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Effects of Ti target current on the crystal structure, morphology and chemical structure of TiC films as deposited by dual-target DC magnetron sputtering technique

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Abstract

TiC films were deposited on silicon wafers by dual-target DC magnetron sputtering technique. The effects of Ti target current on the crystal structure, surface morphology and chemical structure of the films were investigated using X-ray diffraction, field-emission scanning electron microscopy and Raman spectroscopy, respectively. The results showed that the TiC films were deposited with the Ti target current increasing from 400 – 600 mA, the surface roughness, grain size and thickness of films increased. As the Ti target current increased, the crystallinity of TiC films increased, the orientation of TiC crystal structure corresponded to (111) and (200) planes. In addition, the Raman spectra revealed that the increasing of Ti target current had also significantly influenced on chemical structure of TiC films.

Keywords: TiC films; Dual-target DC magnetron sputtering; Ti target current

1. Introduction

Titanium carbide (TiC) is one of the widely used hard coating materials due to its high hardness, good corrosion resistance, low friction coefficient, high chemical and thermal stability [1, 2]. Typical applications include high-performance cutting and forming tools, high corrosion-resistant coatings for molten containers, thermal barriers in fusion and biomedical materials [2 – 4]. TiC films can be deposited by different methods such as physical vapor deposition (PVD) [5 – 8], chemical vapor deposition (CVD) [9, 10] and pulsed laser deposition (PLD) [11, 12]. Among various deposition methods, sputtering is one of the most used techniques of physical vapor deposition coating because it is useful to control the coating properties such as coating density, microstructure and mechanical properties [13, 14].

Dual-target DC magnetron sputtering is another technique of sputtering for depositing TiC films, the advantage of this technique is that the TiC films is deposited without using any gaseous carbon sources such as methane (CH_4) or acetylene (C_2H_4). The properties of deposited TiC films depend on the applied dual-target currents of Ti and C is an important deposition parameter [5, 6]. Therefore, the study of applied dual-target current of Ti and C is useful to understand the properties of the TiC films to develop for desired characteristics. In this work, we present results on TiC films deposited on silicon wafers by dual-target DC magnetron sputtering technique and investigate the effect of Ti target current on the crystal structure, surface morphology and chemical structure of the deposited films.

2. Materials and Methods

The TiC films were deposited on silicon wafers by dual-target DC magnetron sputtering system. Fig. 1 shows the schematic of the apparatus used in this experiment. The dual-target DC magnetron sputtering system was designed and built by the Vacuum Technology and Thin Films Research Laboratory, Faculty of Science, Burapha University. The vacuum chamber had the diameter of 310 mm and the height of 370 mm, and the cathodes used in the experiment were 75 mm diameter magnetron cathodes, using targets of Ti with the purity of 99.995% and graphite with the purity of 99.999% (Kurt J. Lesker Company). The sputtered gas was Ar with the purity of 99.999%, and the flow rate was controlled with a type 1605 flow controller (Edwards Corporation).

