PREPARATION OF SIC PIECES FROM DIFFERENT CARBONACEOUS MATERIALS BY REACTIVE INFILTRATION

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Introduction
There has been in recent years an increasing demand for materials that offer a good strength, toughness, oxidation resistance and high thermal conductivity at elevated temperatures. Silicon carbide is one of the most attractive materials that combines these properties [1-4].
Reaction-bonded silicon carbides (RBSC) are fully dense engineering ceramics formed by silicon carbide produced *in situ* by a chemical reaction between silicon and carbon [5,6]. Fabrication of RBSC usually involves the forming of a compact of carbonaceous precursors which is heat-treated under inert atmosphere and later infiltrated with liquid silicon. The properties of RBSC pieces are strongly affected by the carbonaceous precursor material and the preparation conditions, which determine the structure and reactivity of the carbon precursor. In this work the effect of different carbon precursors is evaluated.

Experimental
a) Preparation of carbon semicokes and pyrolysis of woods.
Six semicokes were prepared by pyrolysis under inert atmosphere of three different petroleum residues in a laboratory-size pilot plant (V, D and E). Pyrolysis were carried out under nitrogen pressure, 1 MPa, at different temperatures 460-520ºC.
Pieces of four different kind of wood (Pine, cedar, almond, olive) were also pyrolysed at 520ºC and 1.MPa.
b) Conformation and heat-treatment of raw-semicokes and woods.
All the semicokes were ground for 1 h in a ball-mill and sieved to a particle size <60 µm. Three gram samples were conformed under uniaxial pressure at room temperature to give compacts of 50x10x5 mm. Forming pressure was optimised for each material in a range of 50-200 MPa.
The compacts obtained with the semicokes and the pyrolysed wood pieces were heat-treated at 1450 during 1h, under inert atmosphere, using a heating rate of 1 ºC/min.
c) Infiltration of the compacts.
The resulting compacts were infiltrated with silicon at 1450ºC during 3 hours, under flowing Ar (60ml/min)

Results and Discussion

Table 1 show some properties of the heat-treated (1450ºC) carbon precursor used in the work. There is a large difference in the properties of the carbon derived from the two kind of precursor. Carbons obtained from wood have a much lower bulk density and larger porosity than those obtain from semicokes. When the helium density of the powders are compared, semicokes have a much larger density as a result of the larger graphitizability of the semicoke. When comparing the different kind of woods, almond and olive gave the largest densities and lower porosities. In the case of the semicokes, there are no large differences in density and porosity values when comparing the semicokes obtained from the three different petroleum residues. These values are achieved by an optimisation of composition of the semicoke and also the forming pressure of the compacts. The mechanical properties of all the carbon compacts are very poor, specially those prepared using the semicokes, because of the large porosity.

Table 1. Properties of the heat-treated carbon pieces (1450ºC)

Sample		ρ_{bulk} (g/cm^3)	ρ_{Powder} (g/cm^3)	Porosity (%)	Bending strength (MPa)	E (GPa)
Wood	Pine	0.32	1.58	78	19	2.6
	Cedar	0.30	1.44	78	13	1.3
	Almond	0.66	1.31	49	17	0.6
	Olive	0.65	1.31	51	21	0.6
Semicoke	V-A	1.20	2.13	43	5	1.1
	V-B	1.24	2.15	43	5	1.2
	D-A	1.16	2.15	46	1	0.5
	D-B	1.21	2.08	42	3	0.9
	E-A	1.28	2.12	41	5	1.0
	E-B	1.27	2.12	41	5	1.0

Table 2 show the data of the infiltrated compacts prepared from the carbon precursors. After infiltration the materials still exhibit a large difference originated by the two kind of precursors. SiC pieces obtained from wood have lower density, larger porosity and lower bending strength than those obtained from petroleum semicoke. Samples obtained with almond wood exhibited the best mechanical properties. These later samples were analysed by XRD and the analysis confirmed the total conversion of carbon to ß-SiC. No carbon or Si were found in the analysis. Only by ashing the samples at 800ºC during 5h, a very small amount of silicon was found. The low mechanical properties are caused by the large porosity of the compacts. In spite of this fact, the samples obtained with almond have elevated mechanical properties.

In the case of the infiltrated compacts made from petroleum semicokes the samples may have problems of inhomogeneity caused by unreacted carbon. To avoid this problem the porosity of the carbon precursor must be larger than 40 %. The use of this large amount of initial porosity causes that the final pieces have a porosity larger than 6 %,thus affecting to the mechanical properties. However bending strength are quite high, larger than 230 MPa. When comparing the samples obtained from the different petroleum residues, the bending strength values are larger for the materials obtained with the D and lower for the V. This effect is thought to be caused by the different reactivity of the carbon precursor during the infiltration process, lower for carbon obtained with residue D. Although all these semicokes are graphitizable carbons, the semicokes obtained form V have a lower graphitic order than those obtained from D. The differences in graphitic structure have a direct effect on the reactivity, thus affecting the bending strength values. Thus, the best graphitizable carbon offers the best mechanical properties.

Table 1. Properties of the RBSC SiC pieces

Sample		ρ_{bulk} (g/cm^3)	ρ_{powder} (g/cm^3)	Porosity (%)	Bending strength (MPa)	E (GPa)	CTE (K^{-1})
Wood	Pine	1.03	2.49	59	10	5	$3.44\ 10^{-6}$
	Cedar	1.03	2.65	61	30	1	$3.38\ 10^{-6}$
	Almond	1.97	2.57	23	150	47	$3.26\ 10^{-6}$
	Olive	1.27	1.97	36	55	31	$2.81\ 10^{-6}$
Semicoke	V-A	2.56	--	--	145	78	--
	V-B	2.37	--	--	10	6	--
	D-A	2.66	3.02	12	235	97	$3.89\ 10^{-6}$
	D-B	2.69	2.87	6	105	92	$4.36\ 10^{-6}$
	E-A	2.60	2.84	9	135	76	3.6110^{-6}
	E-B	2.60	2.80	7	215	76	$4.04\ 10^{-6}$

Conclusions

The kind of carbon precursor in the preparation of compacts for silicon infiltration is a very important factor affecting the reactivity and the properties of the SiC formed. Pieces of high bending strength have been prepared using a rather porous material such as wood.

Acknowledgements

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