MICROSTRUCTURE OF SILICON CARBIDE GRINDING TOOLS

By

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a) *Silicon carbide grinding grains*

Silicon carbide became known at first as an artificial material but was found later also in nature in small amounts. The mineralogical name for silicon carbide is moissanite. In the iron meteorite found in Canon Diablo (Arizona), green silicon carbide interlaced with diamond has been detected by *Moisson*.

The formation of silicon carbide was first observed by *Berzelius* (1810, 1824), then by *Despretz* (1849) and *Cowles* (1884). The invention of a process for its production on industrial scale is connected with the activity of *Acheson* (1891). In their experiments for the production of diamond, *Despretz* and *Acheson* succeeded in producing silicon carbide by chance. Namely, they tried to 'recrystallize' carbon in a clay melt. Meanwhile, an extremely hard crystalline substance was formed the chemical composition of which was at first quite unknown. *Acheson* considered it to be a carbon-containing corundum and thus coined for it at first the name carbocorundum and later simply the name carborundum. Investigations showed later that a new compound corresponding to the overall formula SiC has been discovered. *Acheson*, on recognizing the availability of the novel substance for use as grinding grain, patented his process and founded the first silicon carbide factory (Carborundum Co., 1891) at the Niagara. Of the carbon–silicon phase diagrams found in literature, one of the very detailed forms is shown in Fig. 1/a.

The technical significance of silicon carbide was immense already in the years following its discovery and the development of the process for its production on industrial scale. In the first years its fields of application included the industry of grinding tools and the manufacture of refractories. The manifold use of silicon carbide in special fields followed in the period after World War II, when its application was extended to quite diversified new fields. Besides of the classic products of silicon carbide (electric industries, production of refractories and machining tools), novel products made of silicon carbide were semiconductors, rocket tips, nozzles, fuel elements and heat exchangers used in nuclear technique. Recently, also heat insulators, surface protecting agents and catalyst carriers of very favourable properties are produced of

silicon carbide. Threads of silicon carbide thinner than those of rayon are highly resistant to heat and are elastic. They can be used mainly for the production of fireproof coatings. Also the protecting shields of spacecraft are produced from silicon carbide.

Silicon carbide has two polymorphous modifications: the rhombohedral and hexagonal α-silicon carbide applied also as grinding grain, and the cubic β-silicon carbide.
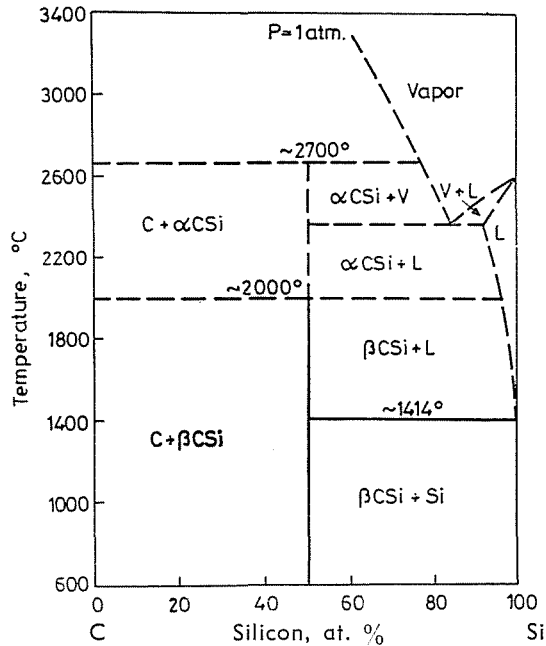

Fig. 1. a) Phase diagram of carbon—silicon

The crystal lattice of the silicon carbide pertaining to the regular system is of sphalerite type (ZnS–T_d). Its structure can be considered to consist of two cubic face-centred Si and C lattices which are shifted by 1/4 space diagonal in relation to each other. Into the (111) planar network of Si atoms fitting very compactly in positions denoted consecutively by ABC, a similarly built-up C-lattice fits in a way that it is shifted in the direction of the (111) plane--normal by the $z = 3/4$ part of the lattice distance (111). In this way the apexes of the Si tetrahedrons are located in the mass centres of the C tetrahedrons whereas the apexes of the C tetrahedrons in the mass centres of the Si tetrahedrons. Thus the SiC belonging to the regular system is built up of Si–C double planar networks which follow each other in ABC order. Several poly-types of α-silicon carbide are known which differ from the β-silicon carbide and from each other only in the order of the stratification of the double net-

works. In the order of this stratification two layers denoted by the same symbol cannot follow each other. The number of polytypes is fairly high, up to the present more than thirty ones have been discovered.

The most important polytypes present in technical silicon carbide are shown in Fig. 1/b.

Modification		Ramsdell symbol	Stacking sequence	Number of double layers in the elementary cell	Crystal system	c/a ideal
β-SiC		3 C	ABC ABC ...	3	regular $T_d^2 - F\,43m$	$\sqrt{6} = 2.450$
α-SiC	SiC I	15 R	ABCBACABACBCABC...	15	rhombohedral $C_{3V}^5 - R\,3\,m$	$5\sqrt{6} =$ $= 12.247$
	SiC II	6 H	ABCACB ...	6	hexagonal $C_{6V}^4 - P\,6_3\,mc$	$2\sqrt{6} =$ $= 4.899$
	SiC III	4 H	ABAC...	4	hexagonal $C_{6V}^4 - P\,6_3\,mc$	$4/3\sqrt{6} =$ $= 3.266$

Fig. 1/b More important modifications of silicon carbide

Owing to the very diversified crystal structure of silicon carbide, its properties are extremely manifold. Recently, it is mentioned as the most complex inorganic compound.

b) *Interaction of silicon carbide grinding grains and ceramic bonding materials*

Whereas at high temperature at the phase boundary of electrocorundum grains and ceramic bonding materials a well observable bonding layer of a width exceeding sometimes even 100 μm can form on the processing effect of the vitreous bonding material, in the case of silicon carbide this layer attains only a width of a few micrometers. Vitreous bonding materials cannot be used for bonding silicon carbide because they decompose silicon carbide chemically during firing. Thus, only raw materials compacting like china can be applied as bonding materials. The adhesion of the china-like bonding material is shown in Fig. 2/a Also the records of the measurement of practically equal microhardness in Fig. 2/b indicate that the properties of bonding material remain unchanged also in the environment of the phase boundary, and no transition layer of differing composition and behaviour can be observed by this method.

