

Production of Positrons via Pair Creation from LCS Gamma-rays and Application to Defect Study in Bulk Materials

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A new positron production and measurement apparatus has been developed at a synchrotron radiation facility. Highly energetic positrons were created via pair creation in a Pb target by implantation of 16.7 MeV photons generated via inverse Compton scattering of a Nd laser beam from a 1 GeV electron beam circulating in the storage ring at the NewSUBARU synchrotron radiation facility. These positrons, with an energy of around 8 MeV, are separated using a magnetic field and directly implanted into a thick sample to detect defects. By using laser Compton scattered (LCS) photon generated positrons, we performed positron annihilation Doppler broadening measurement for fatigue stress applied iron with a thickness of 2 mm. Vacancy type defects in nondestructive fatigue stress applied iron specimens were successfully detected by this LCS-positron apparatus.

1. Introduction

Positron annihilation techniques are very powerful methods for nondestructive tests of various materials such as fatigue damage, high energy particles irradiation damage and impurity clusters in steels and semiconductors and so on [1–5]. In recent years, many positron beam facilities, such as positron micro-beam, polarized positron beam, positronium time of flight spectroscope and positron microscope, have been constructed and developed [6–11]. Typically, the maximum positron energy is a few tens of keV at these positrons beamlines. Such slow positrons are annihilated at the near surface, with a maximum range of a few microns, in solid materials. In contrast, highly energetic positrons with an energy of MeV order can penetrate several millimeters in bulk materials, such as metals and semiconductors. Therefore, radioactive isotopes such as ²²Na ($E_{\max} = 0.54$ MeV) are commonly used for positron annihilation techniques as a positron source and, ⁷²Se ($E_{\max} = 3.3$ MeV) and ⁶⁸Ge ($E_{\max} = 1.9$ MeV) have been mainly used for defects research in bulk materials. For instance, the implantation depth of 0.54 MeV positrons, which is the maximum energy of positrons emitted from ²²Na, is calculated to be around 100 μm in bcc iron. Moreover, the energy profiles of these positrons from radioactive sources have wide distributions. Also, the positron emission intensity is not constant but decays gradually. Therefore, an energetic positron beam is the best way to get defect information in bulk samples.

In almost all positron beam apparatus, either a radioactive isotope with beta-decay or pair production using linear accelerators and nuclear reactors are used as the positron source. However, in those methods, positrons are first moderated then reaccelerated. Theoretically, electron-positron pair creation is the simplest method to obtain energy controlled positrons. Recently, some groups have successfully generated highly energetic positron-electron pairs by pair creation from inverse Comp-

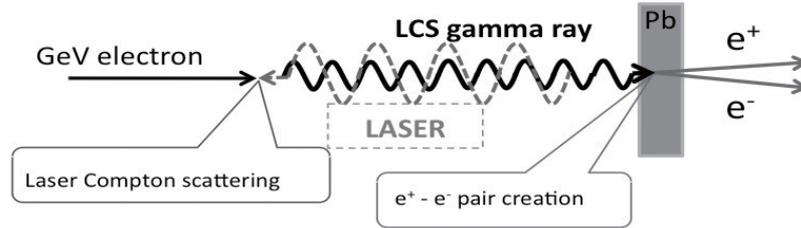


Fig. 1. Schematic illustration of LCS gamma ray generation and positron electron pair creation system.

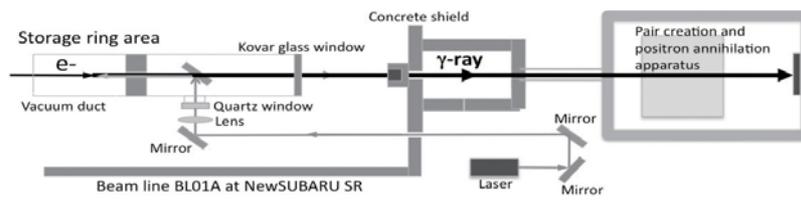


Fig. 2. Schematic illustration of experimental set up for positron annihilation Doppler broadening measurement and high energetic positron generation via pair creation by using storage ring.

