



UNIVERSIDADE ESTADUAL DE CAMPINAS
SISTEMA DE BIBLIOTECAS DA UNICAMP
REPOSITÓRIO DA PRODUÇÃO CIENTÍFICA E INTELLECTUAL DA UNICAMP

Versão do arquivo anexado / Version of attached file:

Versão do Editor / Published Version

Mais informações no site da editora / Further information on publisher's website:

<https://iopscience.iop.org/article/10.1088/1742-6596/605/1/012002>

DOI: 10.1088/1742-6596/605/1/012002

Direitos autorais / Publisher's copyright statement:

©2015 by IOP Publishing. All rights reserved.

DIRETORIA DE TRATAMENTO DA INFORMAÇÃO

Cidade Universitária Zeferino Vaz Barão Geraldo

CEP 13083-970 – Campinas SP

Fone: (19) 3521-6493

<http://www.repositorio.unicamp.br>

Re-carbonized vitreous carbon substrates for optical applications

F Nascimento^{1,2}, A J Damião¹, M V R Santos¹, F D Origo¹, J F Reis^{1,2},
L Trevenzoli³, A C M Gaspar⁴ and J Baesso⁴

¹Instituto de Estudos Avançados, São José dos Campos, Brazil

²Instituto Tecnológico de Aeronáutica, São José dos Campos, Brazil

³Faculdade de Engenharia Química, Universidade Estadual de Campinas, Campinas, Brazil

⁴Escola de Engenharia de Lorena, Universidade de São Paulo, Lorena, Brazil

E-mail: damiao@ieav.cta.br

Abstract. Imaging optical systems components for satellites must have low specific mass and high stiffness, as weight is a problem for payloads and stiffness is essential to keep the substrate front surface shape. In this work, Re-carbonized Vitreous Carbon (RVC) was tested as a substrate material. The process to obtain RVC is different from the traditional process to obtain the Monolithic Vitreous Carbon (MVC). It is essential to understand the process to evaluate the surface roughness data. This work describes the process to obtain RVC, as a candidate for optical component substrate, and the results of its surface roughness measurements.

1. Introduction

The traditional process to obtain the Monolithic Vitreous Carbon (MVC) is well described in the literature [1-4]. Vitreous (or glassy) carbon has low specific mass (1.5 g/cm^3) and good mechanical strength [5], combining glassy and ceramic properties [6]. In principle, it is a good material for optical substrates applications. Mainly in aerospace imaging reflective optical components low weight is essential. But the state-of-art thickness limit in this material is 10 mm [2] and could be a problem for many applications. This limit is caused by stress due to the exit of volatile gases formed during the poly (furfuryl alcohol) resin (PFA resin) carbonizations process. It is common to find 20% of the samples broken after carbonization [7]. In order to avoid the stress, a new route [8] was used to produce the substrates. The broken samples were crushed and ball milled (Retsch PM 100). The powder was classified by size. Powder metallurgy technique was applied, using the same resin as lubricant and humectants. The pressed (400 MPa) green samples were carbonizes at 1000 °C. Samples till 30 mm thick were produced, with no breaks during carbonization. The sample surfaces had the surface finishing: curve generation, grinding and polishing. The flatness and roughness were monitored during the process.



2. Material and methods

One conventional route to obtain vitreous carbon [9] can be very shortly summarized as:

1. Furfuryl alcohol + sulfuric acid → PFA resin
2. PFA resin + p-toluenesulfonic acid → polymerized resin
3. Polymerized resin + low gradient heat → vitreous carbon

All the steps depend on very controlled temperature gradient and can last 10 days. The shortly resumed route used in this work can be described as:

- a) Furfuryl alcohol + sulfuric acid → PFA resin
- b) PFA resin + p-toluenesulfonic acid → polymerized resin
- c) Polymerized resin + high gradient heat → partially carbonized resin (T=600 °C)
- d) Partially carbonized resin + ball milling + size classification → partially carbonized resin grains
- e) Partially carbonized resin grains + PFA resin + uniaxial press → green samples
- f) Green samples + medium gradient heat → re-carbonized vitreous carbon (T=1000 °C)

As the partially carbonized resin will be milled, the temperature gradient in step “a” can be high (1°C/min) in opposition to 0.1°C/min in step 3 in the conventional route. In step “f” temperature gradient of 0.5 °C/min can be used, as the amount of volatiles is much lower and also the volatiles can leave of the sample among the grains during the (final) carbonization process. None of the samples broke at the end of the process, which lasted 5 days. The number of open and closed pores on the substrate depends on: the amount of catalyst acid added to furfurylic; the proportion of resin added to the partially carbonized resin; the powder grain size chosen in step “e”. Smaller grains lead to smoother surfaces [10]. As the uniaxial pressing is done using one moving piston, the neutral line appears in one surface. Hardness measurements showed a factor of 3 between the front and back surfaces. The front part was polished and the back part was honeycomb milled, in order to lower the weight.

