of SiC whiskers and $Al_2O_3$ powders. The Young's modulus and thermal diffusivity/conductivity measured in this work were compared to values reported in the literature for similar materials.

## 2 Experimental Procedures

### 2.1 Materials preparation

Three high-purity sub-micron $\alpha$-$Al_2O_3$ powders commercially available (A1, A2, A3) were used and combined with two different, $\beta$-SiC whiskers sources (W1, W2) as shown in Table 1. The dimension of whiskers W1 were 0.3–1 $\mu$m diameter and 20–50 $\mu$m length and of W2 were 0.05–1·5 $\mu$m diameter and 5–200 $\mu$m length. They were also made by different producers (Table 1). The $Al_2O_3$ powders and whiskers were wet processed in order to improve the homogenisation of the mixtures. The resulting powders were hot pressed in argon at temperatures of 1900°C under a pressure of 50 MPa for 20 to 60 min. A set of samples were obtained by varying raw materials and whisker volume fractions (28–39 vol.%).

As reported in detail elsewhere,[12] a good homogeneity in the distribution of whiskers was obtained with a preferential orientation in the plane perpendicular to the hot pressing direction (see Fig. 1(a) and (b)). The porosity of the final composites was always under 1% (Table 1). For this reason, the $SiC_w$ volume fractions were obtained by the 'rule of mixtures' and the density data (considering 3.98 g/cm$^3$ and 3.21 g/cm$^3$ as the theoretical densities of $Al_2O_3$ and SiC, respectively). The density was carefully measured on each sample using Archimedes' method by following the ASTM procedure.[13] With regard to the mechanical properties, the composites behave like similar materials reported in the literature; a room-temperature four-point bending strength (MOR) up to 650 MPa was obtained.[12]

Using the same processing conditions (60 min, 50 MPa) but lower sintering temperature (1400–1500°C), reference samples of $Al_2O_3$ were prepared in order to analyse the properties of the matrix alone. Figure 2 shows that the dimension of $Al_2O_3$ grains for a reference material (a) is comparable to that found for the composite (b) due to the contemporary effects of the higher sintering temperature and the presence of a second phase. The slight difference cannot produce a variation in the thermal properties for the $Al_2O_3$ phase between reference materials (A1–A3) and composites.[14]

### 2.2 Young's modulus measurements

The Young's modulus $E$ was determined by using a flexural resonance method based on an electrostatic excitation and laser modulation technique for detecting vibration amplitude.[15] By using the solution of the flexural vibration equation, $E$ is given as:

$$E = f_i^2 \frac{48\pi^2 \rho L^4 T}{(m_i^2 b)^2} \quad (1)$$

where $f_i$ and $m_i$ are the resonance frequencies and vibration constant of the $i$th mode; $b$, $L$ and $\rho$ are the sample thickness, length and density respectively. $T$ is a correction term which depends both on Poisson's ratio $v$ and the $b/L$ ratio.[16] The 30 ×

**Table 1.** Experimental results

| *Specimen* | *Powder producer* | *Type* | *ρ (g/cm³)* | *α (cm²/s)* | *κ (W/mK)* | *E (GPa)* |
|---|---|---|---|---|---|---|
| Alumina reference materials | | | | | | |
| A1 | Alcoa | A-16SG | 3·955 | 0·094 | 28·9 | 396 |
| A2 | Baikowsky | CR-30 | 3·974 | 0·092 | 28·4 | 402 |
| A3 | Sumitomo | AKP-20 | 3·980 | 0·097 | 29·9 | 398 |

| *Specimen* | *Sintering time (min)* | *ρ (g/cm³)* | *$SiC_w$ (vol.%)* | *P (vol.%)* | *α (cm²/s)* | *κ (W/mK)* | *E (GPa)* |
|---|---|---|---|---|---|---|---|
| W1 reinforced composites (W1 whiskers: Tokay Carbon grade-1) | | | | | | | |
| A1 W1 | 60 | 3·764 | 28·0 | 0·3 | 0·134 | 37·8 | 399 |
| A1 W1 | 60 | 3·723 | 33·4 | 0·1 | 0·148 | 40·9 | 404 |
| A1 W1 | 60 | 3·683 | 38·6 | 0·2 | 0·161 | 43·8 | 398 |
| A1 W1 | 30 | 3·697 | 36·8 | 0·1 | 0·152 | 41·5 | 406 |
| A1 W1 | 20 | 3·684 | 38·5 | 0·8 | 0·152 | 41·4 | 399 |
| A2 W1 | 60 | 3·710 | 35·1 | 0·1 | 0·153 | 42·1 | 411 |
| W2 reinforced composites (W2 whiskers: Thaheo Chem. SCW 1-S) | | | | | | | |
| A1 W2 | 60 | 3·680 | 39·0 | 0·6 | 0·124 | 33·7 | 396 |
| A1 W2 | 30 | 3·684 | 38·5 | 0·9 | 0·121 | 32·9 | 391 |
| A1 W2 | 20 | 3·693 | 37·3 | 0·3 | 0·122 | 33·2 | 398 |
| A3 W2 | 30 | 3·710 | 35·1 | 0·1 | 0·120 | 33·0 | 407 |

