

phys. stat. sol. (a) **108**, K81 (1988)

Subject classification: 61.70 and 78.60; S1.61

Departamento de Física de Materiales, Facultad de Físicas,
Universidad Complutense, Madrid¹⁾

SEM-CL of Reaction Bonded SiC

By

J. PIQUERAS, F. DOMÍNGUEZ-ADAME, and B. MENDEZ

Reaction bonded silicon carbides have a complex polycrystalline structure formed by bonding together SiC powders with SiC produced by a reaction between silicon and carbon. The latter SiC has been often described to form an epitaxial coating on the original α -SiC (hexagonal polytype) grains and to nucleate also independently as fine grained β -SiC (cubic polytype). The structure of the epitaxial deposit has been investigated by several authors /1 to 6/. It has been found /5, 6/ that the deposit grows as β -SiC but almost fully transforms to α -SiC during the high temperature processing. The resulting grains have a composite nature with different levels of impurity in the original grains and the epitaxial coating. Some interstitial regions are occupied by residual silicon. In the above mentioned investigations, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) have been frequently used. In particular the secondary electron SEM images clearly show /1, 5/ the different parts - original grains, coating, silicon regions of the grain structure. In the present work the application of the SEM cathodoluminescence (CL) mode to the microstructural characterization of reaction bonded SiC is investigated.

Samples with a thickness of about 1 mm were cut from a 5 mm diameter reaction bonded SiC rod (Goodfellow Metals Ltd.). The samples were mechanically polished and observed in a S4-10 SEM in the CL mode at temperatures between 300 and 100 K. The experimental method for CL measurements, in the range 350 to 850 nm, has been previously described /7/.

Fig. 1 (see on the photo pages before the Short Notes part) shows the CL images of the same specimen area at different temperatures. The CL emission is highly inhomogeneous through the sample. Some regions are highly luminescent as compared with other regions which either show a moderate emission or appear dark in the CL image. Decreasing the temperature from

1) E-28040 Madrid, Spain.

300 to 215 K causes a marked increase of the total CL intensity. Some regions with low emission at room temperature appear very intense at 215 K while dark regions remain unaltered. No changes are observed in the range 215 to 190 K but further cooling to about 140 K produces a luminescence decrease. Comparison of Fig. 1b and c indicates that this effect is not due to the luminescence decrease of all emitting regions but mainly to the decrease in some high emitting regions, as those marked A in Fig. 1b. In addition, a small number of regions, as B in Fig. 1c, show a luminescence increase. Decreasing the temperature to 100 K causes a further rearrangement of the luminescent areas.

The temperature dependence of the CL images is associated to spectral changes. Fig. 2a shows the CL spectrum at 273 K. By decreasing the temperature a broadening of the spectrum and a relative increase of the emission at wavelengths at both sides of the peak is observed. This is shown in the spectrum at 180 K of Fig. 2b. At lower temperatures a band at 410 nm emerges and the spectrum of Fig. 2c, recorded at 100 K, is obtained. These results indicate that the contrast in Fig. 1a (room temperature) is mainly due to the regions with emission in the broad band centered at 530 nm shown in Fig. 2a. At lower temperatures (about 200 K) regions with emission at other wavelengths contribute additionally to the CL causing the intensity increase observed in Fig. 1b. Below 130 K the luminescence is localized in regions emitting at 410 nm while some regions with high room temperature luminescence show a poor emission at low temperature.

Some sample regions appear dark in the CL image at all temperatures. The lack of emission and their sparse distribution in the sample suggest that these regions correspond to the residual silicon observed /1, 5/ in backscattered and secondary electron SEM images. On the other hand, the SiC grains cannot be readily recognized in the CL image. This is probably due to the impurity distribution within the grains with a concentration which is higher in the epitaxial coating than in the grain core. The impurity distribution would cause an inhomogeneous luminescence emission in the SiC grains. Close to room temperature the CL spectrum shows a broad complex band with intensity maximum at 520 to 550 nm. Several shoulders and sub-bands, more visible at 180 K (Fig. 2b) indicate that a number of impurity, and possibly defect, levels contribute to this emission. It is known that impurities as boron, nitrogen, and aluminium /8 to