For the more accurate characterization of the phase boundary between silicon carbide and the ceramic bonding material, the main components of

a)
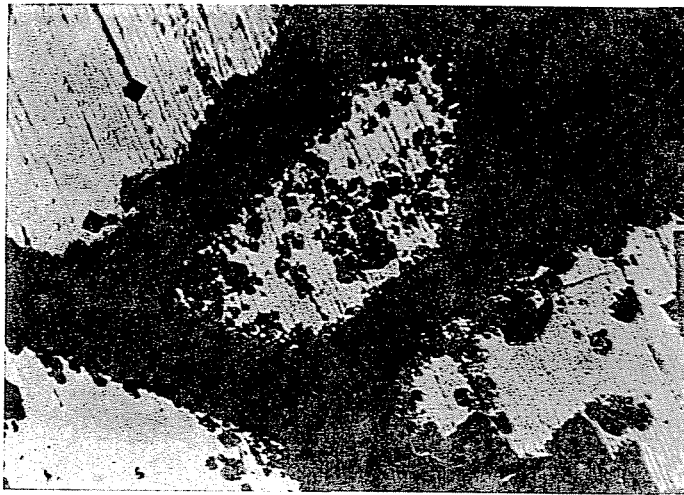
b)

Fig. 2. a) surface of a grain of silicon carbide (1) with a coating layer of ceramic bonding material (2). ×900; b) light photomicrograph of the polished surface of a grinding tool with ceramically bonded silicon carbide grains with records of microhardness measurement. ×700
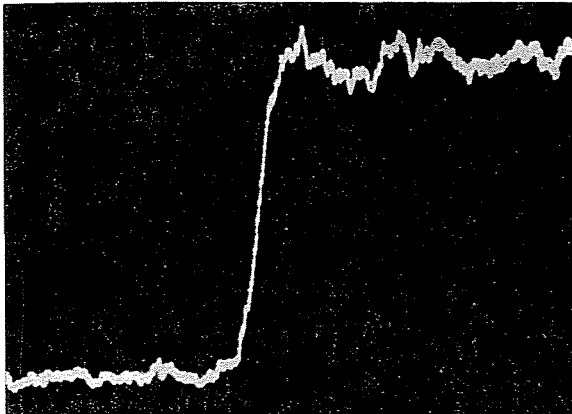1: Silicon carbide grain, 2: bonding material, 3: record of microhardness measurements, 4: air pore

the system grain-bonding material were determined. It is seen in Fig. 3 that the curve of distribution of silicon and aluminium indicating the position of the bonding layer exhibits a very abrupt change in concentration and does not show a bonding layer because its width is too small to be observed by this method.

a)

b)

c)
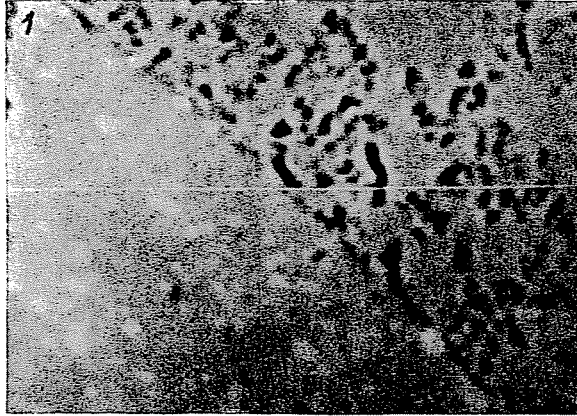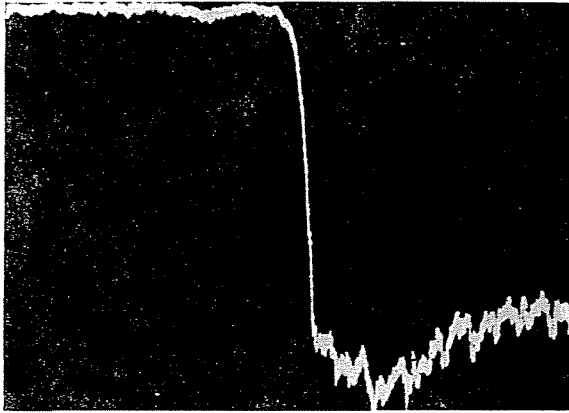
Fig. 3. Electrone probe image of the phase boundary of a silicon carbide grain (1) and of the ceramic bonding material (2). a) Composition with the site of the measurement of distribution. ×1800; b) distribution of Si content; c) distribution of Al content

Owing to the already mentioned diversified and complex crystalline structure, the microcrystalline structure of the silicon carbide grains differs fundamentally from that of electrocorundum grains, and thus it cannot be investigated by the methods applied for that purpose. The morphological

a)
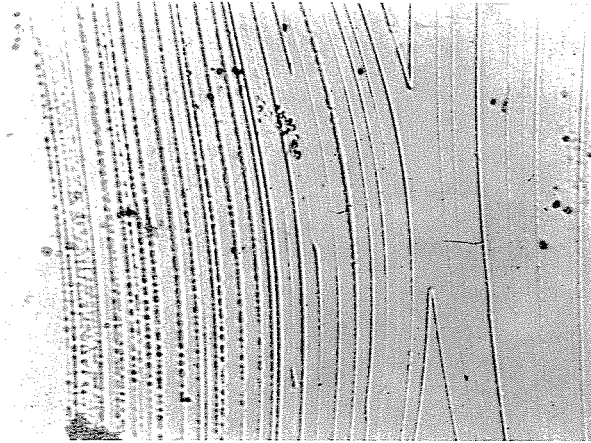
b)

