# FRACTURE TOUGHNESS DEPENDENCE ON TEMPERATURE, LOADING RATE AND OXIDATION TREATMENT FOR SI INFILTRATED SIC

T. Haug*, R.F. Pabst*

Fracture toughness measurements of reaction bonded Si infiltrated SiC ceramics are performed as functions of test temperature, annealing time, loading rate and microstructure. For fine grained materials the fracture toughness increases tremendeously at higher test temperatures and low loading rates. The increase is reduced heavily at very high loading rates. A three dimensional diagram fracture toughness-temperature-loading rate is therefore necessary. A material having a duplex microstructure of similar composition, however, is nearly independent of temperature and loading rate in the measured range.

## INTRODUCTION

SiC ceramics are typically high temperature materials. For use in the high temperature range the fracture behaviour should be known. It has been proved that it is not possible to draw conclusions unequivocally from room temperature measurements because of quasiplastic reactions caused by second phases and impurities. The data are then dependent on loading rate and time (oxidation processes). In any case a three dimensional diagram of fracture toughness $K_{Ic}$ - test temperature - loading rate becomes necessary to account for the special reactions. The temperature-loading rate sensitivity of the fracture toughness may also be influenced by a change in microstructure. Therefore a fine grained reaction bonded Si infiltrated SiC previously measured by Popp (1) is compared with a material having a duplex microstructure. This material was annealed in air at different temperatures and times to change the microstructure additionally by oxidation processes.

## MATERIAL

The Si infiltrated SiC-material studied in this paper is a comercial one having a duplex microstructure which contains coarse SiC grains dispersed in a fine grain size matrix. Table 1 includes some data of the microstructure together with the fracture toughness $K_{Ic}$ data measured at room temperature. Material-2 denotes the material with the duplex microstructure and material-1 the fine grained one which was used in previous

*Max-Planck-Institut für Metallforschung, Institut für Werkstoffwissenschaften, Seestraße 71, D-7000 Stuttgart 1, FRG

TABLE 1 - Material data

| Material | Vol% Si | $\bar{d}_{SiC}$ (μm) | $\bar{d}_{Si}$ (μm) | $\overline{K_{Ic}}$ (23°C) ($MN/m^{3/2}$) |
|---|---|---|---|---|
| SiC(IF)-1 Refel | 13.1 | 3.1 | 7.3 | 4.2 |
| SiC(IF)-2 | 19 | 3 30 | 4 | 3.9 |

studies by reference (1). Material-2 is also reaction bonded having nearly the same content of metalic Si-phase. The fracture toughness measurements of material-2 are compared with those made previously with material-1.

## EXPERIMENTAL

Three point bend specimens having dimensions 30 X 3.5 X 7.0 $mm^3$ and span width 24 mm were used for fracture toughness measurements. An artificial notch of width 70 μm and depth 1.4 mm was introduced by a diamond saw. The fracture toughness tests were conducted in a load controlled manner. Series of six specimens of the as received material-2 were tested at five different loading rates in air at 1100°C, 1200°C and 1300°C. At 1000°C and 1400°C only the lowest loading rate of 1.4 N/s was used. The highest loading rate was 14000 N/s. The highest and lowest loading rate data are equivalent to mean cross-head speeds of 0.016 mm/min and 160 mm/min, respectively. Until fracture occured all specimens were 12-13 min at the test temperature independent of loading rate. Annealing or preoxidation was carried out in air for 10 and 100 hours (notched specimens) at 1100°C and 1300°C. After a heat-treatment of 100h at 1300°C no further grain and oxidation growth should occur ; Heider (2). Series of six heat treated specimens (10h and 100h) were tested at four different loading rates at test temperatures 1100°C and 1300°C.