ton scattering gamma rays at some facilities [12–14]. In the pair creation scheme, the positron energy spectrum, which is widely spread, depends on the gamma ray (photon) energy. By using an analyzing magnet system, we can obtain nearly-monochromatic positron energy at the position of the target material. We have developed a simple pair creation positron beam apparatus with energy of MeV order by laser Compton scattering (LCS) gamma ray from synchrotron radiation electron and incident laser light. In this system, no moderation and reacceleration of positron is needed, and we can obtain positrons with an energy of the order of keV to MeV directly. Also, the positron energy distribution and positron production rate can be controlled by the wavelength and power of the incident laser. Here, we demonstrate an application of this system to the study of defects associated with fatigue damaged thick material. In this paper, the results of positron annihilation Doppler broadening for cyclic fatigue stress applied iron samples without destruction by using this high energy LCS-positron apparatus are presented.

2. Experimental setup

2.1 Generation of positron

The basic idea of the positron generation and positron annihilation system is shown in Fig. 1. Figure 2 shows a schematic illustration of the LCS gamma ray generation and positron annihilation apparatus at the beam-line (BL01A) of the electron storage ring at the NewSUBARU SR facility at LASTI, University of Hyogo. LCS gamma rays an energy of 16.7 MeV have been generated from laser light with a wavelength of 1064 nm from a Nd: YVO₄ laser, which has a power output of 4.5 W, and 1 GeV (200 mA) electron beam circulating in the storage ring [15]. The energy of the LCS gamma rays E_γ was calculated by the scattering energy conservation using the following equation [16, 17]:

$$E_\gamma = \frac{4E_L g^2}{1 + g^2 \theta^2} \quad (1)$$

$$g = \frac{E_k}{m_e c^2} \quad (2)$$

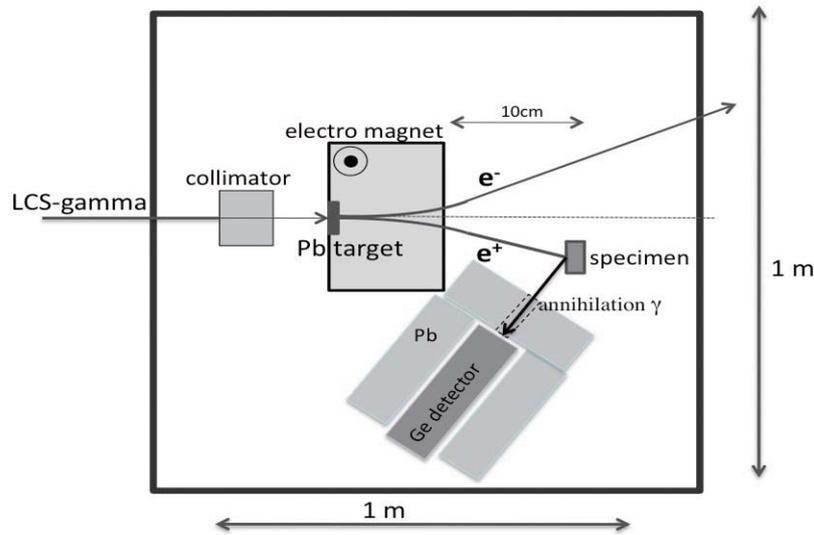


Fig. 3. Schematic illustration of LCS gamma ray generation and positron electron pair creation system.

where g is a relativistic coefficient, c is the velocity of the light, E_L is the energy of laser photon, E_k is the kinetic energy of the electron, m_e is the rest mass of the electron and θ is the scattering angle of the scattered photon against the direction of the electron. The generation rate of LCS gamma rays is about $10^5 \text{ MeV}^{-1} \cdot \text{s}^{-1}$ [18]. As shown in Fig. 3, the LCS gamma ray goes through a collimator of 6 or 3 mm in diameter and irradiates a 3 mm thick Pb plate mounted on the edge of an electromagnet. The positron production rate of this pair creation system was already measured as 1 positron per 23 photons, and the estimated positron flux is about 3600 s^{-1} [18]. The generated positron and electron pairs can be bent to opposite directions according to the magnetic field and are then separated, while the penetrated gamma ray photons are not influenced by the magnetic field. An imaging plate (IP) is set at the sample position to record the positron electron pair and incident gamma ray before positron annihilation measurement. Figure 4 shows an image of the generated positron, electron and gamma ray distribution on the IP. Applying a magnetic field of $B = 0.2 \text{ T}$ causes a separation of around 12 cm between the positron and electron positrons behind the Pb target. From the bending distance from the central point on the image, we can calculate the positron energy around the peak position to be about