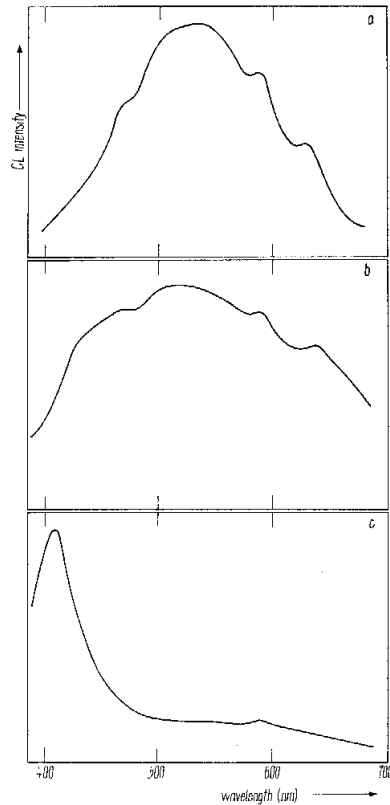


Fig. 2. CL spectrum at a) 273 K, b) 180 K, c) 100 K

10/ are involved in the green and yellow emission of β -SiC: In /10/ a broad CL band peaked at about 540 nm in Al implanted β -SiC is reported. Al has been found /5/ to be the main impurity in the epitaxial layer of the reaction bonded SiC while in the grain core is present in smaller amount. We suggest that in the upper range of temperatures used in this work, impurity related CL emission dominates and the CL images show mainly the impure (100 ppm /5/) regions. At temperatures below 130 K the CL is localized in regions emitting at 410 nm. This wavelength corresponds to the band gap energy of 6H-SiC. The regions imaged at these temperatures would be the purer parts - grain cores - of the SiC grains.

The present results show that SEM-CL provide information, additional to

the information obtained by other electron microscopy techniques, on the structure of reaction bonded SiC.

This work was supported by Ministerio de Educación y Ciencia (Proyecto PB 86 - 0151).

References

- /1/ G.R. SAWYER and T.F. PAGE, *J. Mater. Sci.* **13**, 885 (1978).
- /2/ A.H. HEUER, G.A. FRYBUPG, L.U. OGBUJI, and T.E. MITCHELL, *J. Amer. Ceram. Soc.* **61**, 406 (1978).
- /3/ T.F. PAGE and G.R. SAWYER, *J. Mater. Sci.* **15**, 1850 (1980).

- /4/ L.U. OGBUJI, T.E. MITCHELL, and A.H. HEUER, J. Amer. Ceram. Soc. 64, 100 (1980).
- /5/ J.N. NESS and T.F. PAGE, J. Mater. Sci. 21, 1377 (1986).
- /6/ L.U. OGBUJI, T.E. MITCHELL, and A.H. HEUER, J. Mater. Sci. 14, 2267 (1979).
- /7/ J. LLOPIS and J. PIQUERAS, J. appl. Phys. 54, 4570 (1983).
- /8/ A.P. ANDREEV, E.E. VIOLIN, YU.M. TAIROV, and O.A. FAYANS, Soviet Phys. - Semicond. 7, 208 (1973).
- /9/ S.H. HAGEN and A.W.C. VAN KENENODE, phys. stat. sol. (a) 33, 97 (1976).
- /10/ A.P. ANDREEV, E.E. VIOLIN, K.D. DEMAPOV, YU.M. TAIROV, and V.E. UDALTSOV, Soviet Phys. - Semicond. 15, 119 (1981).

(Received March 24, 1988)

See Short Note by J. PIQUERAS et al.

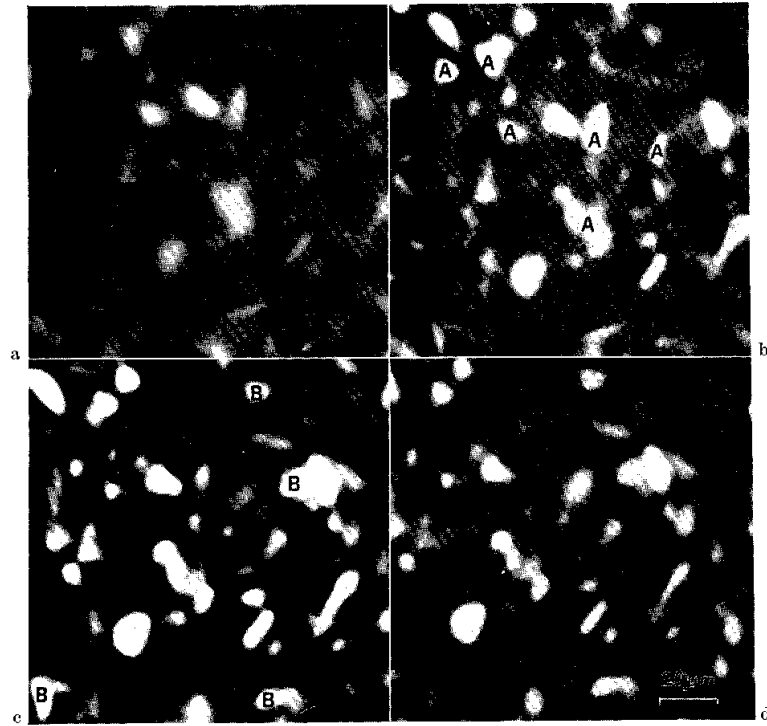


Fig. 1 CL images of the same sample region at a) room temperature, b) 215 K, c) 140 K, d) 100 K