## RESULTS

### As received condition

Figure 1 illustrates the three dimensional dependence, fracture toughness $K_{Ic}$ - test temperature - loading rate (cross-head speed) for the material with the duplex microstructure (material-2) and the fine grained material (material-1). At the low cross-head speed (0.024 mm/min), the fine grained material exhibits a tremendeous increase in fracture toughness $K_{Ic}$ of about 300% at a test temperature of 1200°C in air. At very high loading rates the $K_{Ic}$-value decreases to the room temperature value. In contrast to material-1, the material having the duplex microstructure (material-2) is almost insensitive to the variation of test

temperature and loading rate. There is only a slight reduction of fracture toughness with increasing loading rate. The reduction increases with temperature. The $K_{Ic}$-value at a test temperature of 1300°C and tested with a loading rate of 1.4 N/s gives data which are 28% lower than those at a loading rate of 14000 N/s. At a test temperature of 1100°C the difference is only 15%. The loading rate effect is therefore much less distinct compared to material-1.

Heat treated condition

Figures 2 and 3 show the fracture toughness as functions of annealing time and loading rate for both the test and annealing temperatures 1100°C and 1300°C. The fracture toughness $K_{Ic}$ proved to be larger if the slow loading rate 1.4 N/s ≃ 0.016 mm/min is used independent of temperature and annealing time. At a temperature of 1300°C the annealing time has a stronger influence for a high loading rate 1400 N/s ≃ 16 mm/min. So, at 1.4 N/s the $K_{Ic}$-value increases nearly 5% whereas at 1400 N/s the increase is nearly 18%. Similarly, at a temperature of 1100°C : 4% increase at 1.4 N/s but 15% at 1400 N/s. On the whole all $K_{Ic}$-values measured at a test temperature of 1300°C exceed those measured at 1100°C.

Figures 4 and 5 give the fracture toughness as functions of loading rate and annealing time for the two test and annealing temperatures 1100°C and 1300°C. Up to the loading rate of 14 N/s the $K_{Ic}$-value is almost independent of annealing time irrespective of the test and annealing temperatures 1100°C and 1300°C. Also, for high loading rates and temperature 1100°C there is only a small increase of fracture toughness with annealing time. At 1300°C, however, and high loading rates the increase in fracture toughness is remarkable.

## DISCUSSION

It has been shown previously, that for the fine grained material-1 the fracture toughness is extremly sensitive to loading rate and temperature. This behaviour was traced back to the free Si which is plastic at high temperatures and low loading rates and brittle at high loading rates. It is resonable to assume that dislocation movement is intensified with increasing temperature. Dissipative processes like the diffusion of point defects are also likely. These processes are thermally activated and important if $T > T_S/2$. Both processe depend heavily on local conditions. It is likely that the individual effects or the combinations of the energy dissipative processes result in a change of grain size and shape and the grain slip characteristic, which may also influence microcrack formation. On the whole, all mechanisms should heavily depend on the SiC grain size.

Material-2 (duplex microstructure) has a larger content of free Si (19 vol%) than the fine grained one (material-1, 13 vol%). From this it is likely that both materials would show nearly the same fracture toughness behaviour as functions of test temperature and loading rate. Suprisingly that is not the case.

Consequently the microstructure of SiC grain sizes should largely influence the plasticity effect of the free Si-phase. It

is assumed that the large SiC grains with material-2 reduce considerably the energy dissipation resulting in lower $K_{Ic}$-data and in an insensitivity to temperature and loading rate.

The heat treatment or pre-oxidation process at high temperatures depends on the porosity and the annealing time. At low temperatures ($\simeq 1100^0C$) pores and channels at the specimen surface are not closed by an oxidation layer. So, oxidation within the specimen can occur. At high temperatures ($\simeq 1300^0C$), however, the oxidation proceeds so quickly that pores and channels are closed in a short time and oxidation inside the specimen is prevented. Material-2 is very dense so that oxidation inside the specimen is of secondary importance. Oxidation is therefore limited to or near the surface.

