Structural Analysis of Advanced Materials

Edited by Moussa Karama

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Selected, peer reviewed papers from the International Conference on Structural Analysis of Advanced Materials (ICSAAM – 2009), September 7-10, 2009, Tarbes, France

Edited by

Moussa Karama

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Preface

International Conference on Structural Analysis of Advanced Materials (ICSAAM – 2009)

The increased use of advanced materials in high efficiency structures – electronic devices, medical equipment, planes and vehicles – requires improved reliability, resistance to degradation, failure and life-span forecasts under a wide variety of loading conditions. The development of materials with advanced structural properties is becoming a key factor in industrial and technological progress. The aim of the conference is to provide a forum for engineers, and researchers, scientists and industrial experts, to present their work and discuss the present situation with regard to advanced and associated technologies, experimental techniques, numerical analyses and recent developments in the field of advanced materials. ICSAAM 2009 will include conferences by internationally renowned researchers as well as oral and poster presentations covering aspects of research and advanced material technology.

Congress website: http://www.enit.fr/ICSAAM2009/

Our conference establishes a link between process, materials and structure specialists. There is a strong connection between the various fields of research and their industrial applications. It enables companies and research organisations to discuss new means already used or usable in industry and to present their innovations. The conference tries to attract younger researchers and scientists by introducing a system of reduced participation costs and presentation of their work during plenary sessions; 20% of the participants are PhD students.

Our Scientific and Technical committee includes representatives from many countries: Europe, USA, Canada, Israel, Algeria, Tunisia, Morocco, Japan, Latin America and many regions of France - in particular Midi Pyrénées and Aquitaine. Eminent colleagues include Professor George Papanicolaou (University of Patras), Professors Costica Atanasiu and Gheorghiu Horia (Polytechnic University of Bucharest) with whom we decided to organise the 2009 ICSAAM congress in Tarbes (September 7-10, 2009) with the Laboratoire Génie de Production at the Ecole Nationale d'Ingénieurs in Tarbes.

The success of previous congresses (2005 in Roumania and 2007 in Greece) is without doubt related not only to the topics suggested and the efforts made to promote closer links between Research and Industry, but also to the effective support provided by the many varied complementary organizations.

Professor Moussa Karama Chairman ICSAAM 2009 <u>http://www.enit.fr/ICSAAM2009/index.html</u> Guest Editor Special issue Advanced Materials Research

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DETECTORS IN BARRIER STRUCTURES OF METAL- LAMELLAR SEMICONDUCTORS

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Keywords: semiconductor, detector, Shottki barrier, photoconductivity, irradiation, absorption coefficient, voltage.

Abstract. Recently peaceful use of the nuclear energy and radioactive nuclides has increased the radiation pollution factor in the world and radiation safety problems have become actual ones. The development of the nuclear power engineering, protection and processing of radioactive wastes of nuclear reactors, the use of radioactive isotopes in national economy, nuclear explosion, industrial wastes and etc. may cause the radioactive pollution of the environment. In the case of such pollution the environment as well as living organisms are exposed to radioactive radiation (α -, β -, γ - etc.). Radioactive substances emit radioactive rays and as their decay time varies for natural and artificial radioactive substances the time of their environmental effect ranges from some years to million years.

In consequence of radiation effect new specific properties – ecological, psycological, biological and so on are observed. It's necessary to determine the harmful effect of radioactive pollution areas on the environment as well as human life and radiation dose in order to protect the area, foods and people from this effect. It is of great importance to make dosimeters capable of registering γ -radiation dose in a wide energy range and with high effectiveness and radiation-resistant devices to reveal the singularities of radioactive radiation and assess its safety risks in order to protect environment and living organisms from these effects [1-4]. The dosimeters made on this purpose can be used during environmetal control, radioecological service at sanitary-and-epidemiologic stations, detection of radioactive materials and plants at customs service, emergency cases and military dangers. The pollution areas, radiation type and nature, equivalent dose of γ -rays and exposure dose rate is determined by dosimeters and the prospecting for radiactive materials and ionizing sources are realized.

