Transactions of the Materials Research Society of Japan 24 [3] 311-314 (1999)

Electrical and Optical Properties of Woodceramics Thin Films Prepared by rf Magnetron Sputtering

Kiyokazu Kasai, Hiroyuki Endo and Kiyotaka Shibata*
Department of Electronics, Aomori Polytechnic College, Iidume, Goshogawara-shi 037-0002, Japan
Fax: 81-173-32-3203, e-mail: kasai@cc.aomori-pc.ac.jp
*Department of Electronics, Ibaragi Polytechnic College, 864-4, Suifu-cho, Mito-shi 310-0005, Japan
Fax: 81-292-25-6740, e-mail: shibata@ibaraki-pc.ac.jp

Abstract: Highly resistant, high-transmittance woodceramics thin films were prepared by employing rf magnetron sputtering of a woodceramics target in an argon plasma. A series of the films were deposited as a function of substrate temperature. The substrate temperature was varied in the range 50–500°C. The electrical and optical properties of the films show a dependence on the substrate temperature. Films deposited below 300°C were insulative, $\rho > 10^{10}$ Ωcm. Films deposited at 50°C had a density of 1.9-2.2g/cm^3 comparable to that of single crystal graphite. Below 200°C, films had higher transmittance than typical diamond like carbon films in the visible and infrared regions. Infrared C-H absorption spectrum was observed by FT-IR.

Keywords: new carbon films, woodceramics, high resistivity, high transmittance, C:H structure

1. Introduction

Woodceramics obtained by carbonizing woody materials, e.g., scrap wood, are new carbon materials recently developed for many industrial uses[1]. Therefore, the research on the properties of woodceramics has been done for applications in various fields, such as temperature sensors[2], humidity sensors[3], self-lubricating materials[4], and electromagnetic shielding materials[5].

To date, all of the research is on the bulk woodceramics, and few research on the properties of thin films of woodceramics has been conducted thus far. Because the applications of woodceramics are expanding, thin film technology is becoming increasingly important, especially as films used for sensors in electronic circuits, as the self-lubricating materials inside bearings and as optical materials.

Table I. Sputtering condition for Woodceramics films

Target	Woodceramics disk (Sintered at 1200°C)
Target diameter	100 mm
Sputtering gas	Argon (99.99%)
Deposition pressure	5×10^{-2} Torr
Substrate temperature	50-500°C
Input power	20 W
Substrate	Pyrex, alumina
Target substrate distance	5 cm
Sputtering time	20 h
Film thickness	1.2-2.3 μm

Through this study, it is expected that a new character can be found for thin films which are not found in bulk woodceramics.

We studied the electrical, physical, and optical properties of woodceramics thin films, deposited by rf magnetron sputtering using a woodceramics disk and varying substrate temperature.

2. Preparation of Woodceramics Thin Films

Schematic diagram of deposition chamber used in this work was shown in Figure 1. The deposition conditions were presented in Table I.

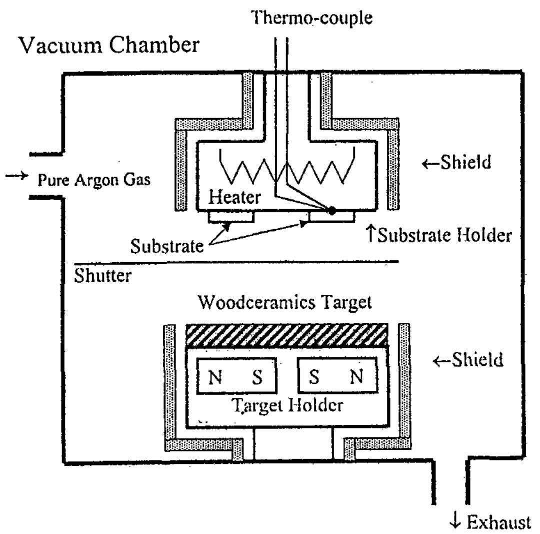

Fig. 1 Schematic diagram of deposition chamber.

Woodceramics thin films were prepared by rf magnetron sputtering of a bulk woodceramics target in pure argon gas (99.99%) at about 5×10^{-2} Torr. The target used was sintered at 1200°C and was a disk 100 mm in diameter and 10 mm thick. Films were deposited varying substrate temperatures 50-500°C. Pyrex glass and alumina sheets were used as substrates and placed at 5 cm above the target. Sputtering was done at 20 W rf power for 20 hours, yielding woodceramics thin films 1.2-2.3 μm thick.