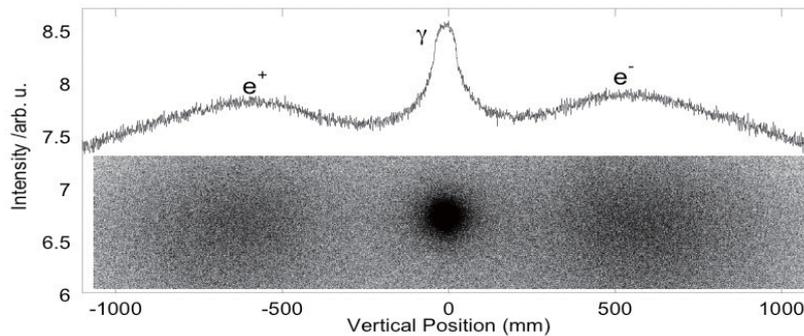


Fig. 4. Generated positron and electron profile on imaging plate after separation pass through magnetic field.

8 MeV. The bulk sample is placed where the peak position of positron profile was measured by the IP. Each sample was mounted on a x - z -rotate movable stage. Annihilation gamma rays were emitted from the sample and were detected by a high purity Ge detector. To reduce the background, the detector was covered by 50 mm thick lead blocks and the annihilation gamma rays were restricted by a 30 mm diameter aperture. As can be seen in Fig. 5, the 511 keV gamma signal, which is the signal of positron annihilation, was measured when the laser photons were incident into the storage ring. In contrast, an extremely low background and no peak at 511 keV was observed when the sample was removed. This shows that generated positrons were successfully implanted and annihilated in the bulk sample. The rate of positron annihilation events in the iron sample was estimated to be of the order of 1000 s^{-1} from the counting rate of positron annihilation gamma rays detected by the Ge detector located 10 cm from the sample. This result is in good agreement with the report of positron production measured at the same facility by Horikawa *et al.* [18] showing that positron annihilation takes place in the sample.

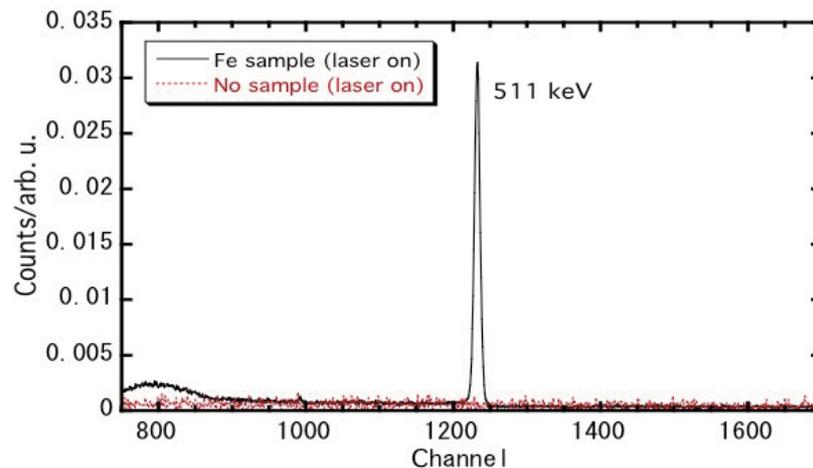


Fig. 5. Positron annihilation gamma energy spectrum emitted from the iron sample (solid line) placed at the peak position of positron intensity on IP and removed sample (broken line) when the laser beam is active.

3. Fatigue test and positron annihilation measurement

3.1 Sample preparation

Pure iron samples were prepared for fatigue stress test with a thickness of 2 mm and a width of 10 mm as shown in Fig. 6. In order to anneal out the effective lattice defects and dislocations for positron methods, the samples were annealed at 1073 K for 30 min in a vacuum of 1×10^{-4} Pa. Fatigue tests were performed at room temperature with a SHIMADZU-EHF-F1 fatigue machine. The conditions of the fatigue test are described elsewhere [19]. The expected fatigue life for these samples corresponds to the number of stress cycles ($100 \%N_f = 2.3 \times 10^5$) at a stress amplitude of $450 \text{ N}\cdot\text{mm}^{-2}$. This value was estimated by repeating the fatigue test until fracture with several samples and calculating the average result.