Oxidation or heat treatment processes normally increase the fracture toughness. This is illustrated in figures 2 and 3. The highest increase was found at an annealing and test temperature of $1300^0C$ for annealing times 10h and 100h. The increase is probably due to oxidation reactions, notch ground blunting and micro-crack healing. With higher annealing and test temperatures the loading rate effect becomes more pronounced. This is naturally due to an enhanced plasticity reaction at higher temperatures. The annealing time has a remarkable influence at high loading rates (140 N/s $\simeq$ 1.6 mm/min ; figures 4 and 5). Probably the oxide layer changes during testing (e.g. by crystallisation) and a re-oxidation will occur. As the re-oxidation occurs during the loading process the influence of loading rate can not be explained conventionally.

From the above discussion, it is possible that for the as received specimens tested at low loading rates (long test time) the oxidation is nearly completed resulting in similar $K_{Ic}$-values as measured for annealed specimens.

## CONCLUSIONS

Contrary to the results obtained with the fine grained reaction bonded Si infiltrated SiC tested previously by reference (1), the material (duplex microstructure) studied in this paper shows a slight dependence of fracture toughness on the test temperature and the loading rate. This is because the larger SiC-grains greatly influence the plasticity and energy dissipation effects of the free Si-phase. Pre-oxidation (heating experiments) increases the fracture toughness. This is especially the case at high annealing and test temperatures and high loading rates.

## SYMBOLS USED

$K_{Ic}$ = fracture toughness ($MN/m^{3/2}$)
$T$ = temperature ($^0C$)
$T_s$ = melting temperature ($^0C$)