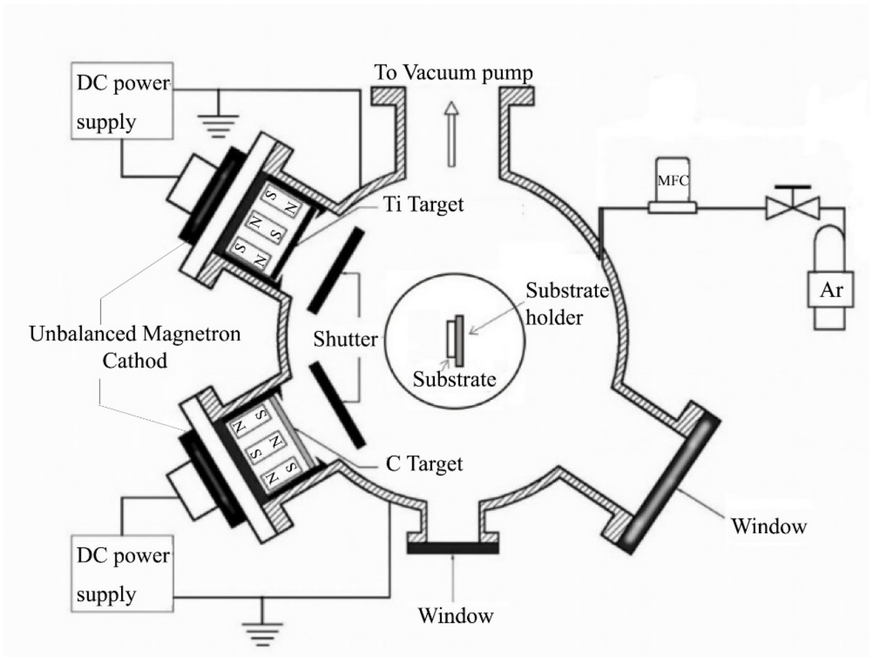

Fig. 1 Schematic of the dual-target DC magnetron sputtering apparatus used.

Deposition procedures, the samples were placed at 10 cm from the targets. The vacuum chamber was then evacuated to a base pressure of about 5 × 10^{-5} mbar to decrease the contamination in the deposition process, and the process pressure was kept constant at 3.60 × 10^{-3} mbar with the Ar gas flow rate was fixed at 4.20 sccm. The voltage of graphite target of 450 V, and the current of graphite target of 300 mA were kept constant whereas the currents of Ti target were varied from 300 to 600 mA related to the voltage of Ti target in the range between 280 – 290 V, and the deposition time used for coating was fixed at 15 min. Table 1 is a summation of the deposition parameters used in this study.

The crystal structure of deposited TiC films was characterized using X-ray diffractometer (XRD, Bruker D8 Discover) with Cu Kα radiation (λ = 1.54056 Å). The crystallite size of the films was determined from Scherrer's formula. The surface morphology and thickness of the films were examined by field-emission scanning electron microscope (FE-SEM, Hitashi S-4700). The chemical structure of the films was investigated by Raman spectroscopy (confocal Raman, NTEDRA spectra NT-MDT), working at wavelength of 532 nm.

Table 1 Detailed deposition parameters for the deposition of TiC films.

Targets	Ti and graphite
Substrate	Si (100) wafer
Target – substrate distance	10 cm
Base pressure	5×10^{-5} mbar
Sputtering pressure	3.60×10^{-3} mbar
Deposition time	15 min
Graphite Target voltage	450 V
Graphite Target current	300 mA
Ti Target voltage	280 – 290 V
Ti Target current	300, 400, 500, 600 mA

3. Results and Discussion

Fig. 2 shows the XRD patterns of the TiC films deposited on silicon wafers at different Ti target currents. At Ti target current of 300 mA, the deposited film is amorphous phase. For Ti target current of 400 to 600 mA, the transformation of the films from amorphous phase to crystalline phase. The XRD patterns of film exhibited a face-centered cubic structure and the orientation of TiC structure corresponded to (111) and (200) planes (JCPDS card no. 321383). The increasing of the crystallinity of TiC films related to the Ti target current increased. When the Ti target current is lower, ions of Ti sputtered from Ti targets cannot bond with ions of C sputtered from graphite target due to their low energy and quantity. Only if the energy and quantity of Ti ions increased to threshold with increasing Ti target current, the Ti bond with C can form compound of TiC [15].