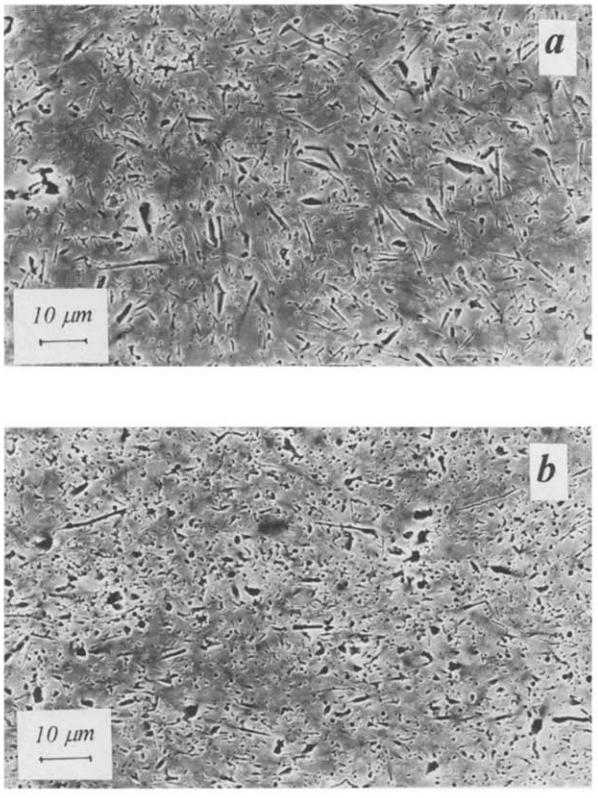


**Fig. 1.** Aluminia–$SiC_w$ microstructure. Polished sections chemically etched: (a) perpendicular and (b) parallel to the hot-pressing direction (sample A1W1). The outlines of the whiskers are clearly visible and have a preferential orientation in the hot-pressing plane.

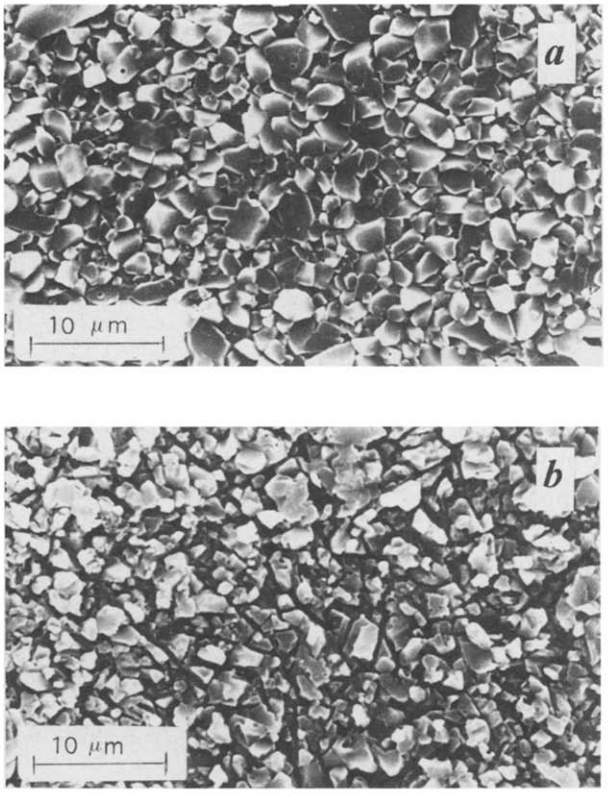


**Fig. 2.** Microstructure of (a) reference alumina (A1) and (b) alumina $SiC_w$ composites (A1W1) showing the grain size of the alumina phase. In (b) the whiskers were completely removed by the chemical etch.