## REFERENCES

1. Popp, G., 1981 Dissertation University Stuttgart

2. Heider, personal comunication

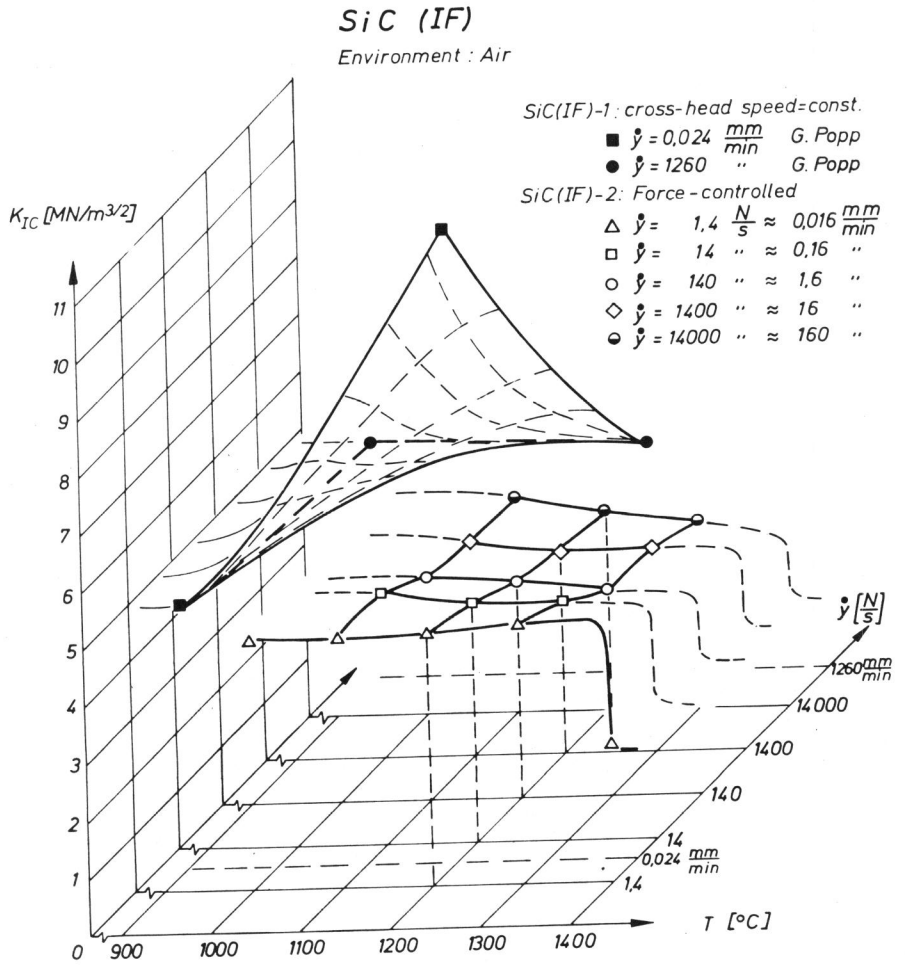


Figure 1 $K_{Ic}$ as functions of temperature and loading rate for two Si infiltrated SiC ceramics with different microstructures

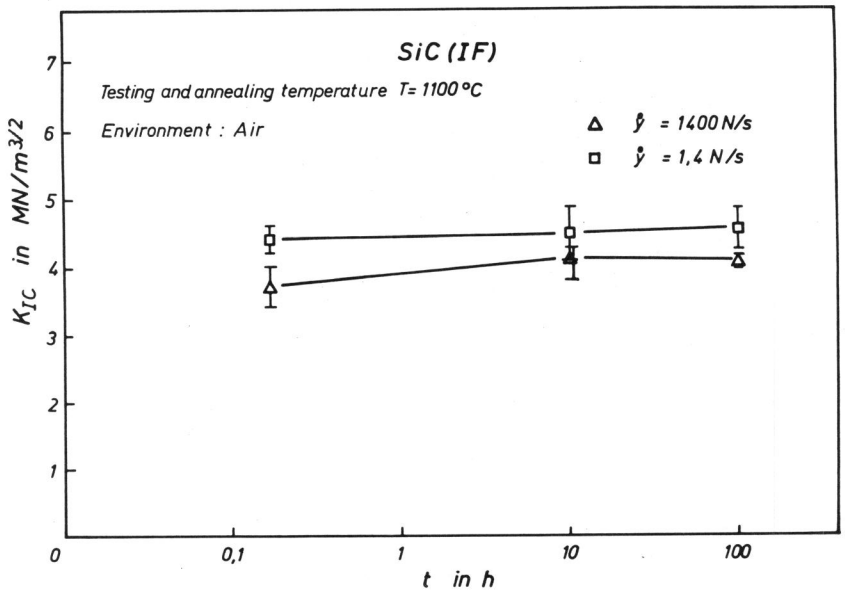


Figure 2 $K_{Ic}$ as functions of annealing time and loading rate at an annealing and test temperature of 1100°C

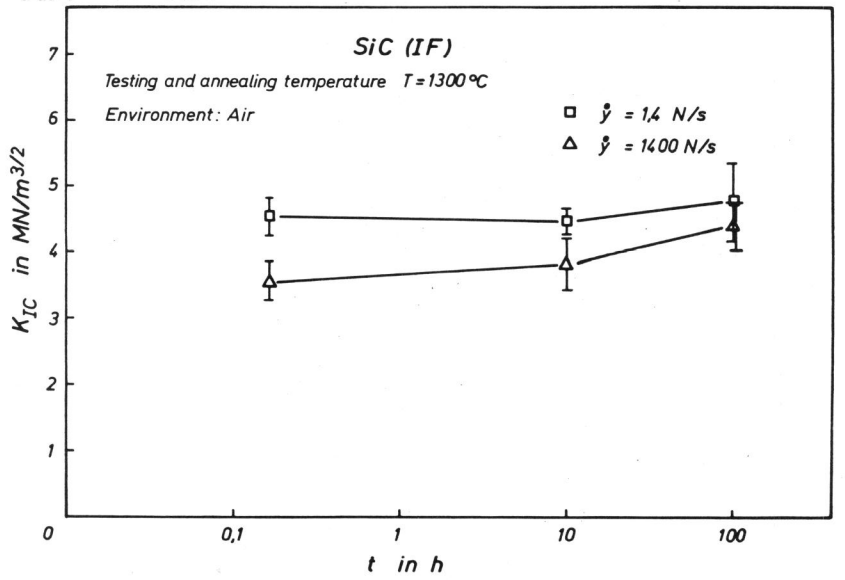


Figure 3 $K_{Ic}$ as functions of annealing time and loading rate at an annealing and test temperature of 1300°C