Film thickness was measured by scanning electron microscopy (SEM) photograph of its cross section. Film density was measured using a micro- balance, surface area, and thickness before and after deposition. Electrical resistivity at room temperature was measured using an ultra high resistance meter after aluminum electrodes 25 mm in width, 10mm in gap length was deposited. Optical properties were measured using a double beam spectrophotometer. SEM and FT-IR were used to analyze film microstructure.

3. Result and Discussion

We studied the relationship between substrate temperature and deposition rate at 20 W input power (Figure 2). At 50°C,, the deposition rate is about 2 nm/min and decreases with increasing temperature reaching a value of 1 nm/min above 300°C,.

Figure 3 shows the mass density of films vs. substrate temperature. At 50°C, films have a density of 1.9-2.2 g/cm^3, which is comparable to that of single crystal graphite reported to be 2.26 g/cm^3 and to that of diamond like carbon (DLC) films prepared by dc magnetron sputtering of graphite reported to be 2.1-2.2 g/cm^3 [6]. At 100°C, density decreased sharply, to about 1.0 g/cm^3.

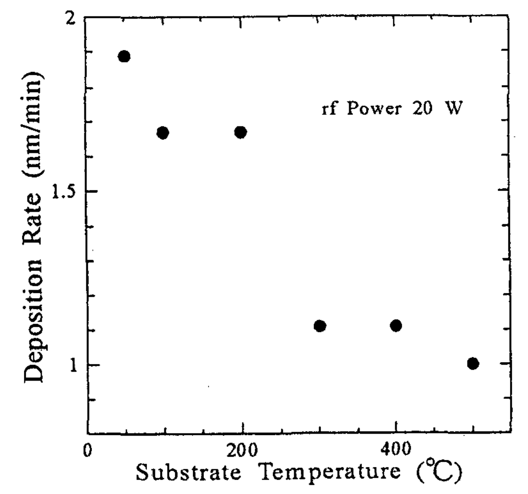

Fig. 2 The relationship between substrate temperature and deposition rate at 20W input power.

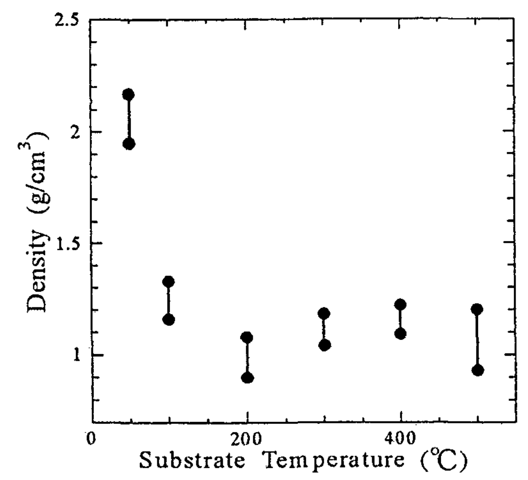

Fig. 3 The mass density vs. substrate temperature.