A scintillation method of the registration and the spectrometry of ionizing radiation is a more developed one among the different methods of detection. This is conditioned to a high extent by the fact that scintillation materials differ for their high registration efficiency, sufficient temporal and energy resolution. The demand for scintillation detectors rises for their sensitivity and registration efficiency of ionizing radiation, space resolution and high-performance, lack of hygroscopicity, capacity to be applied under extreme climatic conditions (temperature stability, high radiation field) due to the development and creation of the new generation of high-sensitive and high-performance radiation equipment (including nuclear power engineering, space researches under extreme conditions, geophysical instrument engineering).

In comparison with other crystals A^3B^6 semiconductor crystals are much more radiationresistant, have high sensitivity and anisotropic properties in ultraviolet (UV) spectrum region. Scintillation semiconductor detectors (SD) on the base of metal- A^3B^6 lamellar semiconductors are perspective materials for electron, x- and γ -irradiation. High photoconductivity in UV-spectral region exceeding marginal region is observed in these semiconductors[5].

One of the main parameters of electron, x- and γ -irradiation SDs is a collection efficiency of charge carriers. In the given work the collection of charge carriers in the detectors on the base of metal-GaSe, GaS and InSe lamellar semiconductors has been investigated. The mode of SDs functioning is based on the generation of electron-hole pairs in contact to a metal-semiconductor and

their collection at applying reverse bias voltage. The optimal values of sample thickness, the voltage of electric field applied on contacts and photon energy in incident light have been theoretically determined which supplies effective functioning of the detector. The photoconductivity (PC) has been calculated depending on electric field voltage and light absorption.

A spatial distribution of non-equilibrium electrons $\Delta n(x)$ along the crystal thickness during their excitation at several values of the absorption coefficient: $\alpha = 1 \text{ cm}^{-1}$, $\alpha = 10 \text{ cm}^{-1}$, $\alpha = 100 \text{ cm}^{-1}$ has been theoretically investigated. During the generation of the electrons at $\alpha = 1 \text{ cm}^{-1}$ non-equilibrium electrons are distributed at zero bias (V=0) more or less uniformly along the crystal thickness. At imposing external field photoexcited electrons may pass into the crystal depth. The bias voltage is applied at so high values that all excited electrons reach back electrode (the opposite one) for efficient functioning of the detector.

It has been theoretically determined that PC in the region of low energy values on negatively charged irradiated surface during rise in voltage increases but at an opposite polarity of the applied voltage decreases. PC decreases up to a limiting value in the region of high energy values during rise in voltage, after which the marginal PC increases in four orders. Besides, during rise in voltage the margin of PC moves to a short-wave region. By this way, the optimal values of the parameters have been determined at which a PC maximum is observed in the given structure.

The obtained theoretical results agree with the experimental data. The investigated n-InSe samples with $\rho \sim 10^6$ Ohm·cm specific resistance at room temperature have been grown by the Bridgman method.

The surface-barrier structure has been formed by the vacuum deposition of Au film with 2-2.5mcm thickness on a freshly cleaved InSe surface. Ohmic contacts have been pasted on the opposite surface of the crystal by a silver paste.

In a wide range of direct (U=0.05-100V) and reverse bias voltages the current of the investigated structure follows the I~Uⁿ power law. Besides, n power exponent for direct and reverse currents in the voltage region up to U \leq 5V turns out to be close to the unit which is usually connected with tunneling of the charge carriers or is explained by the current restricted by a space charge.

The typical spectral dependence of the obtained structures' PC at 300K temperature has been studied. During the illumination of the structure by the side of Au, PC is observed in 0.36-1.8 mcm spectral region. At the values of U=0.05-1V reverse bias photosensitivity increases which can be explained by an exciton decay at hv=1.6eV quantum energies. The dependence of a PC maximum on the electric field has been formulated.

On the base of the conducted investigations we have come into conclusion that x- and γ -irradiation detectors can be constructed on the base of metal – GaSe, GaS or InSe semiconductor which will have high sensitivity and differ according its speed.

Introduction

SDs are the most perspective materials in optoelectronics because of the small size of an active area and the possibility of providing high sensitivity by means of a highly active area creation [6]. To find ways of optimization conditions for the operation of SDs is of great interest. The optimization of SDs parameters depends on both internal structures of an active element and external conditions. Scintillation SDs on the base of metal – A^3B^6 lamellar semiconductor are perspective materials for electron, x- and γ -irradiation. One of their main parameters is efficiency of a charge carrier collection [6, 7]. The study of a charge carrier collection problems in A^3B^6 – based x- and γ -ray detectors is now actual as the lifetime of the carriers in these detectors is much less than in other semiconductors and in order to collect carriers at small thickness (1-2 mm) of the crystal it is important to put high voltage (100-300 V). These semiconductors are interesting as high PC in UV-spectral region exceeding marginal region [8, 9] is observed in these semiconductors.