10 × 3 mm samples were supported on two sharp knives that were positioned on the nodal lines corresponding to the fundamental resonance mode. The measurements were performed under vacuum to avoid damping and the frequency shift due to the presence of the air. In these conditions the accuracy of measurements was estimated to be ± 1%.[15]

### 2.3 Thermal diffusivity/conductivity measurements

Room-temperature thermal diffusivities were measured by the laser-flash technique.[17,18] A 50 J Nd/glass laser (Lumonics Ltd) with a pulse width of approximately 500 μs and a wavelength of 1·06 μm was used as the pulse source. The laser intensity was attenuated by a copper sulphate solution in order to maintain the temperature rise of the sample rear-surface below 1°C. The rear temperature rise of the sample was measured with an InSb infrared detector (EG&G Judson, J10D-M204-R04M 60). The signal was pre-amplified (EG&G Judson PA-9-44) and then transmitted to a low-noise amplifier (Stanford Res. Sys., SR 560) with variable gain and filtering. Afterwards, the signal was recorded by an accurate (12-bit) digital waveform recorder (Hewlett Packard, 5183 T), and fed to a personal computer via an IEEE-488 bus, programmed to control the apparatus and to sample 8192 data points. The thermal diffusivity $\alpha$ was then calculated by both measuring the fractional time rises (10–90%) and by using a least-squares analysis; the data agreed to within 1%. All the measurements were performed with the energy heat pulse along the hot-pressing direction. At least three samples (square-shaped 10 × 10 × 3 mm) for each specimen were considered.

Thermal conductivity values $\kappa$ were calculated by using specific heat literature data[19] and the experimentally obtained values of thermal diffusivity and density.

## 3 Results and Discussion

The experimental results are shown in Table 1. In general, the elastic properties of the composites did not reveal any dependence on the raw materials selected. On the contrary, a remarkable increase in the thermal conductivity was observed when W1 whiskers were used as the reinforcing phase.

### 3.1 Matrix properties

The properties of the different specimens of alumina (A1, A2, A3) measured in this work are in good agreement with those reported in the literature for hot-pressed alumina. The high density of these materials leads to average values of Young's modulus and thermal conductivity which can be

as high as 399 GPa and 29 W/mK respectively. Moreover, the scatter of the data among the three different specimens was always within the accuracy of the measuring techniques. This demonstrates the high reliability of current commercial alumina powders.

The $E$ values for $Al_2O_3$ specimens (399 ± 4 GPa) are in excellent agreement with the results reported by Fisher *et al.* (399·7 GPa) obtained on a sample of $Al_2O_3$ having the same density as the present materials under investigation.[20] The present data are also in a reasonable agreement with a theoretical estimate (402·9 GPa), calculated from the single crystal elastic data and an averaging procedure for polycrystals.[21]

The $\kappa$ values (29 ± 1 W/mK) measured on the three different specimens (A1, A2, A3) correlate well with those reported in the literature for high purity alumina (1–3 $\mu$m grain size).[14]

### 3.2 Composite material properties

The experimental data on Young's modulus were compared to the values predicted by the 'Absolute Bounds Model'.[22] In this model, two limit curves versus one phase volume fraction are given, and the expected values should fall within these limits. Among different bounds, the Voigt–Reuss limits are always valid regardless of the geometry distribution of the phases, i.e. whiskers and matrix. In fact the lower (R) and upper (V) limits are calculated by assuming that the composite consists of layers either parallel or perpendicular to the applied stress. The only requirement is that unknown phases and sliding between phases are not present. Under these conditions, the following inequality for Young's modulus holds:

$$E_R < E < E_V$$

where the upper bound (Voigt) is:

$$E_V = E_1 V + E_2(1 - V) + F$$

and the lower bound (Reuss) is:

$$E_R = E_1 E_2 / [E_2 V + E_1(1 - V)]$$

where $V$ and $E_1$ are the volume fraction and the Young's modulus of alumina respectively, while $E_2$ is the Young's modulus of the SiC whiskers. $F$ is a function of the bulk and shear moduli of both materials and, in this case, can be neglected ($\ll 0{\cdot}1\%$). The application of this model requires knowledge concerning the $E$ value of the single phases. The $E_1$ (399 GPa) value was measured directly on the reference alumina as already reported, whereas the determination of $E_2$ was more difficult, due to the large spread of data reported in the literature for $\beta$-SiC ceramics. For instance, a theoretical evaluation obtained using the elastic constants $c_{ij}$ leads to a Young's modulus in the range 270–510 GPa depending on the relative orientation between the applied stress and the $\langle hkl \rangle$ lattice direction. Using an averaging procedure[22] justified by the random orientation of whiskers in the plane perpendicular to the hot pressing direction, the theoretical estimate of $E_2$ is 420 GPa. This was in perfect agreement with Pezzotti *et al.*[23] who reported the same value for the $SiC_w$ Young's modulus.