Fig. 4. Light photomicrographs of the etched surface of grinding grains of silicon carbide.
a) ×300; b) ×300

features of the surface of silicon carbide used for grinding purposes have been investigated after etching. Though etching serves actually for the study of the morphological and crystal growth mechanisms of silicon carbide single crystals, we used etching also for obtaining indirect information from the behaviour of the grains against various reagents on the expected reactions of grains with

bonding materials differing by type and chemical nature. It must be noted that the light micrographs exhibited in Figs 4 to 12 which show polygonization crystal zones, forms of growth, etching cavities of various forms and other
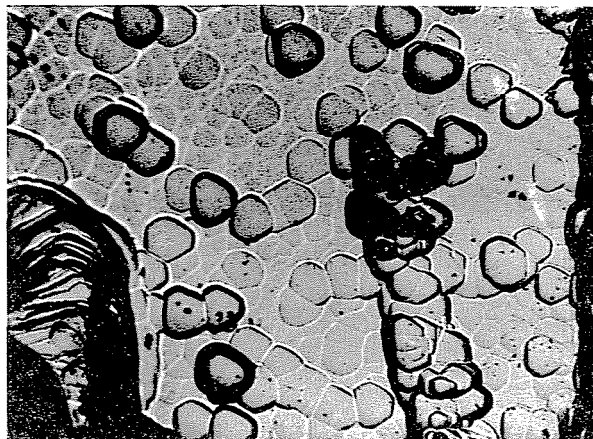
a)
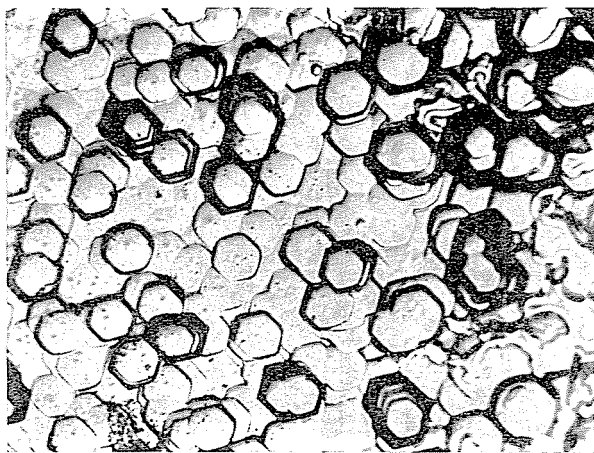
b)

Fig. 5. Light photomicrographs of the etched surface of grinding grains of silicon carbide. a) ×300; b) ×300

inhomogeneities, cannot be considered in this case as directly available information since the processes concomitant to the bonding of silicon carbide grains are extremely complex problems at present uncleared even in literature. In the present state our research does not aim at deeper investigations in this

domain of problems. Owing to the already mentioned structure and behaviour of silicon carbide, even today empirical experience is determining factor of the bonding of silicon carbide.

a)
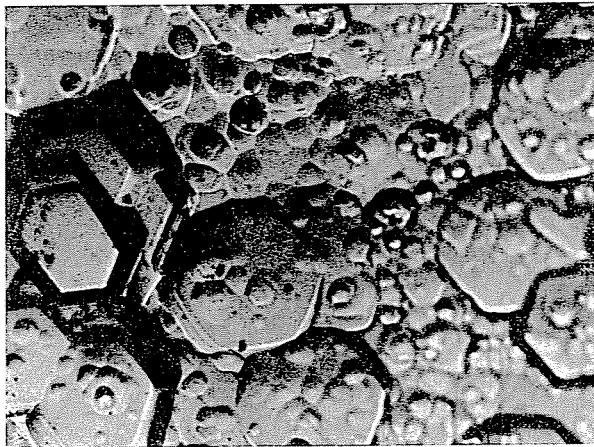
b)

Fig. 6. Light photomicrographs of the etched surface of grinding grains of silicon carbide.
a) ×300; b) ×600

The embedding of silicon carbide grains into the bonding material and the linkage of the coats of bonding material to each other are different from those of the tools of corundum grains, due to the different nature of the bonding materials. Non-melting bonding materials which became compact on firing

do not form a continuous coat around the grains (Figs 13, 14/a, 15, and 16). The non-vitreous nature is observed also in the micromorphology of the bonding material bridges connecting the silicon carbide grains (Fig. 14/b).
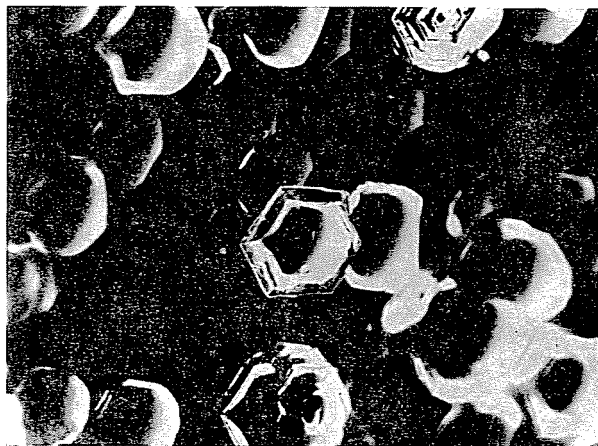
a)
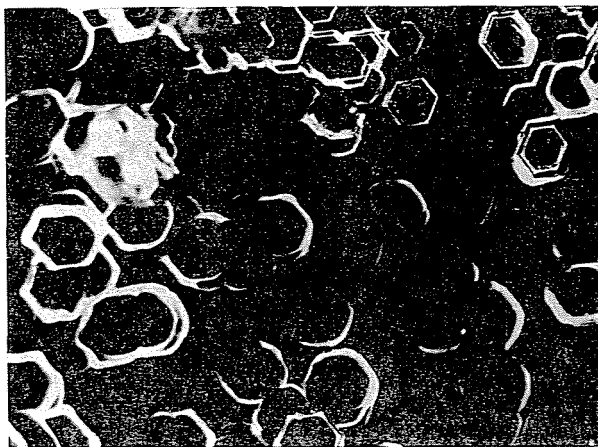
b)

Fig. 7. Light photomicrographs of the etched surface of grinding grains of silicon carbide.
a) ×300; b) ×300

The microcrystalline structure of the bonding material and the way of its linkage are shown there in a plastic manner. Of the scanning electron micrographs (Figs 16 and 21), prepared from the fracture surface and illustrate also the bonding conditions 'below the surface' are very instructive.