In the present work charge carrier collections in x- and γ - ray detectors on the basis of Au–GaS (GaSe, InSe) is investigated. The operation principle of SD is based on the generation of electron-hole pairs in contact to a metal-semiconductor and their collection at the enclosed reverse

bias voltage [7]. The optimal values of the sample thickness, the intensity of the electric field enclosed to the contacts and the energy of falling light photons at which it is possible to provide effective operation of the detector have been defined. The calculation of the PC depending on the intensity of an electric field and light absorption has been carried out.

Mathematical description

The operation principle of SD based on the generation of electron-hole pairs on the contact of a metal-semiconductor and their collection at the enclosed pressure of an inverse displacement has been determined. The simulated semiconductor detector represents planar structure of metal-GaS (GaSe, InSe). A crystal with 2 mm thickness is considered. We shall choose an axis of coordinates on a normal surface of the detector, the beginning of the coordinates x=0 we shall fix on the border of the semiconductor contact with a metal electrode. The electric field is directed along the distribution of falling light beam on the sample (Fig.1).



Fig.1 The scheme of PC excitation (metal - GaS, GaSe, InSe).

The mobility of the holes in the layered semiconductors is much less in comparison with the electrons and consequently a hole component of PC $e\mu_p\Delta p$ can be neglected in comparison with $e\mu_n\Delta n$ electron (e- is electron charge, Δp and Δn – are excess concentration accordingly holes and electrons). The concentration distribution for the electrons is determined according to continuity equation:

$$\frac{1}{e}\frac{dj_n}{dx} - \frac{\Delta n}{\tau_n} + \Phi_0 \alpha \exp(-\alpha x) = 0.$$
(1)

Where j_n – density of an electronic current, τ_n – electron's life time, $\Phi_0 \alpha \exp(-\alpha x)$ – velocity of electron photocarriers generation, Φ_0 – number of the photons falling on a unit area in unit of time, α – optical absorption coefficient. By considering the drift and diffusion components of current we shall obtain:

$$j_n = eF\mu_n + eD_n \frac{dn}{dx}.$$
(2)

Given (Eq.2) in (Eq.1) we shall obtain the equation of a special solution:

$$\Delta n(x) = C \exp(-\alpha x). \tag{3}$$

Where:

$$C = \frac{\alpha \Phi_0 \tau}{1 + \alpha L_{dr} - \alpha^2 L_n^2}$$

 $L_{dr} = \mu_n F \tau_n$

 L_{dr} – drift length – the average distance through which an electron passes along the direction of an electric field during the electron life time τ_n ($\tau_n = 10^{-6}$ s). The intensity of an electric field is determined by an enclosed external voltage F=V/d. The electron diffusion length is:

$$\mathbf{L}_{n}=\left(\mathbf{D}_{n}\boldsymbol{\tau}_{n}\right)^{1/2}$$

Where, $D_n=0.207$ – diffusion coefficient of electrons. According to the known distribution of superfluous electrons on the thickness of the sample and the dark conductivity of the semiconductor we reveal the resistance of a crystal at irradiation:

$$R(\alpha, V) = \int_{0}^{d} \frac{dx}{e\mu_n \Delta n + \sigma_0}$$
(4)

The density of photocurrent is equal to the difference of current at irradiation and darkness:

$$I_{\phi} = V\left(\frac{1}{R} - \frac{\sigma_0}{d}\right) \tag{5}$$

Here σ_0 – dark conductivity of the semiconductor. PC calculated by the formula:

$$\Delta \sigma = \frac{I_{\phi}}{VS}.$$
(6)

S - contact area.

The results of the calculation of the electron distribution $\Delta n (x)$ at their excitation at $\alpha = 1 \text{ cm}^{-1}$, $\alpha = 10 \text{ cm}^{-1}$ and $\alpha = 100 \text{ cm}^{-1}$ are presented on Fig.2a, 2b, 2c, 2d.