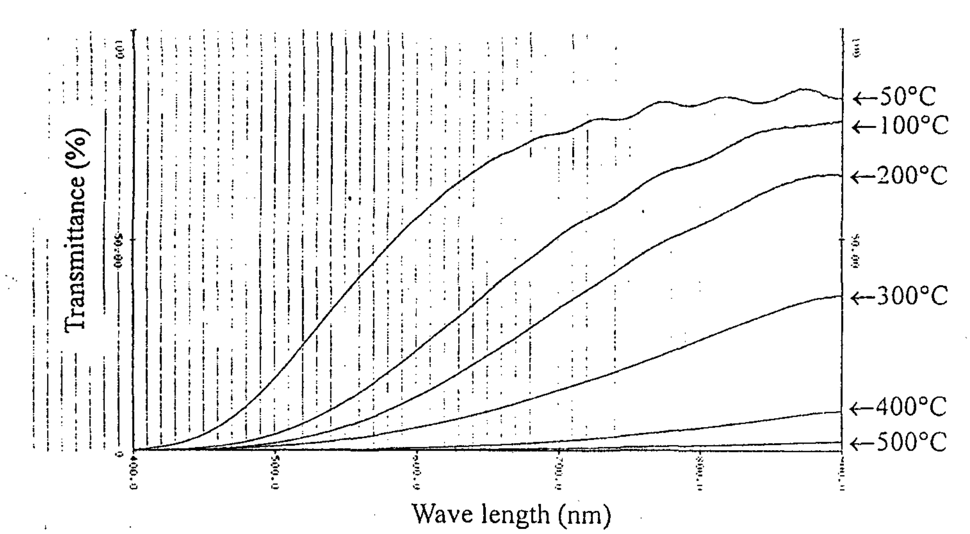

Fig. 4 Transmittance of Woodceramics film on Pyrex substrate under various substrate temperature, film thickness is 1.2-2.3μm.

Figure 4 shows the transmittance of woodceramics films on Pyrex substrates. The transmittance decreases with increasing substrate temperature. Typical DLC films are transparent only in infrared region, but woodceramics films are transparent both in the infrared and visible region at low temperature. We plotted absorption coefficient as a function of wave length with substrate temperature as a parameter (Figure 5), including plots for amorphous hydrogenated carbon (a-C:H) prepared by rf plasma deposition from benzene vapor reported to be low optical absorption[7] for comparison.

Woodceramics films clearly have lower optical absorption than that of a-C:H below 200°C. At 50°C the absorption coefficient is an order of magnitude smaller than that of a-C:H films fabricated from benzene at 700-900 nm.

The film surface was smooth according to SEM observation. Figure 6 shows results of FT-IR analysis. Infrared C-H absorption spectrum[8] was observed at 2800-3000 cm^{-1} in films deposited below 400°C. These results show that woodceramics films are hydrogenerated carbon not pure carbon and hydrogen atoms are the origin of phenol resin impregnated with woody materials before sintering. But the concentration and structure are not clear.

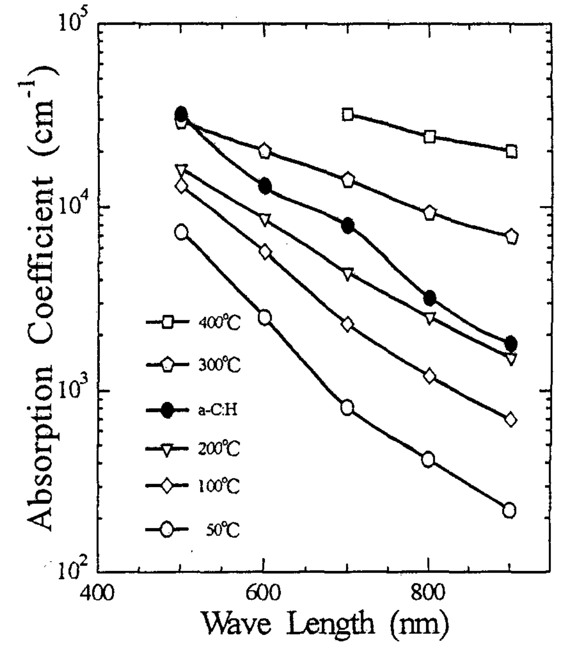

Fig.5 Absorption coefficient vs. wave length for woodceramics films and typical DLC films (Ref.8)