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Electron microscope is suitable for taking micrographs of the phase boundary at greater resolutions (Figs 22 to 24). In these micrographs silicon carbide is unequivocally distinguished from the ceramic bonding material,
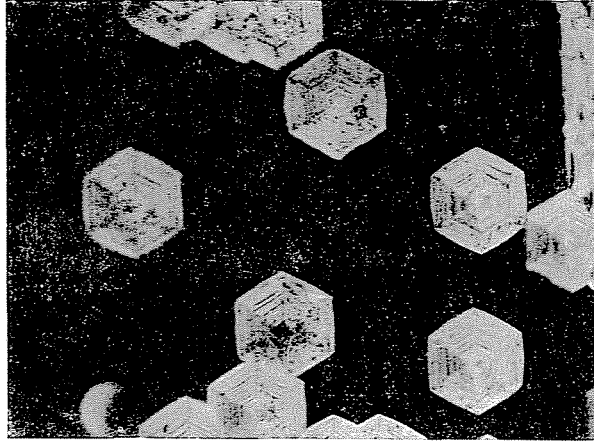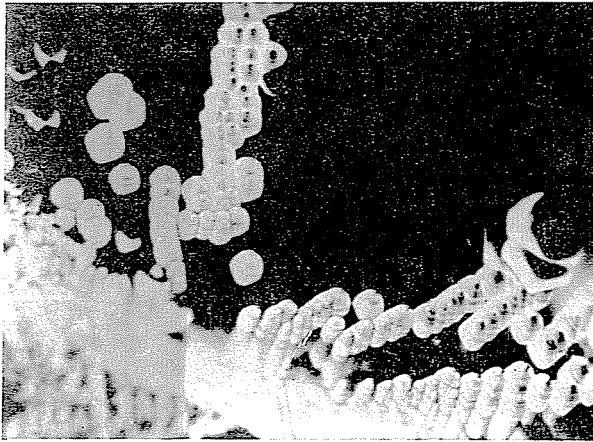

a)
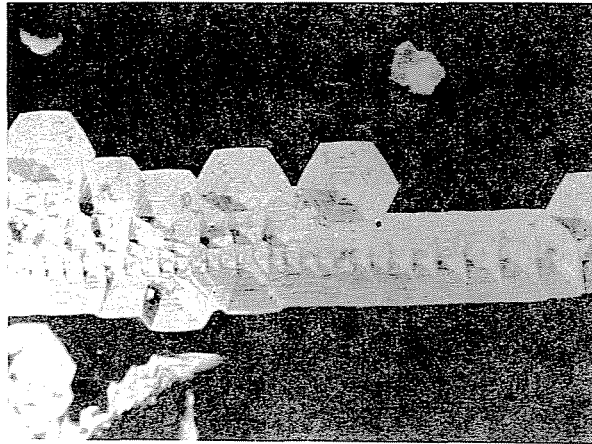
b)

Fig. 8. Light photomicrographs of the etched surface of grinding grains of silicon carbide.
a) ×300: b) ×300

due to differences in their microstructure. Between these two phases the bonding layer formed during firing and separated morphologically in a characteristic way (a layer providing a bond of adequate strength) is recognized in each case. Comparing the width of this layer with that of the transition

layers of the systems of electrocorundum and ceramic bonding material, in case of silicon carbide this width is found to be only a few micrometers. According to literature, the bonding layer is formed during firing in a way
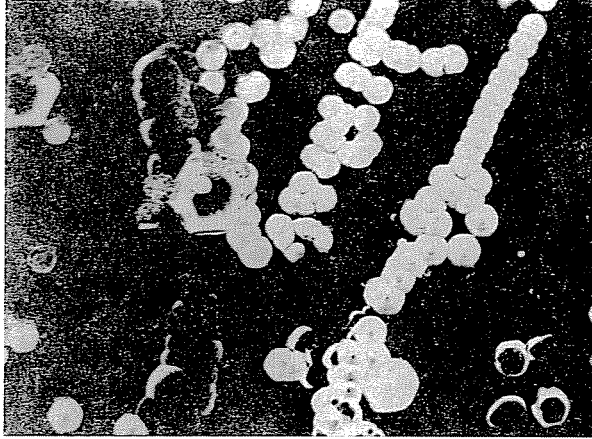
a)

b)

Fig. 9. Light photomicrographs of the etched surface of grinding grains of silicon carbide. a) ×300; b) ×300

that the surface layer of silicon carbide is oxidized by the bonding material to SiO_2 and then the bonding material adheres to this thin SiO_2 film. Up to present, we did not succeed in supporting this presumption by direct experimental evidences. The micromorphological rearrengement of the bonding

a)

b)