Fig.2 Space electron distribution along the sample thickness at different voltages for GaS with an absorption coefficient: a) $\alpha = 1 \text{ cm}^{-1}$, b) $\alpha = 10 \text{ cm}^{-1}$, c) $\alpha = 100 \text{ cm}^{-1}$, d) $\alpha = 1 \text{ cm}^{-1}$ at an opposite polarity of the electrons for GaS.

At the generation of the electrons with a small absorption coefficient (α =1cm⁻¹) nonequilibrium electrons are distributed at zero reverse bias voltage (V=0) more or less in regular intervals on the crystal thickness with recession near to both surfaces because of a superficial recombination (Fig.2a).

In spite of the fact that the sample thickness $d \gg L_n$ at imposing external pulling field photoexcited electrons can come into the crystal depth. Except for an effective detector operation the pressure voltage of displacement is so high that all excited electrons reach the back (opposite one) electrode. At the appendix the reverse bias voltage irrespective of polarity electrons are pushed aside by a field from a frontal surface. It means that if α is so small, that $1/\alpha \gg d$, electron – hole pairs are generated in regular intervals in volume, and under the action of external electric field electrons are delayed from a negatively charged electrode, PC decreases (Fig. 2a). If the optical absorption coefficient α is big enough and the L_n is less than the crystal thickness d $(1/\alpha << d L_n >> d)$, the absorption of photons occurs in the thin layer adjoining to the irradiated surface. At the appendix to a crystal of a negative pressure voltage excited electrons will be delayed from a surface in volume and PC of the sample as a whole increases (Fig.2b, 2c). At an opposite polarity electrons will be pushed out by an electric field from a crystal and PC of the sample will decrease (Fig.2d).

In Fig.3a, 3b, 4a, 4b, 4c the curves of PC calculated at various polarities and various sizes of reverse bias voltage are presented by a formula (Eq.6). As it is evident from Fig.3a, 3b PC in the range of low energy values at a negatively charged irradiated surface voltage increases and at an opposite polarity of the enclosed voltage decreases.



Fig.3 Spectral characteristics of PC in a low energy range: a) for GaS at various polarities and various sizes of reverse bias voltage, b) for 1-GaSe, 2- InSe, 3- GaS.

In the region of high energy values (Fig.4a, 4b, 4c) at increase of the pressure PC decreases up to a limiting value then there occurs an increase edge PC on four orders. Besides, with increase of voltage the edge of PC moves into a short-wave range.

b)



Fig.4 Spectral characteristics of PC in a high energy range a) for GaS, b) for 1-GaS, 2- GaSe, 3-InSe, c) for GaSe at V = -0.01 V voltage.

In Fig.5 the voltage dependences of PC are presented at various values of an absorption coefficient.



Fig.5 Dependence of photoconductivity on a pressure voltage for GaS

The obtained results will be agreed by the experimental data [5, 8, 9]. Recession of PC in A^3B^6 crystals in the high energy region is explained by the photoexcitation of excitons of intermediate type (EIT). The enclosed electric field of a Shottki barrier on the basis of these crystals ionizing EIT eliminates the given recession.

Experiment and results

The obtained theoretical results agree with the experimental data. The investigated samples of n-InSe with $\rho \sim 10^6$ Ohm cm specific resistance at room temperature have been grown by a Bridgman method.

The surface-barrier structures have been formed by Au film vacuum deposition with 2-2.5mcm thickness on a freshly cleaved InSe surface. Ohmic contacts have been pasted on an opposite surface of the crystal by a silver paste.

In a wide range of direct (V=0.05-100V) and reverse bias voltages the current of the investigated structures follows the $I \sim U^n$ power law. Besides, n power exponent for direct and reverse currents in the voltage region up to V≤5V turns out to be close to unit which is usually connected with tunneling of charge carriers or is explained by the current restricted by a space charge (Fig.6).

The typical spectral dependence of the PC of the obtained structures at 300K temperature has been studied. During the illumination of the structure by the side of Au, PC is observed in 0.36-1.8 mcm spectral region (Fig.7). At the values of a reverse bias V=0.05-1V the photosensitivity increases which can be explained by an exciton decay at hv=1.6eV quantum energies. The dependence of a PC maximum on an electric field was formulated (Fig.8).