All the surface conditioning was done using classical generation (Karl Zeiss Curve Generator), grinding (Zeiss grinding) and polishing (Loh PM 2000 and PM 250) machines. The main aspect concerning light scattering is the surface roughness. Values as $\lambda/10$ for flatness and $\lambda/100$ for roughness were the targets.

During the polishing, the flatness was monitored by a Zygo Mark IV phase interferometer and the roughness by a Taylor Hobson PGI 1000. Both equipments belong to a metrological laboratory (LMSO) that is accredited by INMETRO (Brazilian NMI).

3. Results and Discussion

Catalysis of organic compounds highly depend on the amount of each reactant, temperature, among others. The catalysis of furfuryl alcohol is a good example of this [11]. The proportion (% m/m) of sulfuric acid in the furfuryl alcohol catalyses in order to produce furfuryl resin leads to visible differences after carbonization at 1000°C (Figure 1). The size of the pieces is smaller when 1 % m/m of sulfuric acid is used when compared to 1.5 % and 2 %. All samples were carbonized in the same batch. So, the whole process to produce the re-carbonized vitreous carbon can be directed in order to get smaller grain sizes, to increase the packaging.

The proportion resin/partially carbonized resin also plays an important role on the green sample texture. When less than 20% of resin is added, it is very difficult to manipulate the samples, because they are too dry. When more than 30% is added, they become pasty. A set of samples having 20, 25 and 30% of resin in partially carbonized (at 600 and 1000 °C) resin grains is shown in figure 2.

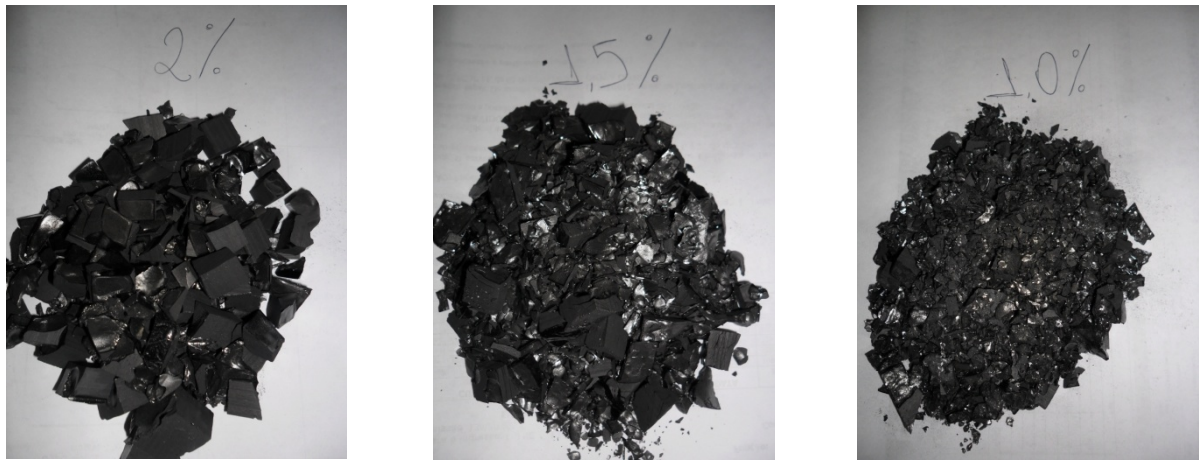


Figure 1. Vitreous carbon pieces after 1000 °C carbonization. The resin had 2% (left), 1.5 % (middle) and 1 % (right) m/m of sulfuric acid catalyst.

Next, the surfaces of the samples had the surface finishing. The first step was the surface generation. Flat surfaces were generated in a traditional curve generator, using diamond tools. The removing rate was very slow (1 $\mu\text{m}/\text{h}$) and lasted for more than 8 h, after a leveling process (Figure 3).

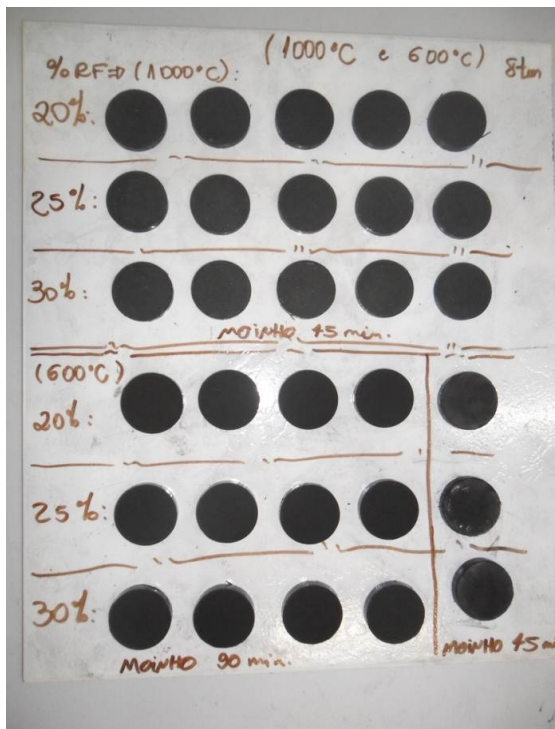


Figure 2. A set of samples having 20, 25 and 30% of resin and resin grains partially carbonized at 600 °C and 1000 °C.