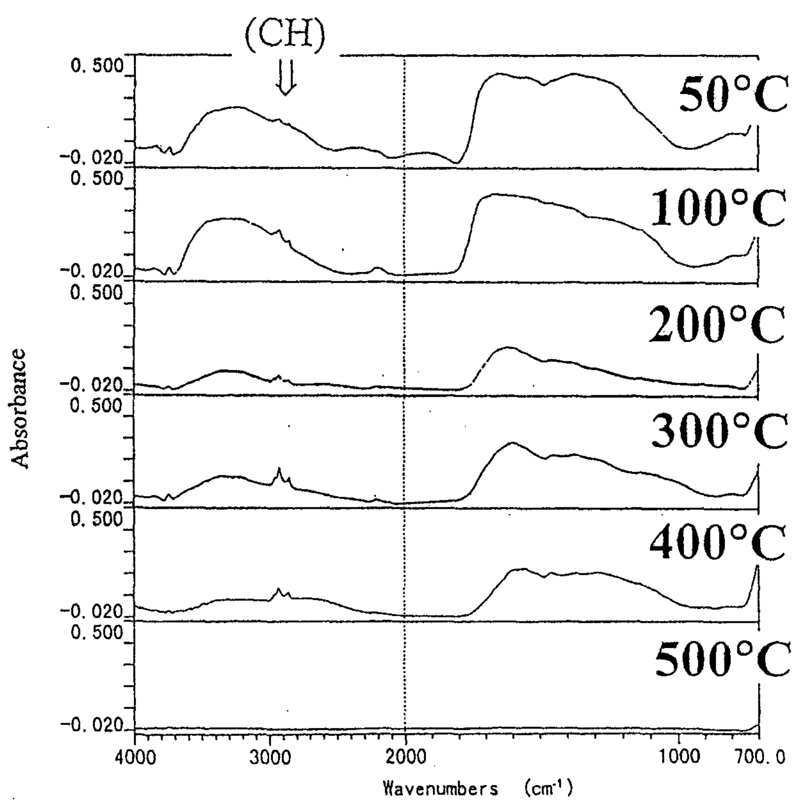

Fig. 6 Results of FT-IR analysis.

Figure 7 shows the electrical resistivity at room temperature vs. substrate temperature. Below 300°C, resistivity is higher than 10^{10} Ωcm. This insulating property is similar to that of typical DLC film. As the temperature increases to about 500°C, the resistivity decreases sharply to about 10^{7} Ωcm.

4. Conclusion

New carbon thin films of woodceramics were obtained by using rf magnetron sputtering of a bulk woodceramics target in argon plasma. Film resistivity, mass density, and transmittance depended on substrate temperature. Films prepared below 300°C have good insulating properties. Resistivity exceeds 10^{10} Ωcm. At 50°C, films density is 1.9-2.2 g/cm^3 comparable to that of single crystal graphite and DLC.

The new carbon films have high transmittance in both infrared and visible regions. Films deposited at 50°C have the lowest optical absorption and the absorption coefficient is an order of magnitude smaller than that of a-C:H films fabricated from benzene at 700-900 nm. Films have C-H bonds. Hydrogen atoms are the origin of phenol resin.

The demonstrated high transparency from visible to infrared makes new carbon coatings attractive for optical applications, e.g., HF and DF lasers.

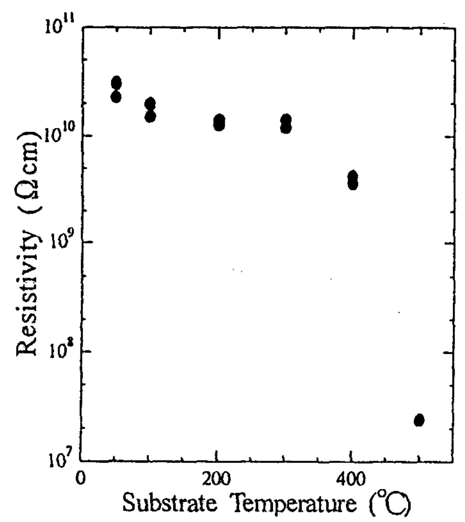

Fig. 7 Electrical resistivity at room temperature vs. substrate temperature.

Acknowledgments

We would like to thank professor Masahisa Otsuka of Shibaura Institute of Technology, Dr. Toshihiro Okabe, Mr. Kouji Saito of Industrial Research Institute of Aomori Prefecture, for many helpful comments.

References

1) T. Okabe and K. Saito, Bull. Ceram. Soc. Jpn., **28**, 32-36 (1993).

2) K. Shibata, K. kasai, T. Okabe and K. Saito, Journal of Porous Materials, **2**, 287-290 (1996).

3) K. Kasai, K. Shibata, K. Saito and T. Okabe, Journal of Porous Materials, **4**, 277-280 (1997).

4) K. Hokkirigawa, T. Okabe and K. Saito, Journal of Porous Materials, **2**, 237-240 (1996).

5) T. Okabe, K. Saito and K. Hokkirigawa, Journal of Porous Materials, **2**, 207-210 (1996).

6) N. Savvides, Journal of Applied Physics, **58**(1), 518-521 (1985).

7) B. Dischler, A. Bubenzer and P. Koidl, Appl. Phys. Lett., **42**(8), 636-638 (1983).

8) B. Dischler, A. Bubenzer and P. Koidl, Solid State Communications, **48**(2), 105-108 (1983).

(Received December 11, 1998; accepted March 18, 1999)