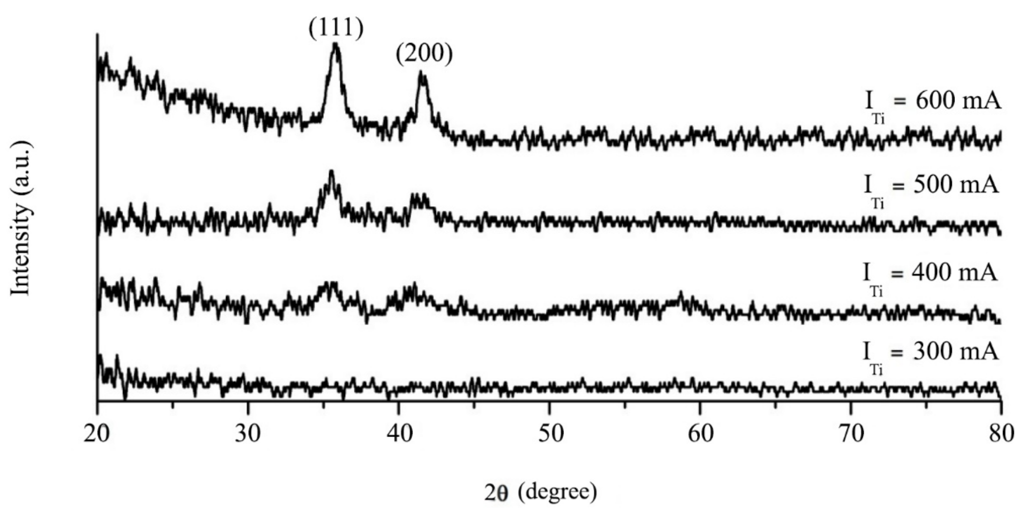

Fig. 2 XRD patterns of TiC films deposited at different Ti target currents**.**

Table 2 presents the values of crystallite size of TiC films deposited at different Ti target currents. The crystallite size of (111) and (200) planes of films increased as the increasing Ti target current. For an increase in Ti target currents, the change in crystallite size is 5.28 – 15.85 nm of (111) plane and is 4.54 – 12.68 nm of (200) plane. A decrease in the full width at half maxima (FWHM) of all the crystallographic peaks of TiC films with increasing Ti target current implying that there is an increase in the crystallite size. A reason for the large crystallite size is the presence of a high amount of Ti in the TiC films [13].

Table 2 Crystallite size of TiC films deposited at different Ti target currents.

Ti target current (mA)	Crystallite size (nm)	
	(111) plane	(200) plane
300	–	–
400	5.28	4.54
500	12.01	10.76
600	15.85	12.68

The SEM micrographs of the surface morphology of the TiC films deposited on silicon wafers at different Ti target currents are shown in Fig. 3. The results revealed that the surface roughness and the grain size of TiC films increased with increasing Ti target current. The Ti content as a function of Ti target current resulted in the evolution of the surface morphologies of the deposited TiC films [15]. When Ti target current was low, a little of Ti content incorporating with C in the films, resulting in dense films is shown in Fig. 3(a). For high Ti target current, the surface morphology of deposited TiC films is looser than that deposited with low Ti target current is shown in Fig. 3(b) – (d). The SEM cross-sectional micrographs of TiC films deposited on silicon wafers at different Ti target currents are shown in Fig. 4. The results showed that the thickness of TiC films increasing with increasing Ti target current. The thickness of films deposited at 300, 400, 500 and 600 mA were estimated to approximately 70, 95, 115 and 140 nm, respectively.

In order to investigate the chemical structure of deposited TiC films, Raman spectroscopy was performed in the range between 200 and 2000 cm^{-1}. The Raman spectra of TiC films deposited as a function of Ti target current are shown in Fig. 5. In all cases, the Raman spectra showed three peaks at Raman shifts of 610, 1358 and 1580 cm^{-1} assigned to TiC structure, D (disorder) and G (graphitic) bands of amorphous carbon structure, respectively. The intensity of TiC peak increased with increasing Ti target current which indicate that increasing of Ti content inside the deposited TiC films. This result supported the XRD analysis indicating increasing of the crystallinity of TiC films during the Ti target current increased [8, 15, 16].