Figure 3. Blocking having 5 samples during polishing process (flat).

Each blocking had 6 or 9 samples. Some samples went directly from generation to polishing. The flatness and the roughness were measured along the surface finishing process. The roughness measurements were performed on an area of 5 mm x 1 mm in the central sample of figure 3. The measurements attended the ISO 4288. The 3D image was obtained by 101 measurements on the 1 mm direction (Figure 4). Open pores can be observed and their origin can be attributed to tearing of small grains during grinding.

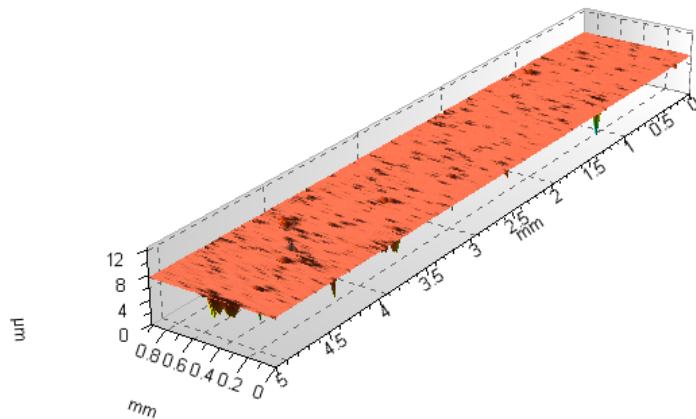


Figure 4. 3D virtual image of the surface of the central sample blocky during the polishing process.

The analysis results of the 3D surface showed a medium Ra value of 66 nm, having a high standard deviation. Both the standard deviation and the mean standard deviation are indicated in Table 1. This high deviation was caused by the presence of open pores on the surface.

Table 1. Main roughness parameter of 101 measurements performed on the central part of the sample.

Amplitude parameters – Roughness profile					
		Mean	Std dev	Min	Max
Rp	μm	0.139	0.0828	0.0461	0.492
Rv	μm	1.72	0.678	0.530	4.30
Rz	μm	1.86	0.726	0.576	4.55
Rc	μm	0.911	0.630	0.176	4.17
Rt	μm	4.15	1.91	0.832	10.3
Ra	μm	0.0656	0.0367	0.0168	0.180
Rq	μm	0.188	0.0908	0.0462	0.442
Rsk		-24.6	14.4	-65.8	-8.48
Rku		467	429	75.9	2187
Material Ratio parameters – Roughness profile					
		Mean	Std dev	Min	Max
Rmr	%	94.6	19.0	0.151	100
Rdc	μm	0.0471	0.0222	0.013	0.121

4. Conclusions

Re-carbonized Vitreous carbon can be considered as a good candidate as optical component substrate. It presented a low surface roughness, even with open pores on the surface. Probably the pores were caused by the removing of grains during the grinding or polishing process. Lower grain sizes will be used in order to get less open pores in the future.

Acknowledgments

To CNPq Projects 559925/2010-7 and 310046/2012-2.

References

- [1] Jenkins G M and Kawamura K 1976 *Polymeric carbons – carbon fiber, glass and char* (New York: Cambridge Universtiy Press)
- [2] Zarzycki J 1991 *Materials Science and Technology – a Comprehensive Treatment* vol 9, ed. R. W. Cahn, P. Haasen and E. J. Kramer, Weinheim (New York , Cambridge)
- [3] Rezende M C 1989 Pore structure and mechanical property relationships in glassy carbon obtained from furfuryl and phenolicresin *Proc. of School of Materials Science (Bath, UK)*
- [4] Edmonds T E 1985 Electroanalytical applications of carbon fibre electrodes *Analytica Chimica Acta* **175** 1-22
- [5] Chekanova V D and Fialkov A S 1971 Vitreous Carbon (Preparation, Properties, and Application) *Russian Chemical Reviews* **40** 413-28
- [6] Cowlard F C, Lewis J C 1967 Vitreous Carbon - A New Form of Carbon *Journal of Materials Science* **2** 507-12
- [7] Dondeo F and Damiao A 2013 Vitreous carbon obtained from glassy carbon powder: advantages and disadvantages *Proc. of the Annual World Conference on Carbon (Rio de Janeiro, BR, 14-19 July 2013)* pp 82
- [8] Patent was requested.
- [9] Oishi S S, Rezende M C, Origo F D, Damiao A J and Botelho E C 2013 Viscosity, pH, and Moisture in the Porosity of Poly(furfuryl alcohol) *J. Appl. Polym. Sci.* **128** 1680-86
- [10] Suryanarayana C 2001 Mechanical alloying and milling *Progress in Material Science* **46** 1-184
- [11] Schmitt C R 1974 Polyfurfuryl alcohol resins *Polym Plast Technol. Eng* **3** 121-58