Fig.6 Current-voltage characteristic of Au-InSe at 300 K: 1) reverse, 2) direct bias



Fig.7 Spectral distribution of PC Au-InSe at room temperature: a) illumination from the semiconductor side, b) $E=4.4 \cdot 10^4 \text{ V/cm}^2$, c) $E=2.8 \cdot 10^4 \text{ V/cm}$: 1) illumination from the metal side, 2) illumination from the semiconductor side



Conclusion

The calculation of PC for a planar Au-p-GaS structure depending on the intensity of an electric field and the light absorption for a configuration when the external voltage is directed along the distribution of exciting light beam was spent, i.e. under the conditions of division of electron-hole pairs and collecting of carriers similarly to how it happens in the x- and γ -ray detector. At such scheme the value of PC and the form of the obtained spectral curves strongly depend on the polarity and size of reverse bias voltage. The optimum values of these parameters at which a PC maximum of the given structure is observed are certain.

It has been determined that PC in the region of low energy values on a negatively charged irradiated surface during the rise in voltage increases but at the opposite polarity of the applied voltage decreases. PC decreases up to a limiting value in the region of high energy values during the rise in voltage, after which a marginal PC increases in four orders. Besides, during the rise in a

voltage margin of PC moves to a short-wave region. By this way the optimal values of the parameters have been determined at which a PC maximum is observed in the given structure.

The photon detector on the base of Au – InSe was developed, obtained and tested in the laboratory "Radiation physics of semiconductors" of Institute of Radiation Problems of ANAS (Fig.9), on which it is possible to create a scintillation detector of γ -radiation [10].



Fig.9 Photon detectors on the base of Au - InSe

The suggested detector-avalanche photodiodes of a new generation allows recording the light of minute intensity. They differ from their analogues according to their small sizes, high sensitivity (10^6-10^7 V/Vt) and operation speed.

The areas of application are in medicine – physiotherapy, blood autotransfusion, human solarization; in agriculture – greenhouse and hothouse agrotechnology; in biotechnology – synthesis of D_2 , D_3 vitamins; in disinfection of water, air, clothes, instruments and food products during long-term storage and epidemics; in astronavigation and UV location; in astronamy - data accessing about physical processes in non-terrestrial objects capable to irradiate UV radiation; in material science – determination of substance composition and electron structure of elements; in ecology – the problem of ozone hole, detection of environmental pollution; in nuclear physics and power engineering – recording nuclear particles with the help of scintillators; in defectoscopy, criminalities [11].

On the base of the conducted investigations we come into conclusion that x-ray and γ -irradiation detectors can be constructed on the base of metal-GaSe, GaS and InSe semiconductor which will have high sensitivity and differ according to its speed.

Summary

PC has been calculated depending on an electric field voltage and light absorption. Spatial distribution of non-equilibrium electrons along the crystal thickness during their excitation at several values of absorption coefficient has been investigated. It has been determined that PC in the region of low energy values on a negatively charged irradiated surface during rise in voltage increases but at an opposite polarity of the applied voltage decreases. PC decreases up to a limiting value in the region of high energy values during rise in voltage, after which marginal PC increases. During rise in voltage margin of PC moves to a short-wave region.

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Abbreviations	Explanations
UV	ultraviolet
SD	semiconductor detector
PC	photoconductivity
EIT	excitons of intermediate type

Metal-A³B⁶-is a barrier structure which is formed by a metal deposition on a lamellar crystal

The influence of some technological parameters on the fracture toughness of ceramic materials

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Keywords: fracture toughness , Vickers micro-indentation, finite element analysis, technological parameters.

Abstract. In order to determine the fracture toughness of the materials presenting high hardness values in the superficial layers, the Vickers micro-indentation was imposed as a reliable procedure. That method became attractive because of the relative simplicity of the experimental technique and because of its low cost. There are several calculus relationships that could be applied using the data provided by that method, in order to determine the material fracture toughness. The determination of fracture toughness using the Vickers indentation method is based on the analysis of radial cracks propagation, from the corners of the indentation trace. The length of these cracks is connected with the material fracture toughness, on the basis of some semi-empirical calculus relations that are taking into account the indentation load and some physical characteristics of the test material, as Young's modulus and Poisson's coefficient.