Fig. 10. Light photomicrographs of the etched surface of grinding grains of silicon carbide.
a) ×300; b) ×300

material in the environment of the phase boundary is clearly seen also in Fig. 23.
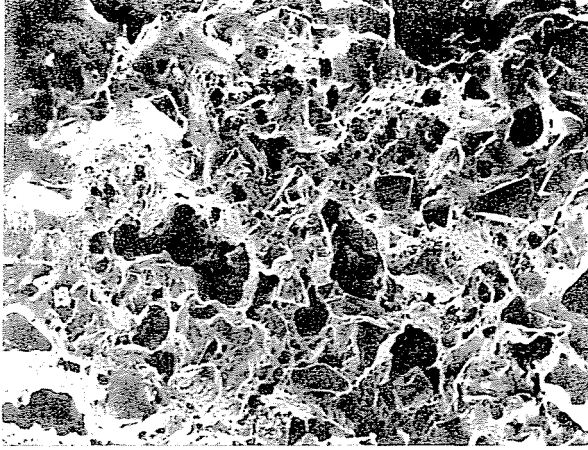
The further investigation of the bonding processes of silicon carbide grinding tools is a part of our future research aims.
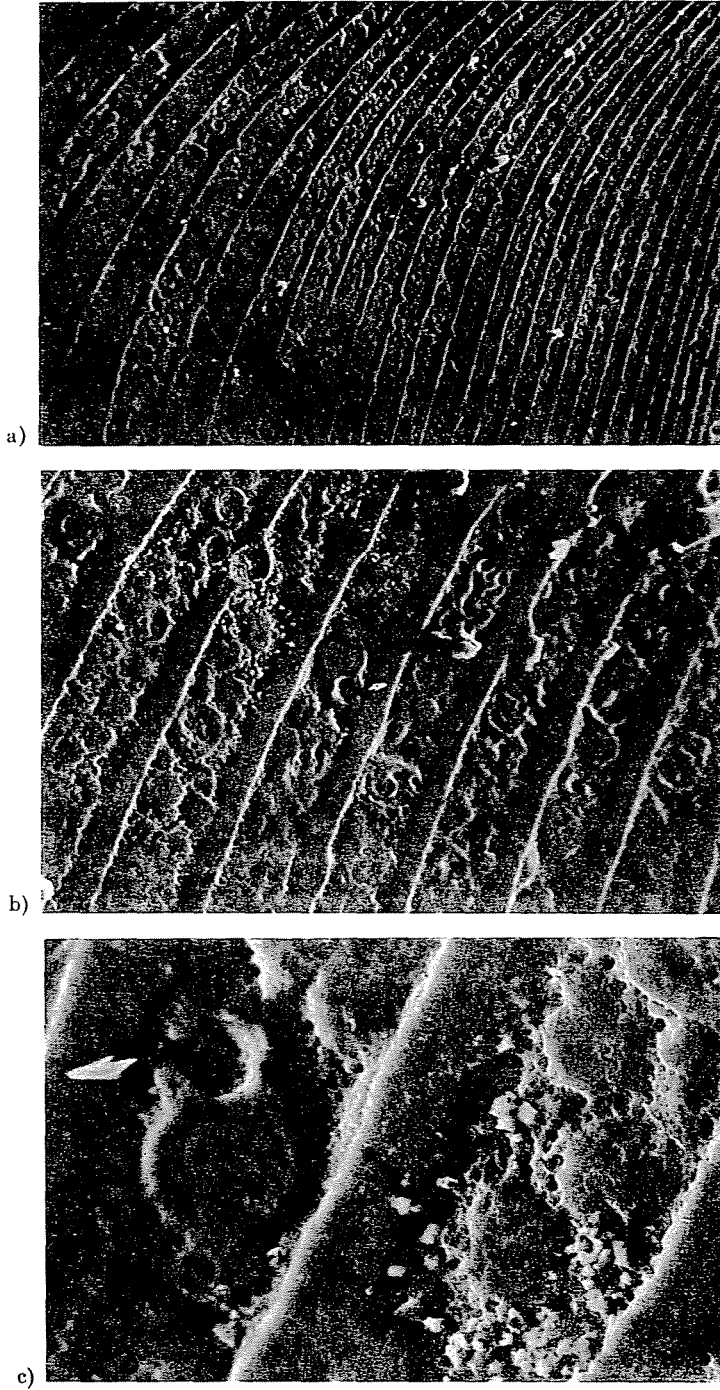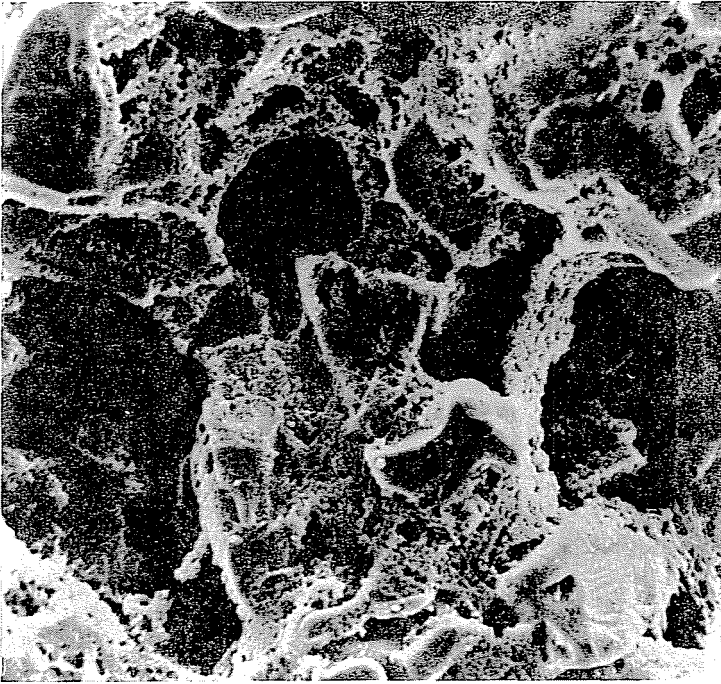