3.2 Doppler broadening measurement

Doppler broadening measurement using a Ge detector was performed for on unstressed, $N_f = 10 \%$ and 100% (fatigue fractured) fatigue damaged iron specimens by using LCS-positron apparatus as

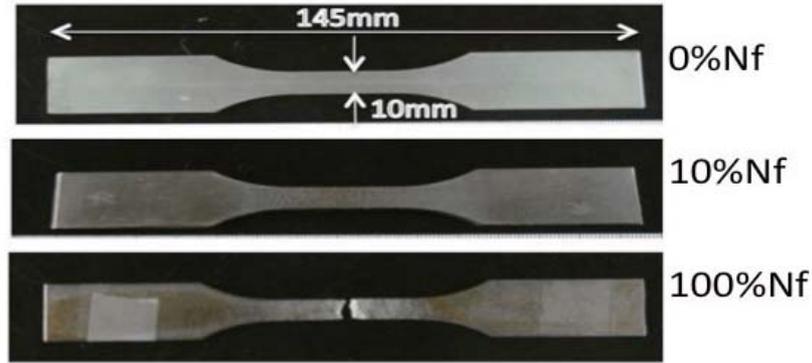


Fig. 6. Fatigue damaged iron samples.

mentioned above. The stopping profile of positrons with energy E is given by

$$P(z, E) = -\frac{d}{dz} \exp \left[-\left(\frac{z}{z_0} \right)^m \right] \quad (3)$$

with the mean penetration depth given by

$$z_0 = \frac{AE^n}{\rho\Gamma(1 + 1/m)} \quad (4)$$

where ρ is the mass density of the sample and Γ the gamma function and m , n and A are empirical parameters depending on the material and conditions [20]. From these Eqs. (3) and (4), the mean penetration depth of 8 MeV positrons into iron is estimated to be around 2–7 mm. This variation comes from various empirical parameter n reported as 1.4 to 1.6 [20–23]. Taking into account this result, annealed pure tungsten plates with a thickness of 0.8 mm were placed in front of the sample as a moderator to ensure positrons annihilate in the iron sample as much as possible. Doppler broadening spectra consisting of more than 2×10^5 counts were measured with an accumulation time of 3 to 5 hours. The windows for S -parameter analysis were defined as $511 \text{ keV} \pm 1058 \text{ eV}$ regions in all Doppler broadening spectra. It is well established that changes in S -parameter correlate well with changes in vacancy type open volume size and concentration [24]. Fatigue damaged samples were placed at the position of the generated positron image on the IP. The counting rate of positron annihilation gamma photons from these specimens was more than 25 s^{-1} in each case. The effective positron annihilation event rate in this sample was then estimated to be more than 10^3 s^{-1} . Figure 7 shows the normalized positron annihilation Doppler broadening spectra of before and after fatigue damaged iron samples. In this figure, the peak height of before fatigue stress applied iron is lower than others, and it increases with increasing fatigue cycles. This behavior is in good agreement with the results of positron measurements using radioactive ^{22}Na as a positron source [18]. This fact reveals that vacancy like lattice defects are generated not only near the surface around $100 \mu\text{m}$ but also in the thick bulk matrix after only 10 %Nf cyclic fatigue stress have been applied. There are many reports that positron annihilation parameters such as positron lifetime and Doppler broadening S -parameter, increase in the early stage of cyclic fatigue stress in various alloys and steels because of the formation of vacancy type open volume defects [25–30]. In our previous work, defects introduced by 10 %Nf cyclic fatigue stress were identified as single or di-vacancy size open volume defects concerning with dislocations, and their concentration is of the order of about 10 at.ppm [24]. Moreover, the change in S -parameter after 10 %Nf fatigue cycles compared to that before stress applied measured by this method is $\Delta S = 0.06$. This value is larger than that measured by conventional RI method ($\Delta S = 0.01$).

This difference possibly arises from the generation of defects more than 10 at.ppm concentration or larger size of open volume defects such as vacancy clusters. A similar result has been reported by Holzwarth and Schaaff by using ^{72}As as a high energy positron source, namely vacancy type defects in fatigue stress applied 316L stainless steel were detected at a depth of around 1 mm [31, 32]. From these results, it can be concluded that we can detect a large amount of vacancy type defects produced by cyclic fatigue stress in the thick bulk matrix of iron using MeV positrons from the LCS-positron apparatus.