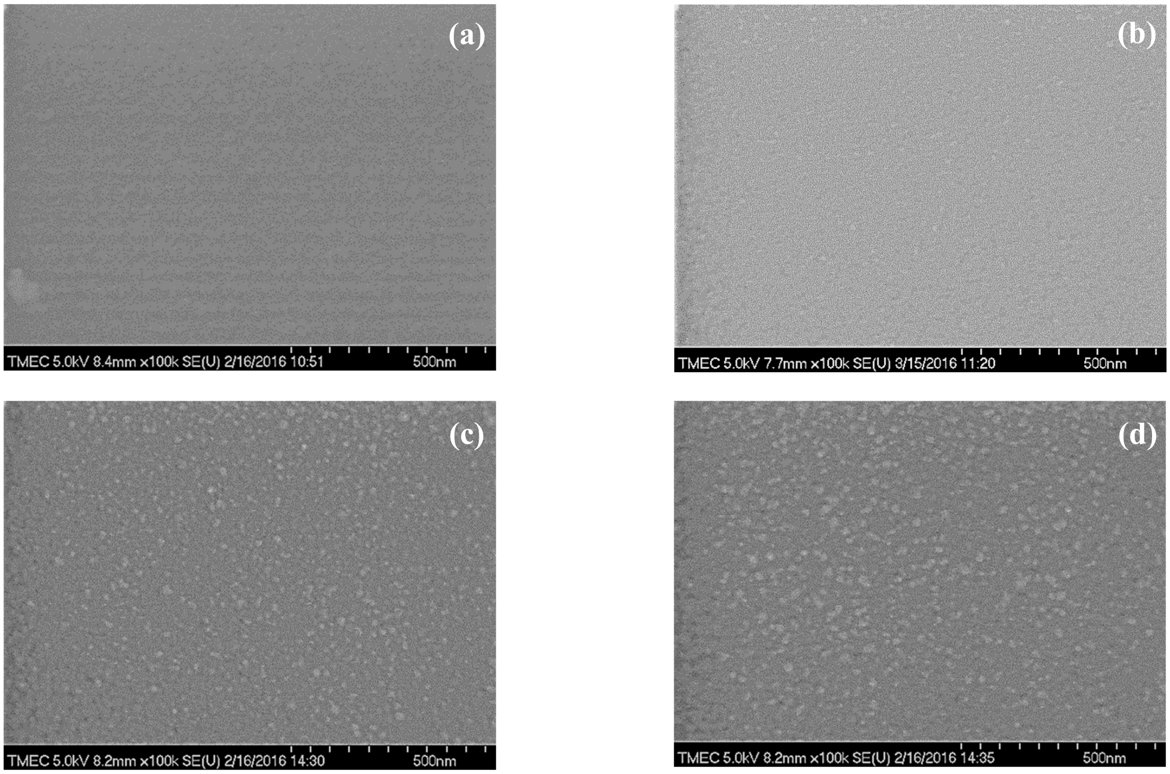

Fig. 3 SEM micrographs of surface morphology of TiC films deposited at different Ti target currents: (a) 300 mA, (b) 400 mA, (c) 500 mA and (d) 600 mA.