Fig. 11. Scanning electron micrographs of the non-etched surface of a grain of silicon carbide.
a) ×150; b) ×440; c) ×1450
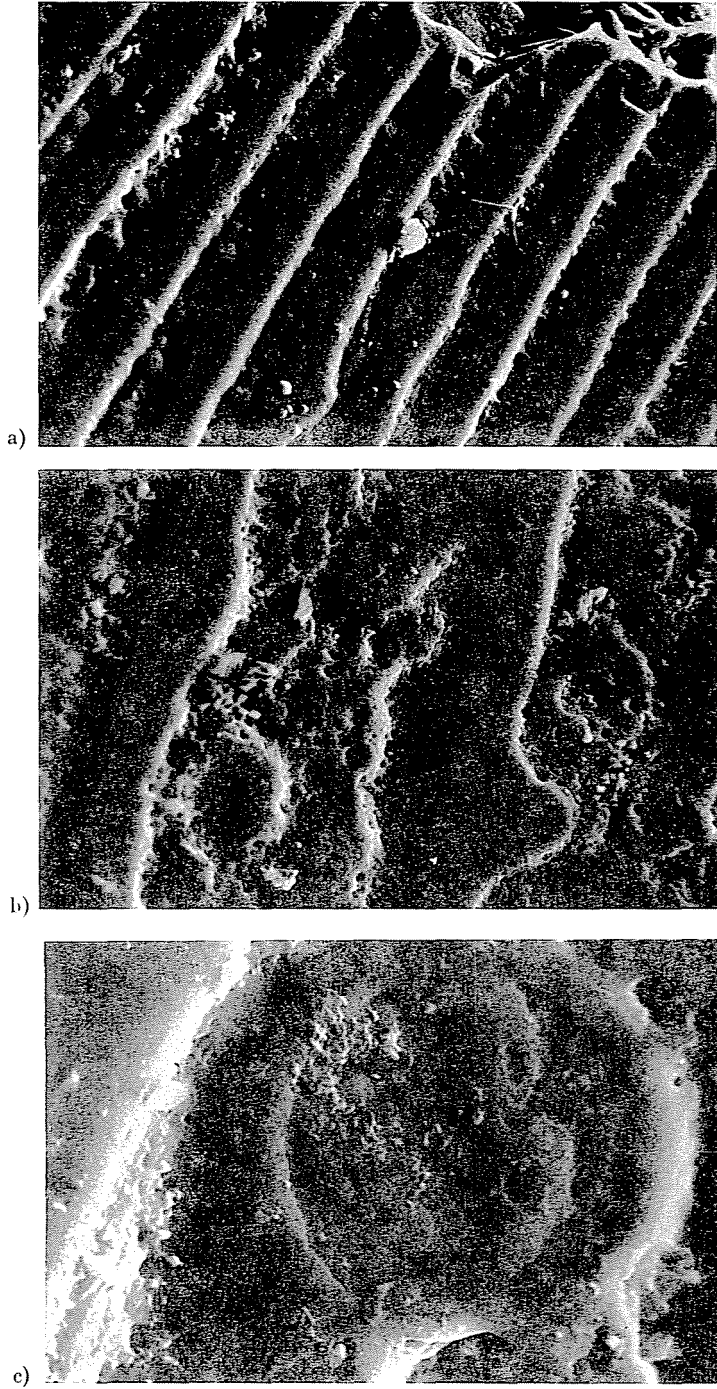
Fig. 12. Scanning electron micrographs of the non-etched surface of a grain of silicon carbide.
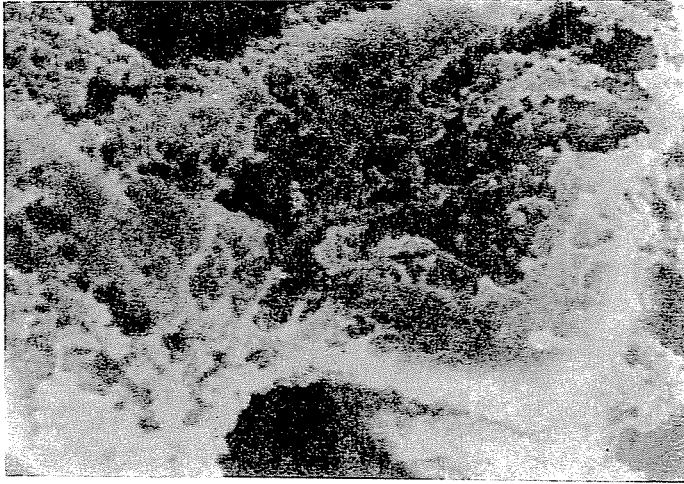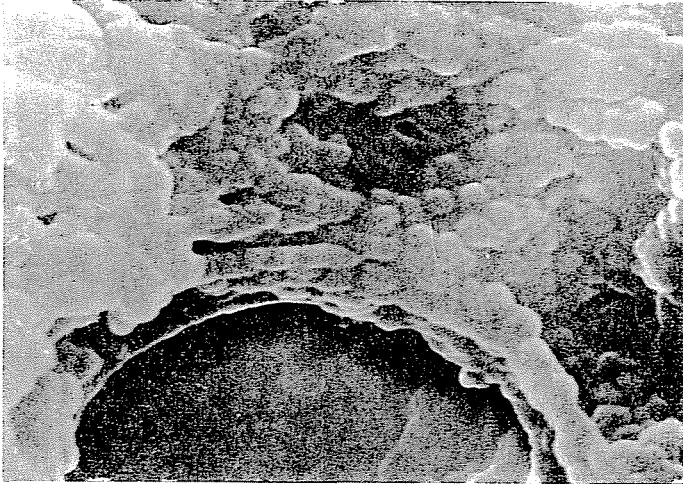a) ×1400, b) ×1400, c) ×3700