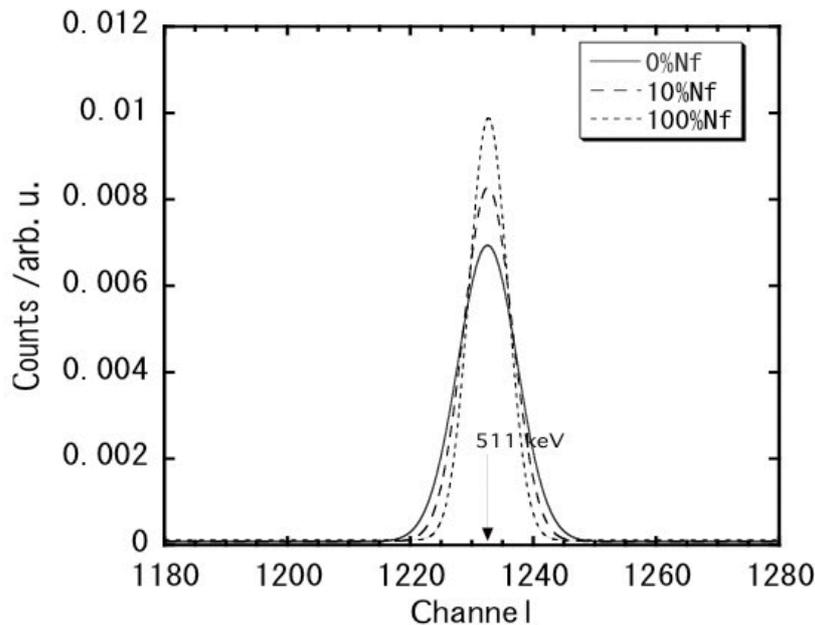


Fig. 7. Positron annihilation Doppler broadening spectra of before and after fatigue cyclic stress applied iron samples.

4. Summary

We have developed a simple, highly energetic positron annihilation measurement spectrometer, the LCS-positron, via pair creation from high energy laser Compton scattered gamma rays using the storage ring facility at NewSUBARU. Using this apparatus, we have successfully detected defects at mm depth in cyclic fatigue damaged thick iron samples non-destructively. We also found that vacancy type defects introduced by cyclic fatigue stress are not only localized near the surface but also exist in the bulk (depth \sim mm) of the iron samples.