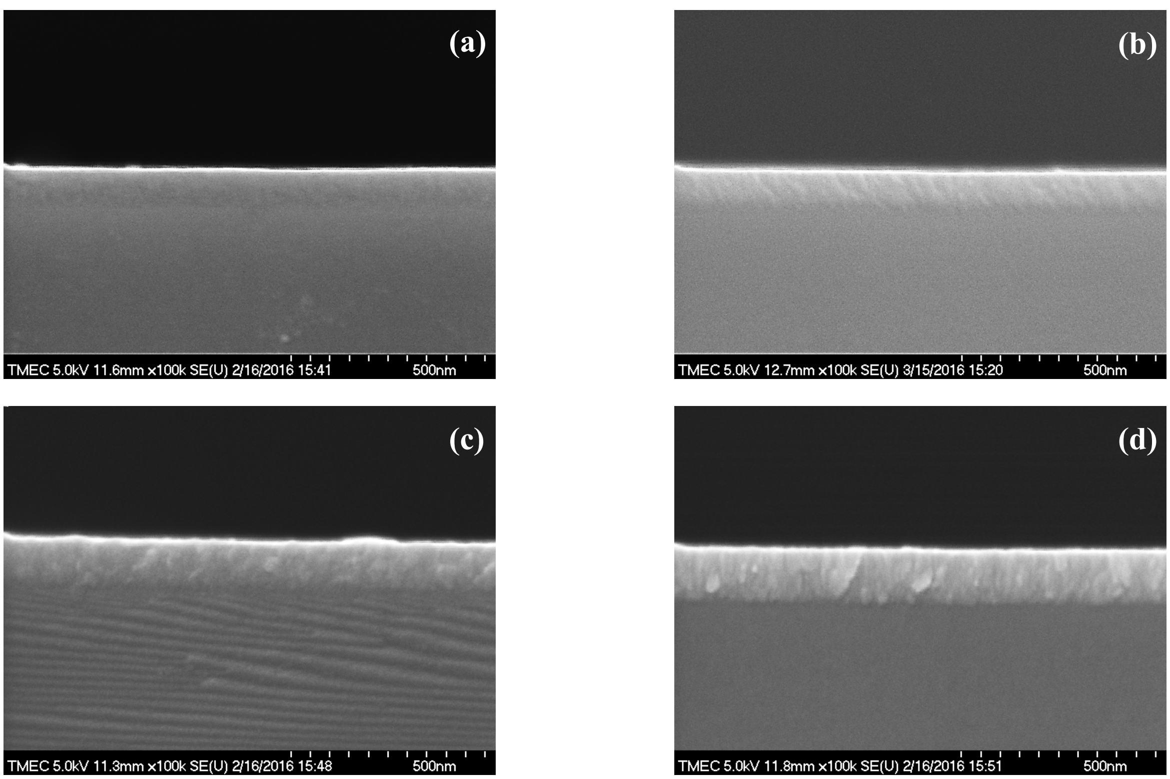

Fig. 4 SEM cross-sectional micrographs of TiC films deposited at different Ti target currents: (a) 300 mA, (b) 400 mA, (c) 500 mA and (d) 600 mA.

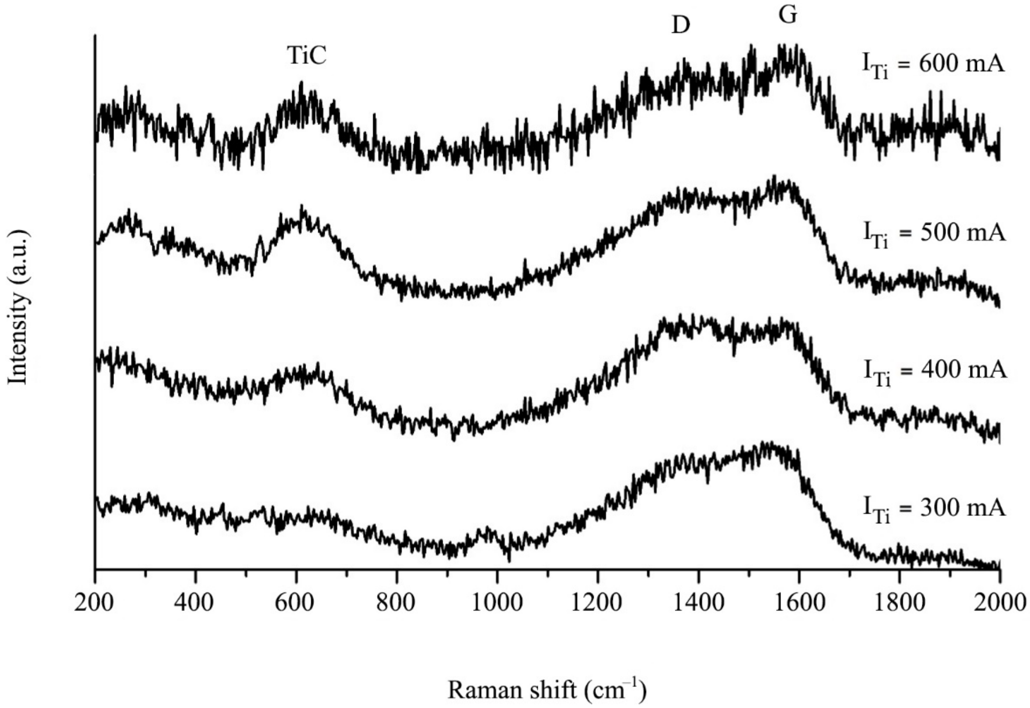

Fig. 5 Raman spectra of TiC films deposited as a function of Ti target current.

4. Conclusion

The effects of Ti target current on the crystal structure, morphology and chemical structure of TiC films deposited on silicon wafers by dual-target DC magnetron sputtering technique were investigated. According to the XRD analysis, the XRD patterns of film exhibited a face-centered cubic structure and the orientation of TiC structure corresponded to (111) and (200) planes. The increasing of the crystallinity and the crystallite size of TiC films related to the Ti target current increased. For the SEM results showed that the surface roughness, grain size and thickness of TiC films increased with increasing Ti target current. The Raman spectra revealed that the increasing of Ti target current had also significantly influenced on chemical structure of deposited TiC films.

5. Suggestions

Although the effects of Ti target current on the crystal structure, morphology and chemical structure of deposited TiC films were investigated in this research. However, it is well known that the properties of deposited TiC films depend on the different deposition parameters such as negative substrate bias, substrate heating and deposition time. We recommended that should the study of different deposition parameters for TiC coating.