a)

b)

Fig. 13. Scanning electron micrographs of the structure of a grinding tool with ceramically bonded silicon carbide grains. a) ×525; b) ×1500
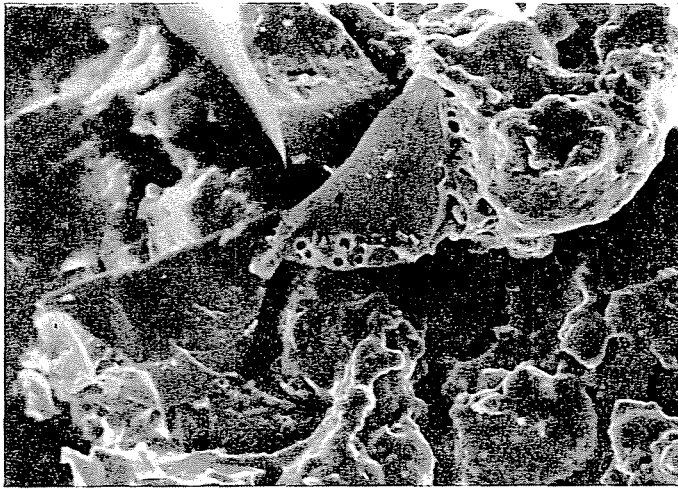
a)

b)

Fig. 14. Scanning electron micrographs of the structure of a grinding tool with ceramically bonded silicon carbide grains. a) Grain surface coated with bonding material. ×8000; b) microstructure of ceramic bonding material bridge. ×23.000
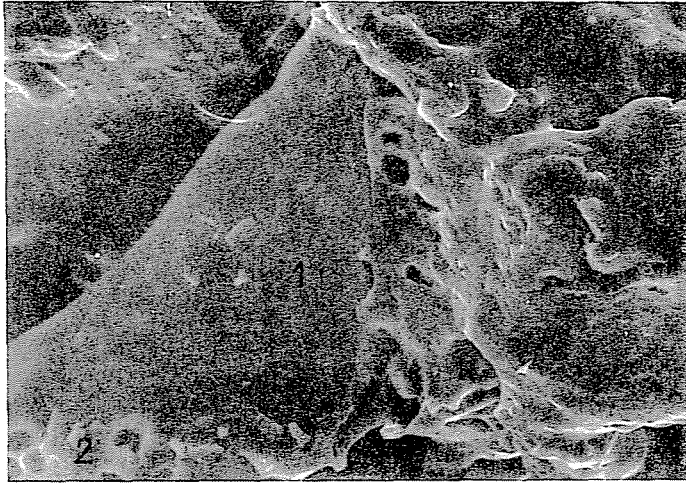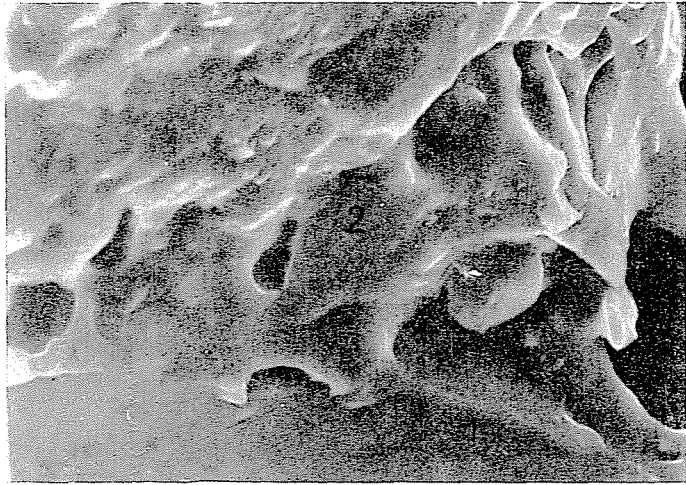
a)

b)

Fig. 15. Scanning electron micrographs of the structure of a grinding tool with ceramically bonded silicon carbide grains. a) Linkage of grains coated with bonding material. ×1250; b) fracture surface of silicon carbide (1) and ceramic bonding material (2). ×600

a)

b)

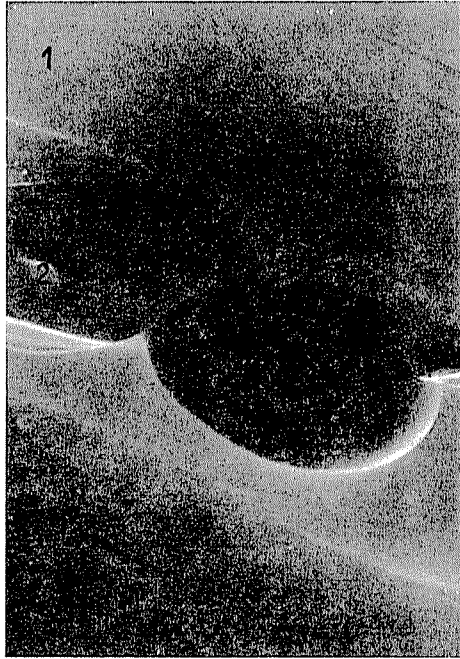Fig. 16. Scanning electron micrographs of the phase boundary of silicon carbide (1) and ceramic bonding material (2). a) ×1200; b) ×3500

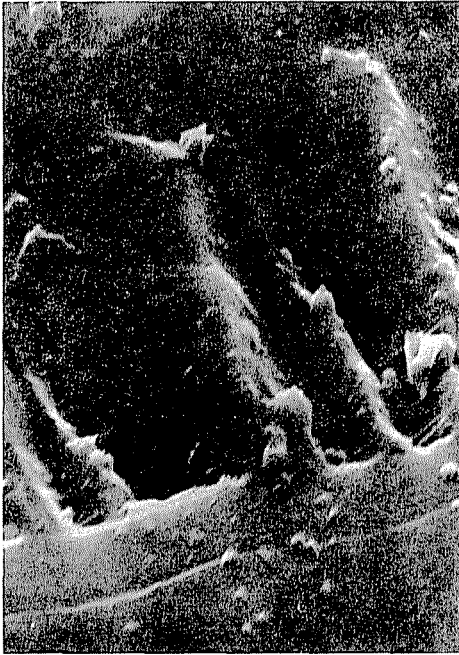*Fig. 18.* Scanning electron micrograph of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×5000