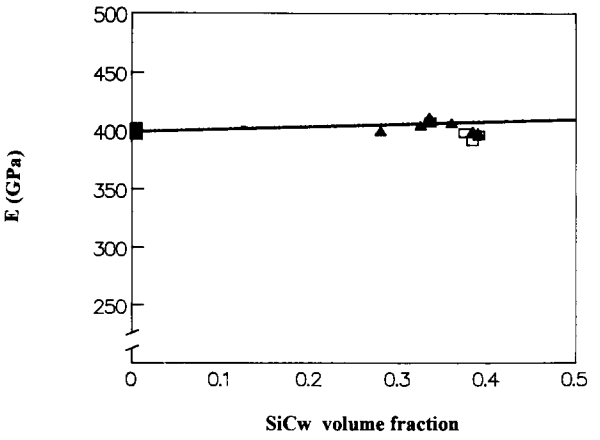


**Fig. 3.** Young's modulus versus whiskers volume fraction. —, The superimposing limits of the Voigt–Reuss theory calculated for a SiC value of 420 GPa; ■, reference aluminas; ▲, composites with W1 whiskers; □, composites with W2 whiskers.

In Fig. 3 the experimental data are plotted as a function of SiC volume fraction and compared to the theoretical VR bounds. These bounds are practically superimposed due to the slight difference between $E_1$ and $E_2$. Significant differences were not found between the two groups of composites, hence the choice of both alumina raw materials and whiskers sources (W1 and W2) is not reflected in a different behaviour of the elastic properties.

The effect of the whiskers on the thermal conductivity is more evident: material reinforced with W1 whiskers has a higher thermal conductivity (~40 W/mK) than W2-reinforced composites (~30 W/mK), regardless of the $Al_2O_3$ type used as the matrix. A different porosity content between the two different composites can be precluded as confirmed also by Young's modulus data. Thus, owing to the same processing conditions and the same matrix properties, such behaviour can be attributed to a difference in the thermal conductivity of the W1 and W2 whiskers ($\kappa_1$ and $\kappa_2$).

In order to explore the previous proposition, $\kappa_1$ and $\kappa_2$ values were extrapolated by using a composite model[3] belonging to the 'Effective Medium Theory' class.[22] In this model the dependence of composite thermal conductivity $\kappa$ on the volume fraction $V_D$ of dispersed phase is expressed as follows:

$$1 - V_D = \frac{\kappa_D - \kappa}{\kappa_D - \kappa_M} \left(\frac{\kappa_M}{\kappa_D}\right)^m \left(\frac{\kappa + n\kappa_D}{\kappa_M + n\kappa_D}\right)^q$$

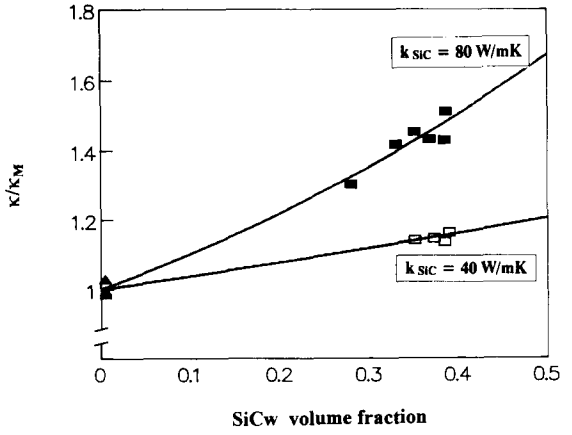


**Fig. 4.** Normalized thermal conductivity versus whiskers volume fraction. —, Theoretical dependence obtained for different $\kappa$(SiC) values; ▲, reference aluminas; ■, composites with W1 whiskers; □, composites with W2 whiskers.