6. Acknowledgement

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7. References

[1] I. Zergioti, A. Hatziapostolou, E. Hontzopoulos, A. Zervaki, G.N. Haidemenopoulos, Pyrolytic laser-based chemical vapor deposition of TiC coating, Thin Sol. Film. 271 (1995) 96 – 100.

[2] M. Shimozuma, M. Yoshino, N. Otsuka, H. Date, Y. Sakai, H. Tagashira, Deposition of TiC films by low frequency (50 Hz) plasma CVD using a substrate bias circuit, Int. Symp. Plasma Chem. 3 (1997) 1131 – 1136.

[3] K. Polychronopoulou, C. Rebholz, M.A. Baker, L. Theodorou, N.G. Demas, S.J. Hinder, A.A. Polycarpou, C.C. Doumanidis, K. Bobel, Nanostructure, mechanical and tribological properties of reactive magnetron sputtered TiC_x coatings, Diamond Relat. Mater. 17 (2008) 2054 – 2061.

[4] M.L.F. Parames, O. Conde, Growth of TiC films by thermal laser-assisted chemical vapour deposition, Appl. Surf. Sci. 109-110 (1997) 554 – 558.

[5] E. Kusano, A. Sato, N. Kikuchi, H. Nanto, A. Kinbara, Preparation of TiC films by alternate deposition of Ti and C layers using a dual magnetron sputtering source, Surf. Coat. Technol. 120 – 121 (1999) 378 – 382.

[6] S. Inoue, Y. Wada, K. Koterazawa, Deposition of TiC films by dual source dc magnetron sputtering, Vacuum. 59 (2000) 735 – 741.

[7] H. Wang, S. Zhang, Y. Li, D. Sun, Bias effect on microstructure and mechanical properties of magnetron sputtered nanocrystalline titanium carbide thin films, Thin Sol. Film. 516 (2008) 5419 – 5423.

[8] A.Z.A. Djafer, N. Saoula, N. Madaoui, A. Zerizer, Deposition and characterization of titanium carbide thin films by magnetron sputtering using Ti and TiC targets, Appl. Surf. Sci. 312 (2014) 57 – 62.

[9] I.Y. Konyashin, Thin TiC_x films chemically vapour deposited onto cemented carbides from the $TiCl_4$-CCl_4-H_2 mixture, Thin Sol. Film. 278 (1996) 37 – 44.

[10] L. Guzman, M. Bonelli, A. Miotello, D.C. Kothari, Process parameters optimization for TiN and TiC formation using reactive ion beam assisted deposition, Surf. Coat. Technol. 100 – 101 (1998) 500 – 502.

[11] F. Santerre, M.A.E. Khakani, M. Chaker, J.P. Dodelet, Properties of TiC thin films grown by pulsed laser deposition, Appl. Surf. Sci. 148 (1999) 24 – 33.

[12] Y. Suda, H. Kawasaki, K. Doi, J. Nanba, T. Ohshima, Preparation of crystalline TiC thin films grown by pulsed Nd:YAG laser deposition using Ti target in methane gas, Mater. Charact. 48 (2002) 221 – 228.

[13] K.H.T. Raman, M.S.R.N. Kiran, U. Ramamurty, G.M. Rao, Structure and mechanical properties of Ti-C films deposited using combination of pulsed DC and normal DC magnetron co-sputtering, Appl. Surf. Sci. 258 (2012) 8629 – 8635.

[14] R.D. Arnell, P.J. Kelly, Recent advances in magnetron sputtering, Surf. Coat. Technol. 112 (1999) 170 – 176.

[15] W.Y. Xin, W.L. Ping, X.Q. Ji, Influence of Ti target current on microstructure and properties of Ti-doped graphite-like carbon films, Trans. Nonferrous Met. Soc. China. 22 (2012) 1372 – 1380.

[16] Y. Cai, R.Y. Wang, H.D. Liu, C. Luo, Q. Wan, Y. Liu, H. Chen, Y.M. Chen, Q.S. Mei, B. Yang, Investigation of (Ti:N)-DLC coating prepared by ion source assisted cathodic arc ion-plating with varying Ti target currents, Diam. Relat. Mater. 69 (2016) 183 – 190.