Fig. 17. Scanning electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×1600

Fig. 19. Scanning electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×4000

Fig. 20. Scanning electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×12,000
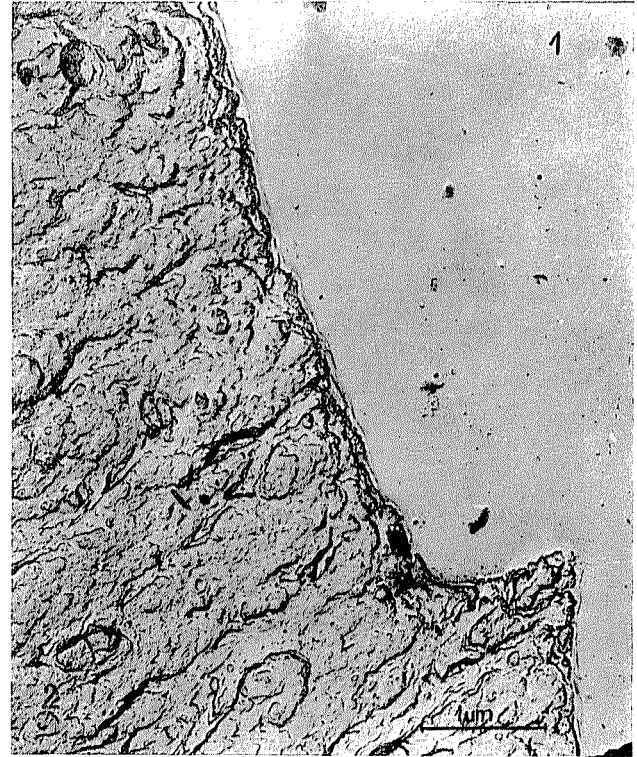
Fig. 21. Scanning electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×10,000

Fig. 22. Electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×26,000

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Fig. 24. Electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×26,000

Fig. 23. Electron micrograph of the phase boundary of a silicon carbide grinding grain (1) and ceramic bonding material (2). ×54,000
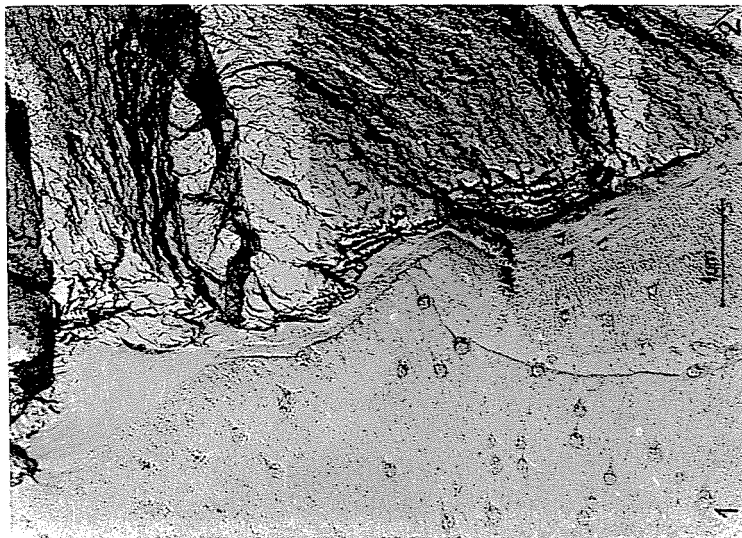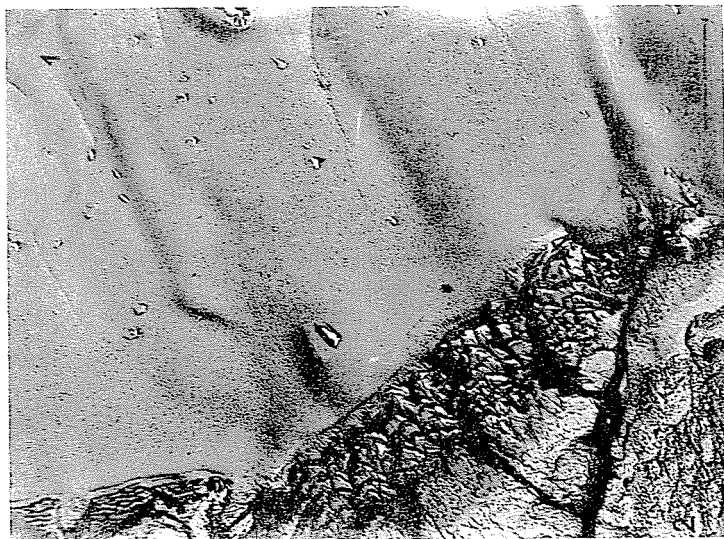
Summary

The technical significance of silicon carbide was immense already in the years following its discovery and the development of the production process at an industrial scale. Owing to the very diversified crystal structure of silicon carbide, its properties are extremely manifold. Recently, it is mentioned as the most complex inorganic compound.

The processes concomitant to the bonding of silicon carbide grains are extremely complex problems uncleared even in literature. Vitreous bonding materials cannot be used for bonding silicon carbide because they decompose silicon carbide chemically during the firing. Thus, only raw materials compacting like china can be applied as bonding materials.

The electron microscope is suitable for taking micrographs of the phase boundary of silicon carbide and ceramic bonding material at greater resolutions. Between these two phases the bonding layer formed during firing and separated morphologically in a characteristic way (layer providing a bond of adequate strenght) can be recognized in each case.

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1. MOSER, M.: Ceramically Bonded Grinding Tools (In Hungarian) Akadémiai Kiadó, Budapest 1971.
2. MOSER, M.: Microstructure of Ceramics. Microstructure and Properties of Electrocorundum and Silicon Carbide Grinding Tools. (In the press.) Akadémiai Kiadó, Budapest.

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