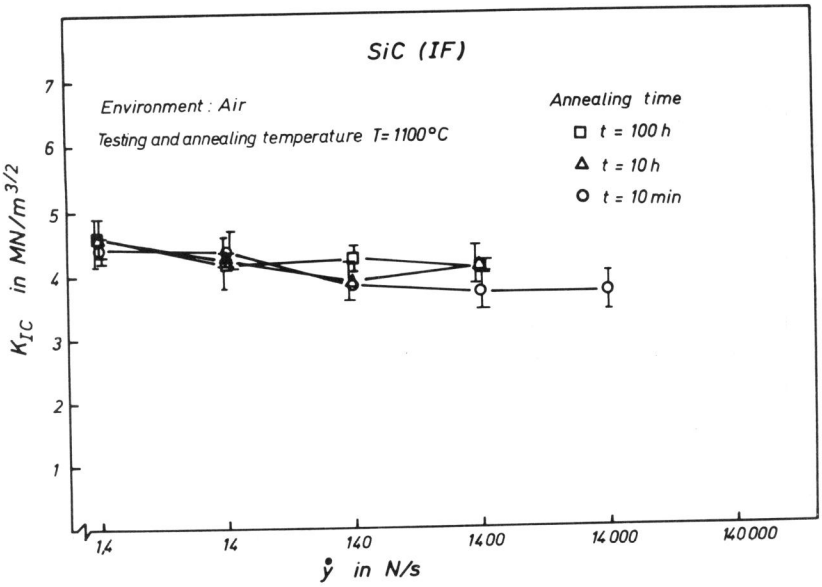


Figure 4 $K_{Ic}$ as functions of loading rate and annealing time at an annealing and test temperature of 1100 °C

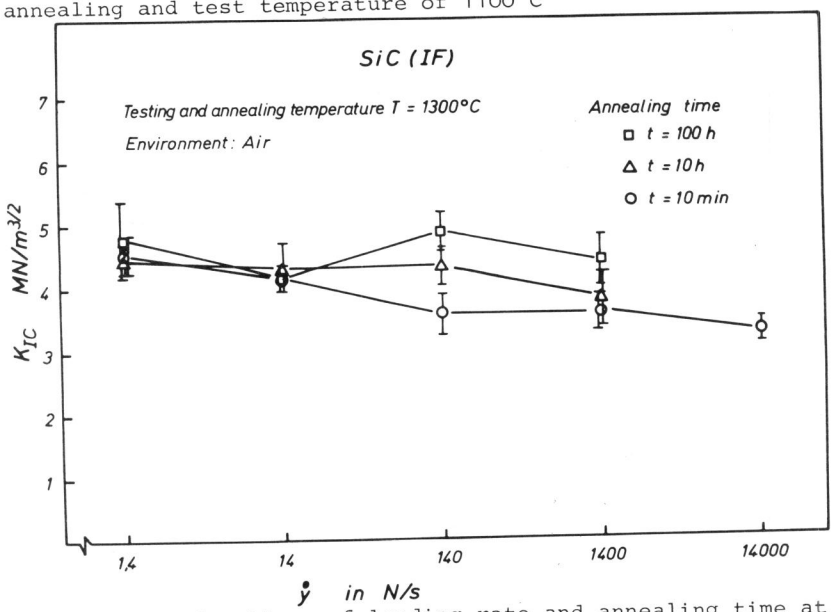


Figure 5 $K_{Ic}$ as functions of loading rate and annealing time at an annealing and test temperature of 1300 °C