$$m = \frac{A}{B} \qquad n = \frac{B}{C} \qquad q = \frac{A}{B} + \frac{D}{C}$$

$$A = F(1 - 2F)$$

$$B = 1 - (1 - F)\cos^2\varphi - 2F(1 - \cos^2\varphi)$$

$$C = 2F(1 - \cos^2\varphi) + (1 - F)\cos^2\varphi$$

$$D = 2F(1 - F)$$

where $\kappa_M$ and $\kappa_D$ refers to the matrix and dispersed phase respectively, and $F$ is a parameter which depends on the average aspect ratio of the dispersed phase. The angle $\varphi$ is the average angle between the principal axes of the dispersed phase and the heat flux. For the case of the whiskers, $F = 1/2$ and $\cos^2\varphi = 0$, due to their preferential orientation perpendicular to the heat flux. If the whiskers were randomly oriented ($\cos^2\varphi = 1/2$), the predicted value for a 30 vol.% whisker-reinforced composite would differ by about 5% from the previous case. This error is comparable to the accuracy of the measuring technique, therefore, the above approximation is always reasonable. The experimental data were normalised to the thermal conductivity of the reference alumina, which was taken to be the $\kappa_M$ value of the matrix alone. The comparison between the experimental results and the theoretical model (Fig. 4) leads to a rough estimate of the thermal conductivity of the whiskers. The values for W1 and W2 were 80 W/mK and 40 W/mK respectively. Such a difference can be linked to the intrinsic differences in the impurity content, crystalline defects and morphologies (e.g. cavities), which have already been reported for these two types of whiskers.[24–26] The W1 whiskers were prepared using very pure starting materials, thus resulting in a cleaner material with a lower content of crystalline defects. In contrast, the W2 whiskers were manufactured from rice-hulls (RH) as indicated by the manufacturer.[27] A similar result was found also by Hasselman and coworkers[5] for mullite composites reinforced with vapour–liquid–solid (VLS) whiskers and RH whiskers. They found a significant difference in the thermal conductivity of these two composites.

The value 40 W/mK, obtained for W2 (RH whiskers), is in good agreement with the literature for similar materials. In fact, it falls between 30 and 60 W/mK, the values obtained by Johnson *et al.*[7] and McCluskey *et al.*[8] for SC-9 RH whiskers (produced by Advanced Composite Materials Co. (formerly a division of ARCO Chemical Co.)). This comparison can be justified by the similarity that exists between SC-9 whiskers and W2 whiskers.[24] Unfortunately, in Ref. 5 an accurate estimation for the thermal conductivity of whiskers VSL was not performed due to the low thermal conductivity of mullite. Therefore, a quantitative comparison with the present whiskers W1 and VLS is not possible.

Both whiskers under study (W1 and W2) had thermal conductivity values that are considerably lower than that found by Slack for a high purity SiC single crystal (490 W/mK),[28] and were also lower than the highest value reported for polycrystalline hot-pressed SiC (270 W/mK).[29] However, it must be considered that the impurities have a detrimental effect on heat transfer properties.[30] For instance, Takeda showed the possibility of obtaining thermal conductivity values from fine-grained (2–5 $\mu$m) hot-pressed silicon carbide in the range 60–270 W/mK, by simply changing the sintering aids.[29] In particular, a value of 70 W/mK was obtained when $Al_2O_3$ (2 wt%) was used as sintering aid. This decrease in the thermal conductivity was explained in terms of the presence of Al in the SiC lattice, which is responsible for an increase of phonon scattering. More specifically, the amount of Al in the SiC lattice reached the solubility limit (about 0·8 wt% in the range 1800–2000°C)[31] during the sintering process.

For the present composites, the diffusion of Al can be roughly estimated by taking into account the processing conditions (60 min at 1900°C) and the diffusion coefficient for the SiC lattice ($D = 4 \times 10^{-14}$ cm$^2$/s).[32] If a very simple geometry is assumed (i.e. infinite defect-free crystal plate) the solubility limit is reached in a 200 nm thick layer. The presence of stacking faults and twins, most typical of whiskers, is responsible for an increase in the diffusion coefficient, hence, a heavy contamination of whiskers can be expected. The different hot-pressing times did not result in different thermal conductivities, as shown in Table 1, thus an almost complete contamination of the whisker phase is supposed, even at sintering times as low as 20 min.

Other effects, i.e. the presence of a thermal

barrier at the matrix–whiskers interface[33] and finite whisker size, are negligible, as demonstrated for these materials by Hasselman and coworkers[7] and McCluskey *et al.*,[8] respectively.

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