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References

- [1] M. J. Puska and R. M. Nieminen: *Rev. Mod. Phys.* **66** (1994) 841.
- [2] A. Seeger, F. Banhart, and W. Bauer: *Positron Annihilation*, Eds. L. Dorikens-Vanpraet, M. Dorikens, and D. Segers, World Scientific, Singapore (1989) p. 275.
- [3] D. O. Welch and K. G. Lynn: *Phys. Status Solidi B-Basic Solid State Phys.* **77** (1976) 277.
- [4] M. Maekawa, Y. Fukaya, A. Yabuuchi, I. Mochizuki, and A. Kawasuso: *Nucl. Instrum. Methods Phys. Res. Sect. B-Beam Interact. Mater. Atoms* **308** (2013) 9.
- [5] M. Straticiuc, I. Pana, I. Burducea, V. Braic, P. M. Racolta, and A. Jipa: *Optoelectron. Adv. Mater.-Rapid Commun.* **6** (2012) 836.
- [6] K. Wada, T. Hyodo, A. Yagishita, M. Ikeda, S. Ohsawa, T. Shidara, K. Michishio, T. Tachibana, Y. Nagashima, and Y. Fukaya: *Eur. Phys. J. D* **66** (2012) 37.
- [7] M. Maekawa and A. Kawasuso: *Nucl. Instrum. Methods Phys. Res. Sect. B-Beam Interact. Mater. Atoms* **270** (2012) 23.
- [8] M. Matsuya, S. Jinno, T. Ootsuka, M. Inoue, T. Kurihara, M. Doyama, M. Inoue, and M. Fujinami: *Nucl. Instrum. Methods Phys. Res. Sect. A-Accel. Spectrom. Dect. Assoc. Equip.* **645** (2011) 102.
- [9] J. P. Sullivan, J. Roberts, R. W. Weed, M. R. Went, D. S. Newman, and S. J. Buckman: *Meas. Sci. Technol.* **21** (2010) 085702.
- [10] F. A. Selim, D. P. Wells, J. F. Harmon and J. Williams: *J. Appl. Phys.* **97** (2005) 113539.
- [11] K. Sakaue, T. Saito, I. Yamazaki, R. Kuroda, M. Washio, T. Hirose, T. Omori, T. Okugi, Y. Kurihara, J. Urakawa, M. Fukuda, M. Nomura, and A. Ohashi: *Int. J. Mod. Phys. B* **21** (2007) 519.
- [12] K. Dobashi, T. Hirose, T. Kumita, Y. Kurihara, T. Muto, T. Omori, T. Okugi, K. Sugiyama, and J. Urakawa: *Nucl. Instrum. Methods Phys. Res. Sect. A-Accel. Spectrom. Dect. Assoc. Equip.* **437** (1999) 169.
- [13] C. Hugenschmidt, K. Schreckenbach, D. Habs, and P. G. Thirolf: *Appl. Phys. B-Lasers Opt.* **106** (2012) 241.
- [14] T. Hirade, H. Toyokawa, T. Ohdaira, R. Suzuki, and H. Ohgaki: *Mater. Sci. Forum* **445-446** (2004) 474.
- [15] D. Li, K. Imasaki, S. Miyamoto, K. Horikawa, S. Amano, and T. Mochizuki: *Appl. Phys. Lett.* **94** (2009) 091112.
- [16] K. Horikawa, S. Miyamoto, S. Amano, and T. Mochizuki: *Nucl. Instrum. Methods Phys. Res. Sect. A-Accel. Spectrom. Dect. Assoc. Equip.* **618** (2010) 209.
- [17] S. Amano, K. Horikawa, K. Ishihara, S. Miyamoto, T. Hayakawa, T. Shizuma, and T. Mochizuki: *Nucl. Instrum. Methods Phys. Res. Sect. A-Accel. Spectrom. Dect. Assoc. Equip.* **602** (2009) 337.
- [18] K. Horikawa, S. Miyamoto, S. Amano, D. Li, K. Imasaki, and T. Mochizuki: *IEEEJ Trans. EIS* **130** (2010) 1784.
- [19] F. Hori, K. Koike, and R. Oshima: *Appl. Surf. Sci.* **242** (2005) 304.
- [20] P. J. Schultz and K. G. Lynn: *Rev. Mod. Phys.* **60** (1988) 701.
- [21] T. Aruga, S. Takamura, M. Hirose, and Y. Itoh: *Phys. Rev. B* **46** (1992) 14411.
- [22] A. Vehanen, K. Saarinen, P. Hautojärvi, and H. Huomo: *Phys. Rev. B* **35** (1987) 4606.
- [23] T. Iwai, K. Murakami, T. Iwata, and Y. Katano: *Nucl. Instrum. Methods Phys. Res. Sect. B-Beam Interact. Mater. Atoms* **315** (2013) 153.
- [24] P. J. Hautojärvi: *Positrons in Solids*, (Springer-Verlag, Berlin, 1979).
- [25] Y. Kawaguchi and N. Nakamura: *J. Jpn. Inst. Met.* **65** (2001) 835. (in Japanese)
- [26] F. Hori and R. Oshima: *Phys. Status Solidi A-Appl. Res.* **191** (2002) 409.
- [27] P. Asoka-Kumar, J. H. Hartley, R. H. Howell, P. A. Sterne, D. Akers, V. Shah, and A. Denison: *Acta Mater.* **50** (2002) 1761.
- [28] I. Müller, K. Bennowitz, M. Haaks, T. E. M. Staab, S. Eisenberg, T. Lampe, and K. Maier: *Mater. Sci. Forum*, **445-446** (2004) 498.
- [29] Y. Kawaguchi, N. Nakamura, and S. Yusa: *Mater. Trans.* **43** (2002) 727.
- [30] N. Maeda, N. Nakamura, M. Uchida, Y. Ohta, and K. Yoshida: *Nucl. Eng. Des.* **167** (1996) 169.
- [31] U. Holzwarth and P. Schaaff: *Phys. Rev. B* **69** (2004) 094110.
- [32] U. Holzwarth and P. Schaaff: *J. Mater. Sci.* **42** (2007) 5620.