In the present paper, fracture toughness was determined on a series of ceramic samples, made of the same material, but with different geometrical shapes and obtained by applying different technological procedures. The influence of some technological parameters on the fracture toughness was evaluated. The material fracture toughness was determined, into the vicinity of the propagated cracks (in a sample that could be a final product), on an area with a specified geometric contour.

As a preliminary stage, a step by step FEM analysis was made, into the Vickers indentation material region, for different values of indentation load. In this manner, it was proved that the maximum stress value, on the perpendicular direction, as related to the crack diagonal plane, is always located at the peak of the indentation trace, and that is the effective start-point of cracking, for this type of indentation.

Introduction

A major goal for the current ceramic research and development is to produce some tough and strong ceramic materials, with a reliable performance. Nevertheless, the fracture toughness of ceramics remains poor, when comparing to that of metals and composites [1]. Precise design and modeling methodologies are therefore necessary, in order to predict the performance of ceramics. For such methods of analysis to be applied, statistical valid data are requested about the studied material, regarding some of its properties as fracture toughness, wear resistance, strength and hardness.

Fracture toughness values are extensively used for characterizing the fracture resistance of ceramics and other brittle materials [2]. The fracture of brittle ceramics is usually controlled by the mode I fracture toughness. A simple dimensional analysis of a body containing a crack with a length of 2a and subject to an applied stress σ shows that the stress-intensity factor at the crack tip K_I is:

$$K_I = \boldsymbol{\sigma} \cdot \boldsymbol{Y} \cdot \sqrt{\boldsymbol{a}} \tag{1}$$

where Y is a dimensionless constant that depends on sample geometry and crack configuration.

Fracture toughness of brittle materials is considered as a material parameter. It is admitted that fracture will occur at a critical stress intensity level, K_{Ic} . Most of the fracture toughness studies for metallic materials are typically using the Chevron notch technique, based on compact specimens

and round notched tensile specimens [3]. These methods require considerable time for sample preparation and for notch geometry control. Moreover, only a few of them could be applied for ceramics, because of their very high brittleness. Although numerous testing techniques are available [1, 2], there is no standard specimen type for determining fracture toughness of engineering ceramics. The choice of a specific technique is determined by the type of information that is needed. Specimen geometry, preparation and manufacture history are critical to correlate the test specimen behavior and the actual component fracture toughness.

A method that is frequently used for determining fracture toughness is the so-called indentation fracture (IF) method [4]. The importance of that method comes from its simplicity and the small material volume that is requested for conducting the K_{Ic} measurements. A Vickers microindentation is implanted onto a flat ceramic surface, and consequently some cracks develop around the indentation, their lengths being inverse proportional to the material toughness. By measuring the crack lengths, it is possible to estimate K_{Ic} for the studied material. One can say that the indentation technique has long been considered an attractive method for assessing the toughness of ceramic materials, due to the ease and low cost of its application. The results of indentation toughness measurements critically depend on the assumption of crack type (the Palmqvist type or the median/radial one) (see Fig. 1), on the equations that are used for fracture toughness calculation, and on some material-dependent and material-independent constants [3].



Fig. 1. The crack formation by Vickers indentation

The measured crack lengths are then connected with the material toughness K_{Ic} using a semiempirical calculus relation [5]:

$$K_{Ic} = \xi \sqrt{\frac{E}{H}} \left(\frac{P}{c^{3/2}}\right) \tag{2}$$

where *P* is the applied load, *E* – the test material Young's modulus, *H* - Vickers hardness number, *c* - the radial crack length, measured from the indentation center, and ξ - an empirically determined "calibration" constant that is usually taken to be 0.016 ± 0.004 .

The Palmqvist crack model equation could also be used for determining fracture toughness, but only when shallow cracks are observed. According to Niihara [6] the following equation is valid, if c/a < 3.5:

$$K_{Ic} = 0.018 \cdot H \cdot a^{1/2} \cdot \left(\frac{E}{H}\right)^{0.4} \cdot \left(\frac{a}{c} - 1\right)^{-1/2}$$
(3)

It must be emphasized that the major difficulties for obtaining some reliable and consistent fracture toughness values are the cracks type determination, together with the precise measurement of crack lengths [7].